

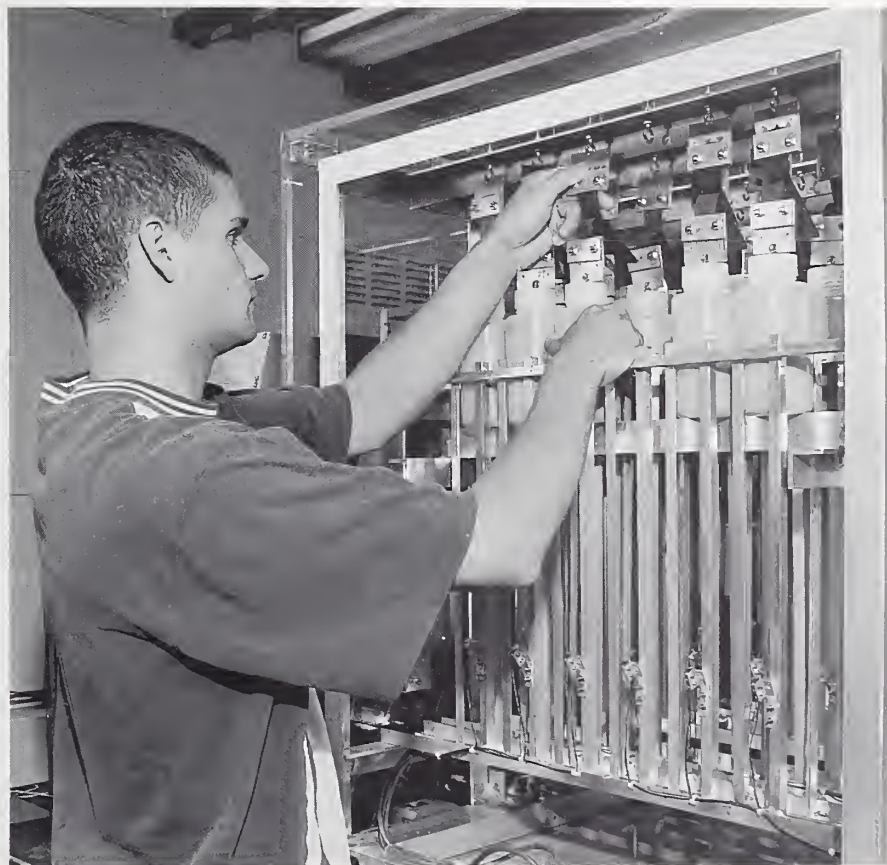


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Performance of Tape-Bonded Seams of EPDM Membranes: Comparison of the Peel Creep-Rupture Response of Tape-Bonded and Liquid-Adhesive-Bonded Seams

Walter J. Rossiter, Jr., Mark G. Vangel, Edward Embree, Kevin M. Kraft, and James F. Seiler, Jr.



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ABSTRACT

A study was conducted to compare the creep-rupture response (i.e., time-to-failure or TTF) of tape-bonded and liquid-adhesive-bonded seams of EPDM (ethylene-propylene-diene terpolymer) roofing membranes. Two commercial tape systems (i.e., tape and primer) and one liquid adhesive were applied to well-cleaned EPDM rubber. The creep-rupture experiments were conducted at 23 °C (73 °F) and 40 % to 45 % 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: $\ln(\text{mean TTF}) = b_0 + b_1 \cdot \text{Load} + b_2 \exp(b_3 \cdot \text{Load})$. A comparison of the fitted curves for the tape-bonded specimens with those for the liquid-adhesive-bonded specimens provided a basis for evaluating the relative creep-rupture response of the two types of bonding systems. Similarly, a comparison of the fitted curves for the replicate data sets of each adhesive system gave a measure of the batch-to-batch reproducibility of the creep-rupture data. The major conclusion was that the tape-bonded specimens had times-to-failure that were, in most cases, comparable to or greater than those of the liquid-adhesive-bonded specimens. And, the tape-bonded specimens provided time-to-failure results that were reproducible between replicate sets.

Key Words: adhesive tapes; adhesive testing; bonding; building technology; creep-rupture; EPDM; microscopy; roofing; seams; time-to-failure

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1. INTRODUCTION

1.1 Background

An important property of an adhesive system is its creep resistance [1]. Creep has been defined by ASTM Committee D-14 on Adhesives as "the dimensional change with time of a material under load, following the initial instantaneous elastic or rapid deformation" [2]. The importance of evaluating the creep resistance of seams of single-ply roofing membranes has been acknowledged by the roofing community. For example, ASTM Committee D08 on Roofing, Waterproofing and Bituminous Materials recently issued ASTM Standard D5405, "Test Method for Conducting Time-to-Failure (Creep-Rupture) Tests of Joints Fabricated from Non-Bituminous Organic Roof Membrane Material" [3]. To date, this method has been mostly applied to seams of EPDM (ethylene-propylene-diene terpolymer) membranes.

EPDM roofing membranes account for approximately one-third of the low-sloped roofing systems installed annually in the United States [3]. In fabricating an EPDM roofing membrane in the field, two sheets of the rubber are overlapped about 75 mm to 100 mm (3 in to 4 in), and the overlapping sheets are bonded together to form a seam. The bonding process typically uses liquid-based contact-type adhesives, although pre-formed adhesive tapes have also been used. The performance of the seam is critical to the watertightness of the EPDM membrane. Experience has shown that EPDM roofing membranes provide satisfactory field performance, but when problems arise, seams are often their source [4].

Because of the importance of seams, over the years manufacturers of EPDM membrane systems and adhesive suppliers have expended considerable effort to ensure their integrity and, from time to time, new adhesive systems have appeared on the market [5-9]. Cotsakis and Senderling [10] have described a test protocol used by one EPDM manufacturer to evaluate adhesive systems. Included in this protocol is the evaluation of the creep performance of seam specimens. However, with the exception of reports from the National Institute of Standards and Technology (NIST) and Beech et al. [11], little data on the performance of seam specimens subjected to creep loading have been reported.

NIST has conducted much research on the creep performance of liquid-adhesive-bonded EPDM seams [12-17]. Limited field observations have suggested that some seam defects result from the rheological (deformation/flow) behavior of the adhesive and not chemical deterioration [14,17]. In our creep-rupture experiments, 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. These creep-rupture experiments have been conducted to determine the sensitivity of seam time-to-failure under creep loading to various variables associated with seam fabrication and environmental exposure.

The results of the creep-rupture experiments afford recommendations for the selection and application of seams such that factors promoting longer times-to-failure are emphasized during seam fabrication. Conversely, those factors that result in reduced times-to-failure are to be avoided. In this regard, past NIST studies [13,17] have found that, for butyl-based liquid adhesives, thickness along with rubber surface cleanness play a major role in extending the creep

lives of seams. This finding provides strong technical evidence that relatively thick adhesive layers need to be applied in the field when EPDM seams are formed.

Another important finding of these studies [13,17] was that increased creep-resistance of the liquid adhesive specimens due to thick adhesive layers and clean rubber surfaces could not be predicted based on short-term strength tests. Consequently, it was concluded that creep-rupture tests are more sensitive to factors that may affect the field performance of seams than short-term strength tests, and that creep testing should be a part of any methodology that evaluates the performance of seams [17]. These findings gave, in part, impetus to the present study, as the sensitivity of the creep-resistance of tape-bonded seam specimens to factors such as load, rubber surface condition, and tape thickness has not been reported.

1.2 Use of Tape Adhesive Systems for EPDM Seams

Traditionally, liquid adhesives have been the most common bonding agents for EPDM seams [6]. Although not employed extensively, some tape systems have also been used for many years. Dupuis [8] has provided a review of the history of EPDM tape systems. In recent years, the industry has seen an increase in their use. For example, a 1994 survey conducted by a trade publication indicated that the number of contractors using tape systems increased by 25 percent from 1992 to 1994 [7]. This trend is expected to continue. Hatgas and Spector [9] have listed reasons contributing to the increased use including: a reduction in the amounts of volatile organic compounds (VOCs) released during seam fabrication, ease of application and decreased application time, and the availability of an adhesive system that has uniform properties such as width and thickness.

The limited experience with current tape systems has shown that performance has been generally satisfactory [18]. Nevertheless, some roofing contractors and consultants have expressed concern that these tape systems are being used in increased quantities without sufficient independent evaluation. Consequently, they have urged that independent studies of the performance of tape-bonded seams be conducted.

1.3 Joint Industry-Government Research Project on Tape Seams

In response to the need for nonproprietary data on tape-bonded seam performance, three EPDM membrane manufacturers, two tape-system manufacturers, and two trade associations have undertaken a joint research project with the National Institute of Standards and Technology (NIST) through a Cooperative Research and Development Agreement (CRADA). The industrial CRADA members are Adco, Ashland, Carlisle SynTec, Firestone, GenFlex, the National Roofing Contractors Association (NRCA), and the Roof Consultants Institute (RCI). The U.S. Army Construction Engineering Research Laboratories (CERL) is also a sponsor. The objective of the study is to compare the performance of tape-bonded and liquid-adhesive-bonded EPDM seams, and to develop a test protocol based on creep testing and recommended criteria for evaluating the performance of tape-bonded seams. The experimental program consists of three 1-year long phases. Phase I is completed and Phase II is underway. Phase III will be considered for implementation near the end of Phase II. In brief, the following was planned:

- In Phase I, the creep-rupture response (time-to-failure) of tape-bonded seam specimens subjected to various peel loads under ambient conditions was compared to that of liquid-adhesive-bonded specimens.
- In Phase II, the creep-rupture response of tape-bonded seam specimens is being investigated under ambient conditions as a function of specimen-application variables such as the presence of primer, rubber surface cleanliness, pressure, application temperature, and tape thickness.
- In Phase III, it is expected that the creep-rupture response of tape-bonded seam specimens will be investigated as a function of test temperature and type of loading (i.e., peel versus shear).

Concurrent with the laboratory experimentation, field inspections of EPDM roofing systems having tape-bonded seams are being conducted and seam samples are being obtained. Mechanical properties of these field-seam specimens will be determined and compared with those of liquid-adhesive-bonded seams removed from roofs in previous studies.

1.4 Objective of this Report

This report presents the results of the experimentation comparing the creep-rupture response of tape-bonded and liquid-adhesive-bonded seam specimens as a function of peel load. These results may be used as a basic reference point against which the results of future creep-rupture experiments on EPDM seam specimens may be compared. In the present study, seam specimens were prepared using two tape systems and one liquid adhesive. The short-term peel strengths of the specimens were measured, and the times-to-failure were determined 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 clearly indicate that, in general, the tape-bonded specimens had times-to-failure that were comparable to, or were greater than, those of the liquid-adhesive-bonded specimens.

2. EXPERIMENTAL

2.1 Seam Specimen Preparation and Replicate Specimen Sets

Two commercial tape systems comprised of a tape and primer (designated Tape System 1 or TS1, and Tape System 2 or TS2) and a commercial butyl-based liquid adhesive (designated LA) were used. This liquid adhesive cures through a moisture-induced reaction. T-peel 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 EPDM sheet. The specimen preparation procedures have been previously described [17,18]. In all cases, the surface of the EPDM rubber was well cleaned [17]. For the tape systems, primer was applied at a rate recommended by their manufacturers using a drawdown blade technique.* Before testing, the thickness of the adhesive for each specimen (tape-bonded and liquid-adhesive-bonded) was measured according to techniques described in Rossiter et al. [17]. All specimens had a minimum age of 28 days when tested. Previous studies [13,18] have shown that this waiting period is sufficient for both tape-bonded and liquid-adhesive-bonded specimens to attain constant strength.

Replicate sets of specimens (i.e., different batches) were prepared at different times to investigate the reproducibility of the peel-strength and creep-rupture data. Two replicate sets of Tape System 1 specimens, four replicate sets of Tape System 2 specimens, and five replicate sets of liquid-adhesive-bonded specimens were included in the Phase I study (Table 1). A replicate set generally contained between 80 and 100 specimens from which those subjected to the peel-strength and creep-rupture tests were randomly selected. Although it was planned to use exactly the same materials (i.e., tapes, primers, or adhesives) in preparing all replicate sets, practical limitations associated with the shelf-lives of primers and adhesives precluded this possibility (see comments in Table 1).

In the case of Tape System 1, no differences existed between the two replicate sets. The same roll of tape and can of primer (designated TS1-1) was used to prepare both sets of specimens. In the case of Tape System 2, the same roll of tape, but three different cans of primers (designated TS2-1, TS2-2, and TS2-3) were used for the four replicate sets. The TS2 Replicate Sets Nos. 1 and 2 were prepared using the same can of primer. Examination of the TS2-1 primer after testing some of the TS2 Replicate Set No. 2 specimens showed that the primer's shelf life had probably reached its limit when these specimens were prepared -- the primer had jelled in the can. However, no evidence of potential jelling was apparent at the time the primer was used. Because of the jelling, a second can of the primer was used to prepare the TS2 Replicate Set No. 3 specimens.

At the time when this third replicate set was being prepared, it was brought to NIST's attention that the formulation of the Tape System 2 primer had been changed, and that the one used to prepare 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

*This technique uses an adjustable knife blade (i.e., the drawdown blade), bar, or rod to control distribution of the adhesive on the substrate [19]. The adhesive thickness is controlled by the distance between the blade edge and the substrate surface.

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 % solids, whereas TS2-3 had 5 % solids. It is noted that the specimens of all four Tape System 2 replicate sets were prepared with the same volume of primer using the drawdown technique.

In the case of the liquid-adhesive-bonded specimens, the major difference between the 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 each used a different can of adhesive; there was reportedly no difference in formulation. LA Replicate Set No. 3 was prepared from the same can used for LA Replicate Set No. 2. LA Replicate Set No. 5 used the same can as LA Replicate Set No. 4, but the former specimens were prepared by a liquid adhesive manufacturer's representative and not by a NIST research staff member. Reasons for NIST not preparing these specimens are discussed later in the report (see Section 3.2.2).

Table 1. Replicate sets of test specimens

Adhesive System ^a	Rep. No. ^b	Primer Designation	Adhesive Designation	Comment ^c
TS1	1	TS1-1	NA ^e	<ul style="list-style-type: none"> • First can of primer used for the first time. • First can of primer used for a second time.
	2	TS1-1	NA	
TS2	1	TS2-1	NA	<ul style="list-style-type: none"> • First can of primer (10% solids) used for the first time. • First can of primer (10% solids) used for a second time. • Second can of primer (10% solids) used for the first time. • Third can of primer (5% solids) used for the first time; the primer having the 10% solids content was no longer available.
	2	TS2-1	NA	
	3	TS2-2	NA	
	4	TS2-3	NA	
LA	1	NA	LA-1	<ul style="list-style-type: none"> • First can of adhesive used for the first time. • Second can of adhesive used for the first time. • Second can of adhesive used for the second time. • Third can of adhesive used for the first time. • Third can of adhesive used for the first time; that is, LA Replicate Set Nos. 4 and 5 were fabricated at the same time.
	2	NA	LA-2	
	3	NA	LA-2	
	4	NA	LA-3	
	5	NA	LA-3	

^aTS1, TS2, and LA indicate Tape System 1, Tape System 2, and Liquid Adhesive, respectively.

^bRep. No. indicates the replicate set number.

^cAll specimens were prepared by NIST research staff with the exception of the liquid adhesive (LA) Replicate Set No. 5.

^dNA indicates not applicable.

2.2 Peel-Strength Tests

For each replicate set, four T-peel strength tests were conducted at room temperature, 23 °C ± 2 °C (73 °F ± 4 °F), at a 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.

2.3 Creep-Rupture Tests

A minimum of eight specimens from each replicate set was included in the creep-rupture investigation. The tests were conducted in peel at room temperature, $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ($73\text{ }^{\circ}\text{F} \pm 4\text{ }^{\circ}\text{F}$), in laboratory-constructed chambers according to the general procedure described in Martin et al. [13]. The relative humidity in the chambers was maintained between 40 % to 45 % using a saturated potassium carbonate solution [20]. Built-in fans gently circulated the air in the chambers. The relative humidity in each chamber was checked using a Labcraft Digital Hygrometer, Model Number 244-354.**

Specimens were conditioned for a minimum of 16 hours in the chambers before applying the load. As indicated, the loads ranged 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 25 mm (1 in) wide specimen having a 2.5 kN/m (14 lbf/in) peel strength, which was essentially the maximum strength measured for a Tape System 2 specimen (Table 2). For a test in a given chamber, all specimens were loaded simultaneously. The times-to-failure (i.e., time under load until which the two rubber strips comprising the specimens completely separated) were recorded (± 1 s) electronically for each specimen using a computerized monitoring and data-logging system

**Certain company products are mentioned in the text to specify adequately the experimental procedure and equipment used. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment is necessarily the best available for the purpose.

3. RESULTS AND DISCUSSION

3.1 Peel-Strength Results

Short-term peel strength measurements were conducted as a quality check for determining if a replicate 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 replicate set prepared. Table 2 summarizes the peel strength data including a description of the major failure mode observed during testing. With the exception of TS2 Replicate Set No. 2, all specimens failed cohesively. Adhesive failure of TS2 Replicate Set No. 2 was attributed to the use of primer that had reached the limit of its shelf life, as discussed in Section 2.1.

Table 2. Short-term peel strength

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

^aThe 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).

^bAverage of four measurements.

^csd indicates standard deviation.

^dCoV indicates coefficient of variation.

^eThe adhesive failure of these specimens was attributed to fabricating the specimens with primer whose shelf life had probably been reached.

For both the tape-bonded and liquid-adhesive-bonded specimens, the strength values were generally comparable to those cited in the literature. In the case of Tape System 1, the average strengths for the two replicate sets were 1.91 kN/m and 1.81 kN/m (10.9 lbf/in and 10.4 lbf/in), which compared favorably with a previously reported value of 1.8 kN/m (10.4 lbf/in) [18]. In the case of Tape System 2, the average strengths ranged from 2.07 kN/m to 2.40 kN/m (11.8 lbf/in to 13.7 lbf/in), which bracketed a previously reported value of 2.2 kN/m (12.6 lbf/in) [18]. The TS2 Replicate Set No.2 specimens, which had the lowest average value of 2.07 kN/m (11.8 lbf/in), failed adhesively. 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 still performed. Finally, in the case of the liquid-adhesive-bonded specimens, the average peel strength ranged from

1.81 kN/m to 1.94 kN/m (10.3 lbf/in to 11.1 lbf/in). This range was comparable to, if not slightly greater than, values ranging from 1.5 kN/m to 1.8 kN/m (8.7 lbf/in to 10.3 lbf/in) given in the literature [13,16,17] for specimens made with butyl-based adhesive applied to well cleaned EPDM.

3.2 Creep-Rupture Results

Table 3 summarizes the number of observed times-to-failure recorded as a function of adhesive system and load. In all, 601 time-to-failure data points were registered for the three adhesive systems tested at the eight loads ranging from 3.1 N to 24.9 N (0.7 lbf to 5.6 lbf). No specimens failed when tested at 3.1 N (0.7 lbf) and, thus, Table 3 does not include reference to this load. At the time of writing this report, the tests at 3.1 N (0.7 lbf) were ongoing, and the specimens had been under load for over 8600 hours without failure.

Table 3. Number of times-to-failure observed during the study

Adhesive System ^a	Rep. No.	Age days ^b	Load, N (lbf)						
			6.2 (1.4)	9.3 (2.1)	12.5 (2.8)	15.6 (3.5)	18.7 (4.2)	21.8 (4.9)	24.9 (5.6)
TS1 (TS1-1) ^c	1	107	14	8	8	8	8	8	8
TS1 (TS1-1)	2	28	8	8	8	8	8	8	8
TS2 (TS2-1) ^c	1	107	14	8	8	8	8	6	8
TS2 (TS2-1)	2	28	8	8	8	8	8	8	7
TS2 (TS2-2)	3	33	8	6	5	8	8	8	8
TS2 (TS2-3)	4	28	7	8	8	8	8	8	8
LA (LA-1) ^c	1	107	NF ^d	8	8	8	8	8	8
LA (LA-2)	2	28	-- ^e	8	8	8	8	8	8
LA (LA-2)	3	82	8	8	8	8	8	8	8
LA (LA-3)	4	28	8	8	8	8	8	8	7
LA (LA-3)	5	29	8	8	8	8	8	8	8

^aThe 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).

^bAge of the specimens when the creep tests were initiated.

^cFourteen (14) specimens from this set were subjected to creep loading at 3.1 N (0.7 lbf). At the time of writing this report, the tests were ongoing and these specimens had been under load for over 8600 hours without failure.

^dNF indicates no failures up to the time of writing this report.

^eTest not conducted.

The experimental design planned for testing eight specimens per adhesive system per load for load values from 9.3 N to 24.9 N (2.1 lbf to 5.6 lbf), and for testing 14 specimens per adhesive system per load for the two lowest load values of 3.1 N and 6.2 N (0.7 lbf and 1.4 lbf). More specimens were to be tested at the lower loads in the event that the times-to-failure were relatively long, resulting in censoring some of the specimens in the set (i.e., terminating the test before all specimens failed). The experimental plan was, with some exception, generally followed. For those tests where less than eight data points are noted in Table 3, an occasional problem with the

data acquisition system prevented registration of the times-to-failure. Also, for the tests at the 6.2 N (1.4 lbf) load, only the initial specimen sets for all three adhesive systems consisted of 14 specimens. The time-to-failure data obtained as the study progressed indicated that, for additional tests at this load, eight specimens in a set would be sufficient.

Table 4 provides a summary of the time-to-failure data. Appendix A gives the time-to-failure for each specimen along with the thickness of the adhesive layer. In Table 4, for each replicate set per adhesive system per load, the minimum, maximum, and mean times-to-failure are given along with the coefficient of variation (CoV) of the mean and the dominant failure mode experienced during delamination. 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.

About three quarters of the coefficients of variation were less than 20 percent. Values greater than 20 percent were observed only for Tape System 2 and the liquid adhesive. Although considerable scatter was present in the individual replicate sets, coefficient of variation values of 20 percent or less are, nevertheless, relatively low for a creep-rupture experiment of EPDM seam specimens. Past NIST data on the creep-resistance of four sets of seam specimens prepared with butyl-based liquid adhesive and cleaned EPDM rubber gave coefficients of variation ranging from 21 percent to 40 percent [13]. Appendix B discusses further the variability within the replicate data sets.

3.2.1 Statistical Analysis. As noted in the introduction, a primary objective of the Phase I study is 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 due to load and adhesive system. 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-a-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:

$$\ln(TTF) = b_0 + b_1L + b_2e^{b_3L} \quad (1)$$

where TTF denotes 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 [21] to model fatigue in metals. The function was fit by nonlinear least squares; the estimated coefficients and standard deviations of the coefficients are given in Tables 5A and 5B along with the residual standard deviations. 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

Table 4. Summary of the creep-rupture data

Adhesive System ^a	Load N (lbf)	Rep. No.	Time-to-Failure, hours				Dominant Failure Mode
			Min.	Max.	Mean	CoV, %	
TS1 (TS1-1)	24.9 (5.6)	1	0.58	0.67	0.61	4.2	Cohesive
TS1 (TS1-1)		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 (4.9)	1	0.83	1.08	1.00	7.9	Cohesive
TS1 (TS1-1)		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 (4.2)	1	1.54	1.85	1.67	6.6	Cohesive
TS1 (TS1-1)		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 (3.5)	1	2.67	3.59	3.19	9.5	Cohesive
TS1 (TS1-1)		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	

^aThe 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 4. Summary of the creep data (cont.)

Adhesive Type ^a	Load N (lbf)	Rep. No.	Time-to-Failure, hours				Dominant Failure Mode
			Min.	Max.	Mean	CoV, %	
TS1 (TS1-1)	12.5 (2.8)	1	5.68	10.54	7.07	13.6	Cohesive
TS1 (TS1-1)		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 (2.1)	1	23.09	33.86	28.51	13.6	Cohesive
TS1 (TS1-1)		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 (1.4)	1	180.9	321.4	237.2	18.2	Cohesive
TS1 (TS1-1)		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)		2	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 (0.7)	1	NF	----	----	----	----
TS2 (TS2-1)		1	NF	----	----	----	----
LA (LA-1)		1	NF	----	----	----	----

^aThe 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).

^bNF indicates no failure; the elapsed time when this report was issued was over 8600 hours.

Table 5A. Coefficients for Bastenaire's function fit to the mean time-to-failure data; the coefficients are based on load in N

Adhesive System ^a	Rep. No.	Coefficients ^b				rsd ^c
		b_0	b_1	b_2	b_3	
TS1 (TS1-1)	1	2.6219 (0.3822)	-0.1272 (0.0150)	13.6944 (0.8513)	-0.2125 (0.0208)	0.0395
TS1 (TS1-1)	2	1.6914 (1.5806)	-0.0938 (0.0536)	11.5375 (0.6924)	-0.1470 (0.0383)	0.0761
TS2 (TS2-1)	1	3.7744 (0.4490)	-0.1141 (0.0174)	12.0934 (0.8059)	-0.2035 (0.0244)	0.0427
TS2 (TS2-1)	2	4.5430 (0.2418)	-0.1691 (0.0105)	17.5443 (2.2621)	-0.2921 (0.0288)	0.0434
TS2 (TS2-2)	3	4.4108 (0.7474)	-0.1444 (0.0300)	11.9009 (2.2403)	-0.2262 (0.0564)	0.0869
TS2 (TS2-3)	4	4.7465 (0.3324)	-0.1484 (0.0141)	15.6649 (2.2480)	-0.2712 (0.0343)	0.0530
LA (LA-1)	1	-1.5144 (1.1524)	0 (0)	17.5047 (1.8328)	-0.0874 (0.0237)	0.2982
LA (LA-2)	2	3.0208 (0.6962)	-0.1289 (0.0244)	13.7938 (1.8128)	-0.1805 (0.0325)	0.0282
LA (LA-2)	3	2.6714 (0.3220)	-0.1354 (0.0143)	10.9500 (4.5607)	-0.3211 (0.0871)	0.0665
LA (LA-3)	4	2.3593 (0.2171)	-0.1048 (0.0095)	9.7595 (2.4758)	-0.3057 (0.0548)	0.0417
LA (LA-3)	5	3.3209 (0.0701)	-0.1381 (0.0035)	83.6798 (53.1306)	-0.6822 (0.1066)	0.0293

^aThe 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).

^bValues in parentheses are the standard deviations of the estimated coefficients.

^cThis 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 5B. Coefficients for Bastenaire's function fit to the mean time-to-failure data; the coefficients are based on load in lbf

Adhesive System ^a	Rep. No.	Coefficients ^b				rsd ^c
		b ₀	b ₁	b ₂	b ₃	
TS1 (TS1-1)	1	2.6219 (0.3822)	-0.5657 (0.0669)	13.6944 (0.8513)	-0.9452 (0.0925)	0.0395
TS1 (TS1-1)	2	1.6914 (1.5806)	-0.4173 (0.2385)	11.5375 (0.6924)	-0.6538 (0.1705)	0.0761
TS2 (TS2-1)	1	3.7744 (0.4490)	-0.5077 (0.0774)	12.0934 (0.8059)	-0.9050 (0.1084)	0.0427
TS2 (TS2-1)	2	4.5430 (0.2418)	-0.7524 (0.0467)	17.5443 (2.2621)	-1.2995 (0.1282)	0.0434
TS2 (TS2-2)	3	4.4108 (0.7474)	-0.6424 (0.1336)	11.9009 (2.2403)	-1.0060 (0.2510)	0.0869
TS2 (TS2-3)	4	4.7465 (0.3324)	-0.6602 (0.0629)	15.6649 (2.2480)	-1.2063 (0.1524)	0.0530
LA (LA-1)	1	-1.5144 (1.1524)	0.0000 (0.0000)	17.5047 (1.8328)	-0.3886 (0.1056)	0.2982
LA (LA-2)	2	3.0208 (0.6962)	-0.5743 (0.1085)	13.7938 (1.8128)	-0.8029 (0.1445)	0.0282
LA (LA-2)	3	2.6714 (0.3220)	-0.6022 (0.0637)	10.9500 (4.5607)	-1.4285 (0.3874)	0.0665
LA (LA-3)	4	2.3593 (0.2171)	-0.4660 (0.0424)	9.7595 (2.4758)	-1.3597 (0.2439)	0.0417
LA (LA-3)	5	3.3209 (0.0701)	-0.6141 (0.0157)	83.6798 (53.1306)	-3.0344 (0.4744)	0.0293

^aThe 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).

^bValues in parentheses are the standard deviations of the estimated coefficients.

^cThis 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.

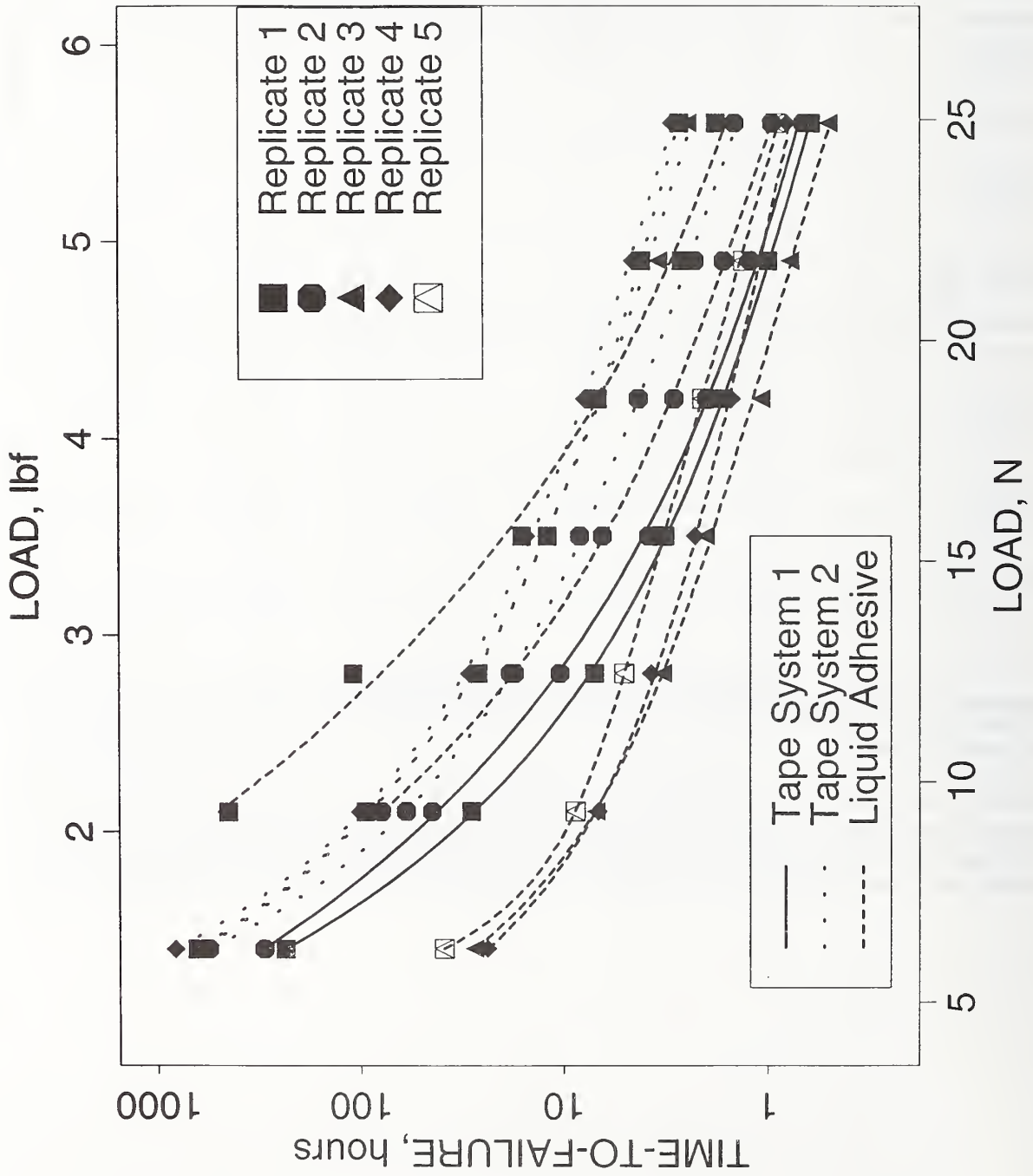


Figure 1. $\ln(\text{mean time-to-failure})$ as a Function of Load; the Data Were Fitted With the Model:
 $\ln(\text{mean TTF}) = b_0 + b_1 \cdot \text{Load} + b_2 \exp(b_3 \cdot \text{Load})$.

linear in load. The nonlinear curves fit to each adhesive-replicate combination will be discussed below. Note that despite the considerable variability in the individual failure times (as evidenced by CoVs in Table 4), the mean times-to-failure appear to be smooth functions of load. That is, the data fall on or are close to the fitted curves for all replicate sets. Note also that the relationship between time-to-failure and load is relatively linear at the higher loads and nonlinear at the lower loads.

Another model that has been often used for relating time-to-failure to load [13] is the power-law model:

$$\ln(TTF) = c_0 + c_1 \ln(Load) \quad (2)$$

If eq (2) is adequate for modeling time-to-failure as a function of load, then the data points in a plot of $\ln(TTF)$ against $\ln(Load)$ should fall on nearly straight lines. Figure 2 provides such a plot for the mean times-to-failure of the 11 replicate data sets. The model was seen to fit the data reasonably well, but it was unable to represent the apparent nonlinearity at the lower loads. Note in Figure 2 that, at the lowest load, 6.2 N (1.4 lbf), the fitted lines underestimate the mean times-to-failure. In contrast, using the Bastenaire model, the fitted curves for all replicate data sets intersect with the 6.2 N (1.4 lbf) mean times-to-failure values. Thus, for the data in this study, eq (1) was considered to be a more appropriate model than eq (2), and the discussions to follow are based on the eq-(1) fits.

Figure 1 provides the basis for discussion of the comparative performance of the tape-bonded and liquid-adhesive-bonded seam specimens. In this figure, the line type represents the adhesive system, and the plot character for the mean times-to-failure represents the replicate set number (see legends on the plot). 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 specimens were generally comparable to, or greater than, those of the liquid-adhesive-bonded specimens. This was particularly the case at the lower loads, for example, 6.2 and 9.3 N (1.4 and 2.1 lbf), which may be 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 % of their short-term peel strength) for any considerable period of time [13,14]. Although the data in Figure 1 are from a laboratory experiment conducted under well controlled conditions, qualitatively the findings should be applicable to field experience. With other factors being equal (e.g., rubber surface condition, magnitude of the load, and workmanship), seams well fabricated 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. Environmental and application factors that may affect creep performance of the tape systems will be addressed in Phase II of this joint industry-government project.

Figure 1 can also be used to provide an estimate of the reproducibility of the time-to-failure data between replicate sets of specimens. For a given adhesive system, the closer is the grouping of fitted curves, then the less variability between replicate sets. It is quite apparent in Figure 1 that

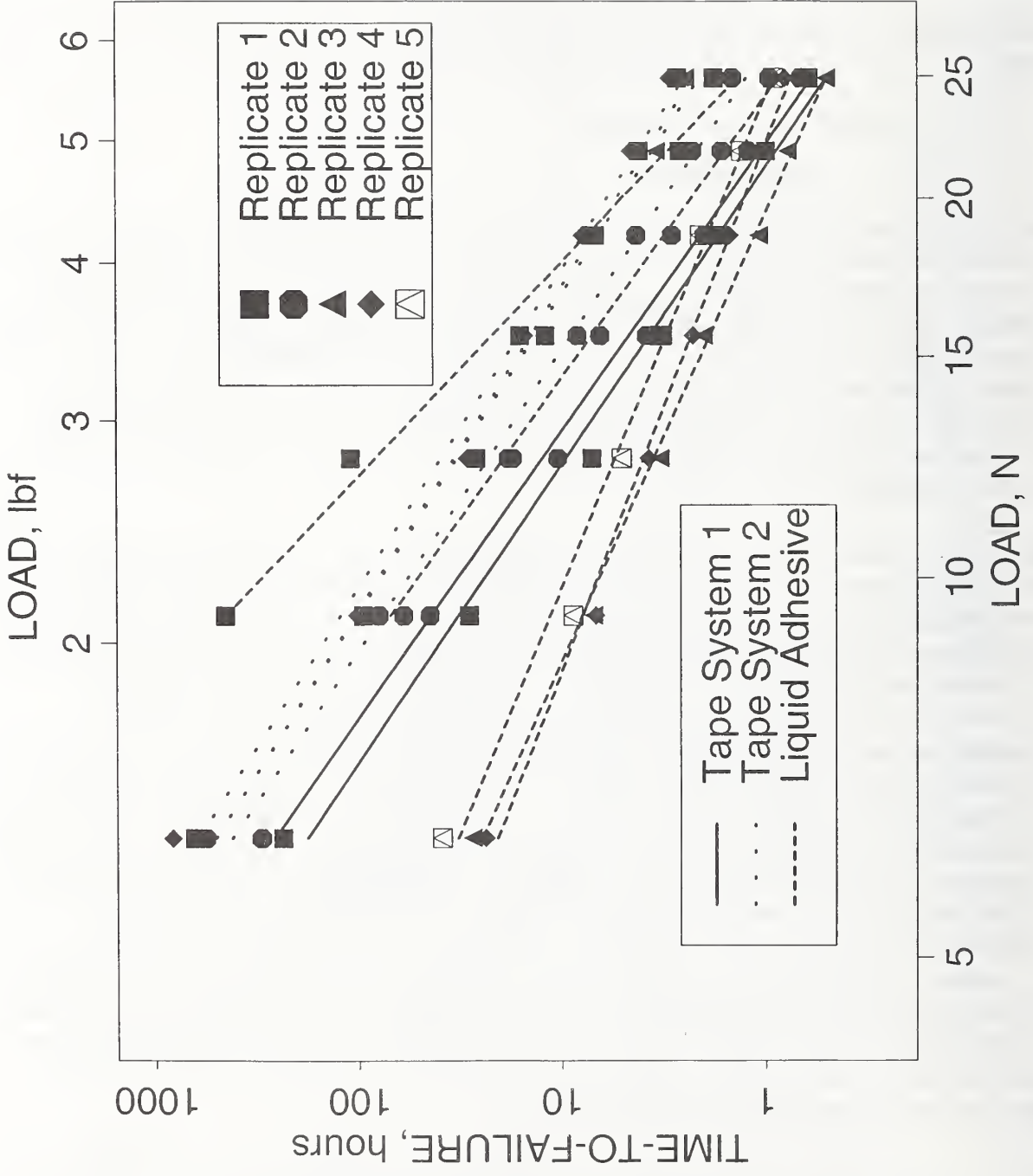


Figure 2. $\ln(\text{mean time-to-failure})$ as a Function of Load; the Data Were Fitted With the Model: $\ln(\text{mean TTF}) = c_0 + c_1 \cdot \ln(\text{Load})$.

the liquid-adhesive-bonded specimens displayed the least reproducibility. 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 specimen sets. A consequence of this wide variability is that, under certain conditions, the liquid adhesive can provide seam specimens which display substantially longer creep lifetimes than either other liquid-adhesive-bonded specimens or tape-bonded specimens. However, the conditions which produced the relatively long-lived LA Replicate Set No.1 specimens are not known and, hence, not predictably reproducible. Section 3.2.2 discusses in more detail the variability between liquid-adhesive-bonded replicate sets.

In comparison to the liquid adhesive, the tape systems gave more reproducible results. This may be because the tapes are factory-made products and not subject to some of the non-controllable 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 (fig. 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 with primer that had jelled 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. The practical lesson learned is to avoid primers whose shelf-lives may be in doubt to avert fabrication of seams that can have reduced creep resistance.

As a final comment on the reproducibility of the Tape System 2, note that TS2 Replicate Set No. 4 showed times-to-failure quite akin to those of TS2 Replicate Sets Nos. 1 and 3 (fig. 3). This suggested that the solids content of the two primers used for Tape System 2 (5 % for Replicate Set No. 4, and 10 % 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 Tape System 1 results, Figure 1 shows that the two curves for this system were close to each other, but at no point did they overlap. TS1 Replicate Set No. 2 always had average 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 Tape System 1 were tested.

Because Figure 1 provided only a qualitative analysis of the reproducibility of the data between replicate sets of a given adhesive system, further analysis was undertaken. To this end, special plots were prepared for each replicate data set for each of the seven loads. Figure 3 is an example of such a plot for tests conducted at 9.3 N (2.1 lbf). The other six plots were similar and are not shown. Figure 3 provides, for each replicate data set, the individual times-to-failure (small circular plot character), the average times-to-failure (large circular plot character), and uncertainty bars representing two standard deviation limits on the means. In cases where the uncertainty bars

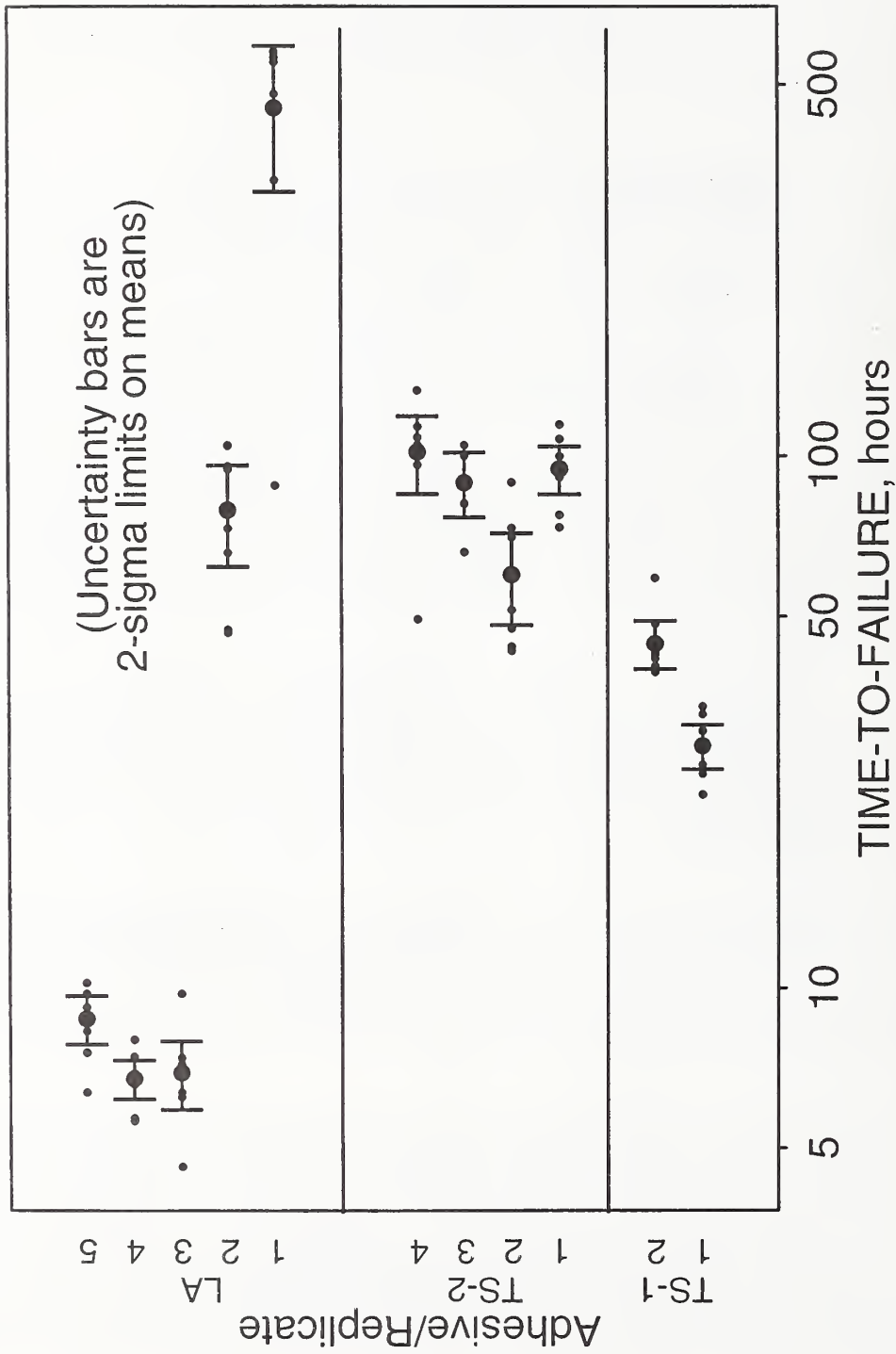


Figure 3. Reproducibility of the Time-to-Failure Data at the 9.3 N (2.1 lbf) Load for Each Adhesive System.

overlapped between replicate data sets, it was concluded that no statistically significant difference between data sets existed.

The wide variability between liquid adhesive data sets is clearly evident in Figure 3, although LA Replicate Sets Nos. 3, 4, and 5 were comparable to each other. TS2 Replicate Sets Nos. 1, 3, and 4 were not statistically significantly different from each other, whereas TS2 Replicate Set No. 2 was statistically significantly different from any of the other three. For Tape System 1, the difference between the two replicate sets was statistically significant. However, as previously mentioned, no practical importance was attached to the statistically significant differences in the case of either tape system.

3.2.2 Variability Between 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. That is why five replicate sets were included in the study -- the variability initially observed between LA Replicate Sets Nos. 1 and 2 warranted further testing. However, an expanded investigation to explain the observed variability of the liquid adhesive was beyond the scope of the study.

Because the specimens in LA Replicate Sets Nos. 1 and 2 were prepared with different cans of adhesive (Table 1), a possible explanation for the variability in the creep results was that the adhesives were different. As a limited test of this possibility, LA Replicate Set No. 3 specimens were fabricated using the same can of adhesive as used for preparing the LA Replicate Set No. 2 specimens. About 2½ months elapsed between preparation of these two sets. As evidenced in Figure 1, the times-to-failure of the LA Replicate Set No. 3 specimens were considerably less than those of the LA Replicate Set No. 2 specimens. This implied that the observed non-reproducibility of the liquid-adhesive-bonded sets may not be associated with the adhesive although, in the case of the difference between LA Replicate Sets Nos. 2 and 3, it was not experimentally ruled out that the adhesive had undergone some unknown change in the can. However, past NIST experience with the liquid adhesive has not given rise to any evidence that unwanted changes occur over a few months time when the adhesive is well sealed.

Another possible reason for the variability in the creep-rupture results of LA Replicate Sets Nos. 1 and 2 was the age of the specimens when subjected to creep testing. Note in Table 3 that the minimum age of these two replicate sets were 107 days and 28 days, respectively, when the creep tests were initiated. To examine the influence of specimen age preliminarily, eight specimens of LA Replicate Sets Nos. 4 and 5^{***} were tested at 15.6 N (3.5 lbf) when they were 174 days old. The original creep tests of these replicate sets were conducted when the two had minimum ages of 28 and 29 days, respectively (Table 3). In the case of the LA Replicate Set No. 4, the younger and older specimens had average times-to-failure of 2.0 hours and 4.3 hours; that is, they differed by a factor of slightly more than 2. In the case of the LA Replicate Set No. 5, the younger and older specimens had average times-to-failure of 5.3 hours and 3.3 hours; that is, they differed by a factor of about 1.6. These limited observations suggested that specimen age might have some

^{***} LA Replicate Sets Nos. 4 and 5 were used because of the availability of specimens.

effect on time-to-failure, but the magnitude of the effect in this one case was considerably less than the difference between LA Replicate Sets Nos. 1 and 2 (fig. 1).

After conducting the creep-rupture tests of LA Replicate Sets Nos. 1 and 2, examination of the delaminated specimens indicated that the failure was cohesive in both cases. However, a subtle difference in the visual appearances of the fractured adhesive surfaces of specimens from the two sets was apparent. LA Replicate Set No. 1 specimens had smoother surfaces, whereas LA Replicate Set No. 2 specimens had surfaces which might be described as pockmarked, cratered, or cellular. Moreover, the fracture of the LA Replicate Set No. 1 specimens seemed to have occurred more or less along the center plane of the adhesive layer. In contrast, the fracture of the LA Replicate Set No. 2 specimens seemed to have taken place closer to one of the EPDM rubber surfaces. The different images of the fractured adhesive layers suggested that, in the case of LA Replicate Set No. 2, their microstructure may have been somewhat porous or cellular, and the cells were ruptured during the creep-rupture delamination.**** In turn, it was considered that the open time (i.e., time interval between application of the adhesive on the rubber adherends and formation of the joint) or relative humidity conditions under which the specimens were prepared may have influenced the microstructure of the adhesive layers. The hypotheses, both of which involve the solvent included: (1) short open times did not allow sufficient evaporation of the solvent, or (2) high humidities affected the rate of the moisture-induced cure of the adhesive such that gaseous by-products of the reaction, or solvent, were trapped in the curing adhesive layer.

A 30-minute open time was used in preparing the LA Replicate Sets Nos. 1 and 2. This was consistent with past NIST experience [17,22] and considered adequate for the present study. Nevertheless, one limited experiment with specimens prepared using a 4-hour open time was conducted when the laboratory relative humidity was about 60 % (measured with a psychrometer). The results were comparable to LA Replicate Set No. 2; that is, no effect on time-to-failure and surface appearance was observed.

All specimens had been prepared in a laboratory where the relative humidity was not controlled. The specimens of LA Replicate Sets Nos. 1 and 2 were prepared in late December and early May, respectively, and the exact relative humidities were not known. When it was decided to prepare another set of specimens (LA Replicate Set No. 4) in further investigating the variability of the liquid adhesive, the relative humidity in the laboratory was about 60 %. This value was considered to be too high for specimen preparation in the event that high humidity was affecting the microstructure of the adhesive layer. At the time, the relative humidity in the liquid adhesive manufacturer's laboratory was about 40 %. Thus, NIST research staff prepared the LA Replicate Set No. 4 specimens in the manufacturer's laboratory using EPDM rubber and liquid adhesive from NIST. Additionally, because the opportunity presented itself to compare the creep-rupture results between NIST-made specimens and manufacturer-made specimens, a set of replicates (No. 5) was prepared by the manufacturer's research staff using the EPDM rubber and liquid adhesive from NIST. Whereas NIST staff used a drawdown technique to apply the adhesive and

**** Similar observations were made of the delaminated LA Replicate Set No. 3 specimens.

a press to exert pressure during specimen formation, the adhesive manufacturer employed a paint brush for adhesive application and a field roller for pressure application.

The results of the creep-rupture tests on LA Replicate Sets Nos. 4 and 5 were comparable to those of LA Replicate Set No. 3 and are included in Figure 1. No important difference in creep-performance between LA Replicate Sets Nos. 4 and 5 was observed, indicating little effect of the two different laboratory application methods. The manufacturer's specimens had thicker adhesive layers, about 23 mm to 25 mm (9 mil to 10 mil),^{*****} than those of the NIST specimens, which were about 18 mm to 20 mm (7 mil to 8 mil). This thickness difference may have accounted for the somewhat longer times-to-failure for the manufacturer-made specimens, as the creep-rupture life of butyl-based adhesive specimens is known to increase with an increase in adhesive layer thickness [13,17].

The surfaces of the fractured adhesive layers of the liquid-adhesive-bonded specimens from Replicate Sets Nos. 4 and 5 were seen to have a distinctly cellular appearance. It was similar to, if not more pronounced than, that observed for the delaminated specimens of LA Replicate Set No. 2. Scanning electron microscopy (SEM) observation of the fracture surfaces was conducted using a representative specimen from each of LA Replicate Sets Nos. 1, 4, and 5. Figure 4 gives micrographs at x10 magnification for the LA Replicate Sets Nos. 1 and 5 specimens. Micrographs of LA Replicate Set No. 4 were similar to that of LA Replicate Set No. 5. The micrographs in Figure 4 show sections of the fractured adhesive surfaces on the two corresponding (i.e., mating) EPDM rubber strips of the delaminated specimens.

The SEM photos clearly show that the microstructures of the fractured adhesive surfaces of the two specimens are distinctly different. The LA Replicate Set No. 1 specimen had an adhesive layer that was generally solid, although some voids were visible. Also, the appearance of the two strips showed no evidence that more adhesive was present on one rubber strip than on the other; i.e., the fracture may have occurred somewhat along the center plane of the adhesive layer. In contrast, the LA Replicate Set No. 5 specimen was quite cellular (or honeycombed). In addition, more adhesive appeared to be present on one rubber strip, as evidenced by the depth of the "cells" on one side versus another. Figure 5 presents further evidence of the difference between the microstructures of the two specimens. Here, micrographs at x25 magnification highlight the relatively solid adhesive layer of the LA Replicate Set No. 1 specimen in comparison to the highly cellular adhesive layer of the LA Replicate Set No. 5 specimen.

Factors contributing to, or preventing, the formation of the cellular microstructure of the liquid adhesive layer were not investigated beyond the limited experimentation just described. Certainly, preparing the specimens at 40 % relative humidity did not prevent cell formation. The limited SEM observations coupled with the time-to-failure data are evidence that liquid adhesive layers with a cellular microstructure have significantly reduced creep lifetimes versus those that are relatively solid. An understanding of the factors responsible for the cellular microstructure of the liquid adhesive layer might suggest a need for guidelines for fabricating seams without the cells.

^{*****} 1 mil = 0.001 in

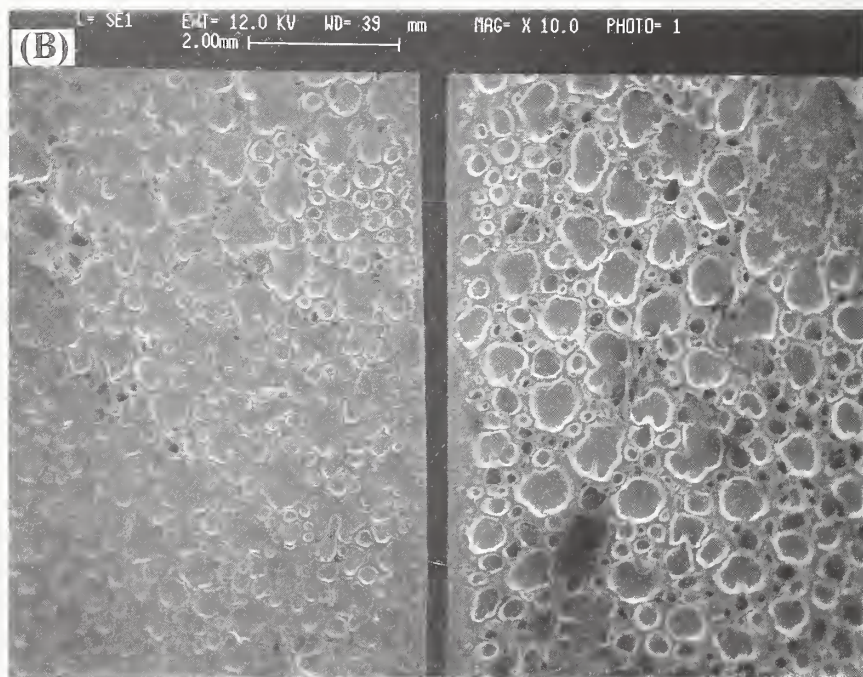
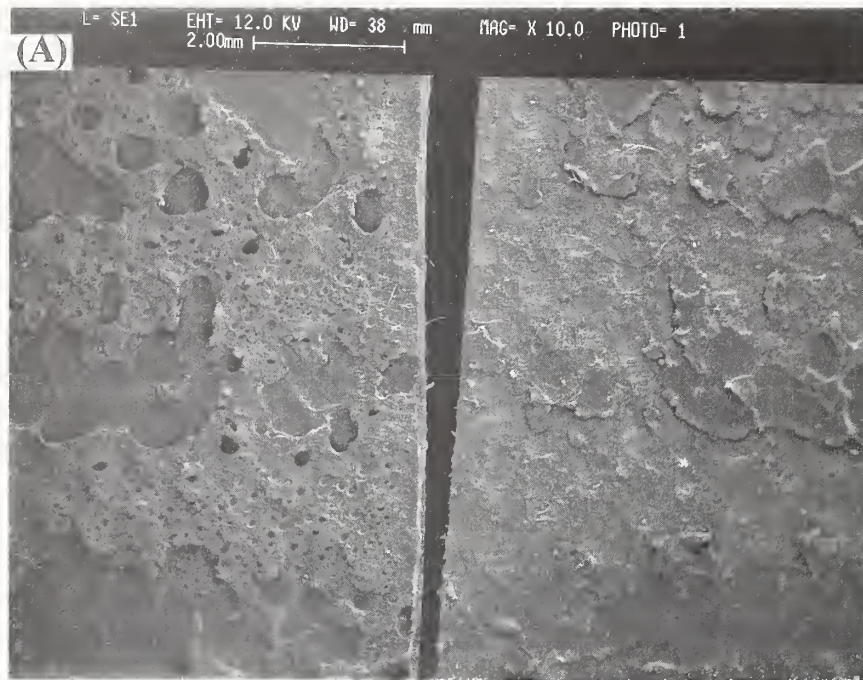


Figure 4. SEM Micrographs (x10 Magnification) of the Fracture Surfaces of Liquid-Adhesive-Bonded Specimens: (A) Specimen from LA Replicate Set No. 1 and (B) Specimen from LA Replicate Set No. 4.

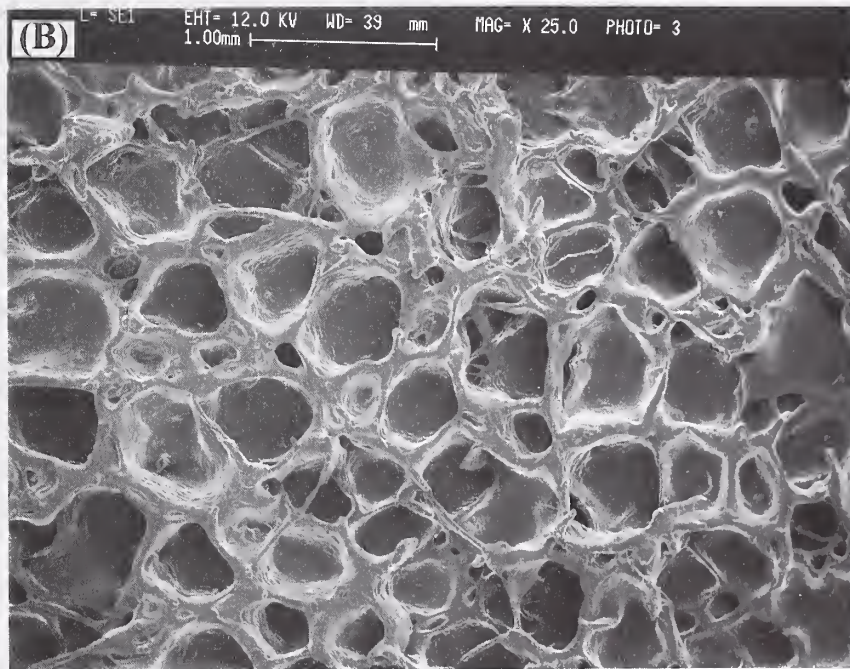


Figure 5. SEM Micrographs (x25 Magnification) of the Fracture Surfaces of Liquid-Adhesive-Bonded Specimens: (A) Specimen from LA Replicate Set No. 1 and (B) Specimen from LA Replicate Set No. 4.

It is of interest to note that, in contrast to the majority of the liquid-adhesive-bonded specimens (i.e., Replicate Sets Nos. 2 - 5), the fracture surfaces of the cohesively delaminated Tape System 1 and Tape System 2 specimens showed distinctly solid layers. No evidence of cellular microstructures were observed. These observations, which were made both by eye and light microscopy at about x25 magnification, were consistent with the fact that the tapes are solvent-free and are cured in the factory before seam fabrication.

3.3 Creep-Rupture Results Versus Peel-Strength Results

Past studies [13,17, 23] 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. 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 creep-rupture tests (fig. 1) showed wide variability. On the other hand, the results of the peel-strength tests (Table 2) were essentially constant. Table 6 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 average times-to-failure of the liquid-adhesive-bonded specimen sets differed by a factor of about 70. 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 tests of specimen strength did not detect the radically different load-sustaining capability of the different replicate sets of liquid-adhesive-bonded specimens. At the relatively high rates of fracture in the peel test, differences in the microstructure of the viscoelastic butyl-based liquid adhesive apparently had no effect. However, at the relatively low rates of fracture in the creep-rupture test, the response of the adhesive liquid was apparently affected by its microstructure.

Thus, for the liquid-adhesive-bonded specimens, this study has again provided evidence of the sensitivity of creep-rupture tests in comparison to peel-strength tests for evaluating factors that may be expected to affect seam field performance. And as a result, as recommended previously [17], it is again stated that creep-rupture testing should be an essential part of any methodology that evaluates the performance of seams. Consistent with this recommendation, one result of this joint industry-government research study will be a basis for the development of a protocol for conducting creep-rupture tests on tape-bonded seam specimens.

Table 6. Comparison of the times-to-failure at 9.3 N (2.1 lbf) and peel strengths of the three adhesive systems

Adhesive System ^a	Rep. No.	Creep-Rupture Results		Peel Strength Results	
		TTF hours	Difference Between Minimum and Maximum	Average kN/m (lbf/in)	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	506.6	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)	

^aThe designation in parenthesis refers to either the primer used for tape systems or the adhesive used for the liquid adhesive system (see Table 1).

4. SUMMARY AND CONCLUSIONS

Tape adhesive systems are being used in increasing quantities for preparing seams of EPDM roofing membranes. A joint industry-government research study has been initiated to develop nonproprietary data on tape-bonded seam performance. This paper has described the results of Phase I of the joint study comparing the creep-rupture response (i.e., time-to-failure) of tape-bonded seam specimens to that of liquid-adhesive-bonded seam specimens. Two commercial tape systems (i.e., tape and primer) and one liquid adhesive were applied to well-cleaned EPDM rubber in preparing the specimens. For all three systems, replicate sets of specimens were tested to determine the reproducibility of the measurements.

Before performing the creep tests, initial short-term T-peel measurements were conducted to assure that the peel strengths were typical of those of specimens prepared with these tape systems and liquid adhesive. In the creep-rupture experiments conducted at 23 °C (73 °F) and 40 % to 45 % relative humidity, specimens were subjected to peel loads ranging from 3.1 N to 24.9 N (0.7 lbf to 5.6 lbf). Times-to-failure were measured as a function of load. For each adhesive system, the data were found to be fitted well by a model relating $\ln(\text{mean time-to-failure})$ and load. Comparison of the fitted curves for the tape-bonded specimens vis-a-vis those for the liquid-adhesive-bonded specimens provided a basis for evaluating the relative creep-rupture response of the two types of bonding systems. Similarly, comparison of the fitted curves for the replicate data sets of each adhesive system gave a measure of the reproducibility of the creep-rupture data. The main conclusion, consistent with the objective of the study, was that:

- Specimens of the two tape-adhesive systems had times-to-failure that were in most cases comparable to, or greater than, those of the liquid adhesive. 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, $\ln(\text{mean TTF}) = b_0 + b_1 \cdot \text{Load} + b_2 \exp(b_3 \cdot \text{Load})$. This model was able to represent the nonlinear behavior of the times-to-failure at relatively low loads. Although often used to represent time-to-failure data as a function of load, the power law model, $\ln(\text{mean TTF}) = c_0 + c_1 \cdot \ln(\text{Load})$, was appropriate only at sufficiently large loads; it underestimated mean times-to-failure at the relatively low loads.
- Both 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. A consequence of this wide variability is that some liquid-adhesive-bonded specimens may have substantially longer times-to-failure than other liquid-adhesive-bonded specimens or tape-bonded specimens. However, until an understanding of the factors resulting in the fabrication of liquid-adhesive-bonded specimens having the relatively longer creep-rupture lives is attained, the preparation of such specimens is not predictably reproducible. It was observed, using scanning electron microscopy, that the fracture surfaces of liquid-adhesive-bonded specimens which gave relatively short times-to-failure had adhesive layers with distinctly cellular microstructures. Such microstructures were not found for the liquid-adhesive-bonded specimens having the longest times-to-failure. Conditions producing the cellular microstructures are not understood.

- Delaminated specimens of both tape systems displayed adhesive layers with microstructures which were not cellular.
- Both 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 seam performance. As indicated, specimens having adhesive layers with quite cellular microstructures had reduced times-to-failure in comparison with those having non-cellular microstructures. In contrast, the microstructure of the adhesive layer apparently had no effect on short-term peel strength. 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. DATA DEVELOPED IN THE STUDY

This appendix contains the time-to-failure (TTF) data for the Tape System 1 (Table A-1), Tape System 2 (Table A-2), and Liquid Adhesive (Table A-3) specimens as a function of load and replicate specimen set. The thickness of the adhesive layer is also given. The following codes are used in the tables:

<u>Column Title</u>	<u>Code</u>
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Adhesive System	1 = Tape System 1
	2 = Tape System 2
	3 = Liquid Adhesive

Failure Mode	1 = Cohesive
	2 = Adhesive

Table A-1. Data for tape system 1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
117	1	24.9	5.6	1	0.582	1.3	0.052	1
16	1	24.9	5.6	1	0.593	1.3	0.053	1
144	1	24.9	5.6	1	0.596	1.3	0.052	1
116	1	24.9	5.6	1	0.610	1.3	0.053	1
193	1	24.9	5.6	1	0.610	1.3	0.050	1
140	1	24.9	5.6	1	0.617	1.3	0.051	1
120	1	24.9	5.6	1	0.626	1.3	0.052	1
151	1	24.9	5.6	1	0.665	1.3	0.050	1
1204	1	24.9	5.6	2	0.617	1.2	0.047	1
1205	1	24.9	5.6	2	0.623	1.2	0.047	1
1234	1	24.9	5.6	2	0.632	1.2	0.047	1
1258	1	24.9	5.6	2	0.643	1.2	0.047	1
1255	1	24.9	5.6	2	0.663	1.2	0.046	1
1220	1	24.9	5.6	2	0.722	1.2	0.047	1
1209	1	24.9	5.6	2	0.727	1.2	0.049	1
1213	1	24.9	5.6	2	0.748	1.2	0.048	1
154	1	21.8	4.9	1	0.828	1.2	0.049	1
12	1	21.8	4.9	1	0.956	1.2	0.049	1
113	1	21.8	4.9	1	0.996	1.3	0.051	1
118	1	21.8	4.9	1	1.008	1.3	0.051	1
166	1	21.8	4.9	1	1.009	1.3	0.050	1
196	1	21.8	4.9	1	1.039	1.3	0.051	1
163	1	21.8	4.9	1	1.063	1.3	0.050	1
152	1	21.8	4.9	1	1.079	1.3	0.052	1
1224	1	21.8	4.9	2	1.144	1.2	0.049	1
1225	1	21.8	4.9	2	1.148	1.2	0.046	1
1237	1	21.8	4.9	2	1.170	1.2	0.046	1
1245	1	21.8	4.9	2	1.204	1.2	0.047	1
1272	1	21.8	4.9	2	1.218	1.2	0.047	1
1205	1	21.8	4.9	2	1.228	1.2	0.047	1
1202	1	21.8	4.9	2	1.289	1.2	0.047	1
1203	1	21.8	4.9	2	1.355	1.2	0.046	1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
115	1	18.7	4.2	1	1.541	1.3	0.051	1
126	1	18.7	4.2	1	1.566	1.3	0.052	1
11	1	18.7	4.2	1	1.606	1.3	0.052	1
114	1	18.7	4.2	1	1.621	1.3	0.051	1
1102	1	18.7	4.2	1	1.662	1.3	0.049	1
1100	1	18.7	4.2	1	1.710	1.3	0.051	1
186	1	18.7	4.2	1	1.793	1.3	0.051	1
149	1	18.7	4.2	1	1.854	1.3	0.052	1
1246	1	18.7	4.2	2	1.644	1.2	0.047	1
1275	1	18.7	4.2	2	1.896	1.2	0.049	1
1250	1	18.7	4.2	2	1.938	1.2	0.049	1
1276	1	18.7	4.2	2	2.001	1.3	0.050	1
1249	1	18.7	4.2	2	2.003	1.2	0.049	1
1254	1	18.7	4.2	2	2.056	1.2	0.049	1
1208	1	18.7	4.2	2	2.233	1.2	0.049	1
1201	1	18.7	4.2	2	2.240	1.2	0.049	1
183	1	15.6	3.5	1	2.673	1.3	0.051	1
170	1	15.6	3.5	1	3.036	1.3	0.053	1
175	1	15.6	3.5	1	3.057	1.3	0.050	1
1103	1	15.6	3.5	1	3.090	1.3	0.052	1
17	1	15.6	3.5	1	3.104	1.2	0.049	1
172	1	15.6	3.5	1	3.440	1.3	0.051	1
194	1	15.6	3.5	1	3.486	1.3	0.051	1
110	1	15.6	3.5	1	3.593	1.3	0.051	1
1211	1	15.6	3.5	2	3.342	1.2	0.047	1
1270	1	15.6	3.5	2	3.356	1.2	0.046	1
1262	1	15.6	3.5	2	3.618	1.1	0.045	1
1232	1	15.6	3.5	2	3.951	1.2	0.047	1
1228	1	15.6	3.5	2	3.962	1.2	0.047	1
1215	1	15.6	3.5	2	4.086	1.2	0.048	1
1227	1	15.6	3.5	2	4.108	1.2	0.048	1
1231	1	15.6	3.5	2	4.119	1.2	0.047	1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
127	1	12.5	2.8	1	5.683	1.2	0.049	1
164	1	12.5	2.8	1	6.301	1.3	0.050	1
133	1	12.5	2.8	1	6.695	1.3	0.051	1
150	1	12.5	2.8	1	6.736	1.2	0.049	1
177	1	12.5	2.8	1	6.982	1.3	0.050	1
130	1	12.5	2.8	1	7.373	1.3	0.050	1
192	1	12.5	2.8	1	8.278	1.3	0.051	1
135	1	12.5	2.8	1	8.543	1.3	0.053	1
1239	1	12.5	2.8	2	8.216	1.2	0.046	1
1253	1	12.5	2.8	2	8.920	1.2	0.046	1
1263	1	12.5	2.8	2	9.501	1.2	0.046	1
1252	1	12.5	2.8	2	9.748	1.2	0.046	1
1248	1	12.5	2.8	2	10.972	1.2	0.046	1
1240	1	12.5	2.8	2	11.012	1.2	0.046	1
1206	1	12.5	2.8	2	12.518	1.1	0.045	1
1229	1	12.5	2.8	2	12.700	1.2	0.047	1
160	1	9.3	2.1	1	23.090	1.3	0.050	1
179	1	9.3	2.1	1	25.313	1.3	0.052	1
14	1	9.3	2.1	1	25.912	1.3	0.051	1
155	1	9.3	2.1	1	26.327	1.3	0.051	1
182	1	9.3	2.1	1	30.366	1.3	0.053	1
124	1	9.3	2.1	1	30.539	1.3	0.050	1
132	1	9.3	2.1	1	32.695	1.3	0.052	1
185	1	9.3	2.1	1	33.855	1.3	0.051	1
1274	1	9.3	2.1	2	39.283	1.2	0.047	1
1219	1	9.3	2.1	2	40.312	1.2	0.046	1
1247	1	9.3	2.1	2	41.670	1.1	0.045	1
1260	1	9.3	2.1	2	41.695	1.2	0.046	1
1212	1	9.3	2.1	2	42.411	1.2	0.047	1
1214	1	9.3	2.1	2	42.578	1.2	0.047	1
1223	1	9.3	2.1	2	48.430	1.2	0.047	1
1278	1	9.3	2.1	2	59.056	1.2	0.046	1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
176	1	6.2	1.4	1	180.937	1.3	0.051	1
191	1	6.2	1.4	1	181.002	1.2	0.049	1
153	1	6.2	1.4	1	203.121	1.3	0.052	1
161	1	6.2	1.4	1	203.582	1.2	0.049	1
187	1	6.2	1.4	1	214.091	1.3	0.051	1
178	1	6.2	1.4	1	218.018	1.3	0.050	1
148	1	6.2	1.4	1	233.190	1.3	0.050	1
167	1	6.2	1.4	1	233.631	1.3	0.051	1
165	1	6.2	1.4	1	234.297	1.2	0.049	1
123	1	6.2	1.4	1	241.560	1.3	0.051	1
180	1	6.2	1.4	1	266.917	1.3	0.050	1
143	1	6.2	1.4	1	287.668	1.3	0.050	1
141	1	6.2	1.4	1	301.290	1.3	0.050	1
146	1	6.2	1.4	1	321.389	1.4	0.055	1
1288	1	6.2	1.4	2	257.99	1.2	0.045	1
1292	1	6.2	1.4	2	269.76	1.1	0.045	1
1225	1	6.2	1.4	2	293.15	1.2	0.046	1
1266	1	6.2	1.4	2	295.50	1.1	0.045	1
1256	1	6.2	1.4	2	297.36	1.2	0.046	1
1259	1	6.2	1.4	2	311.21	1.2	0.047	1
1273	1	6.2	1.4	2	332.60	1.2	0.047	1
1267	1	6.2	1.4	2	358.05	1.2	0.047	1

Table A-2. Data for tape system 2

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
292	2	24.9	5.6	1	2.529	0.90	0.035	1
278	2	24.9	5.6	1	2.579	0.93	0.037	1
285	2	24.9	5.6	1	2.629	0.91	0.036	1
224	2	24.9	5.6	1	2.647	0.97	0.038	1
238	2	24.9	5.6	1	2.740	0.92	0.036	1
262	2	24.9	5.6	1	2.780	0.94	0.037	1
298	2	24.9	5.6	1	2.822	0.96	0.038	1
289	2	24.9	5.6	1	2.966	0.91	0.036	1
2231	2	24.9	5.6	2	0.876	0.97	0.038	2
2212	2	24.9	5.6	2	0.920	0.90	0.036	2
2234	2	24.9	5.6	2	1.301	0.90	0.036	2
2293	2	24.9	5.6	2	1.404	0.91	0.036	2
2263	2	24.9	5.6	2	1.801	0.93	0.037	2
2261	2	24.9	5.6	2	1.869	0.87	0.034	2
2228	2	24.9	5.6	2	2.044	0.88	0.035	2
2427	2	24.9	5.6	3	1.896	0.88	0.035	1
2402	2	24.9	5.6	3	1.910	0.86	0.034	1
2494	2	24.9	5.6	3	2.037	0.88	0.035	1
2438	2	24.9	5.6	3	2.337	0.90	0.035	1
2418	2	24.9	5.6	3	2.410	0.90	0.036	1
2471	2	24.9	5.6	3	2.814	0.92	0.036	1
2439	2	24.9	5.6	3	3.128	0.94	0.037	1
2410	2	24.9	5.6	3	3.217	0.90	0.036	1
2605	2	24.9	5.6	4	2.472	0.87	0.034	1
2673	2	24.9	5.6	4	2.867	0.86	0.034	1
2618	2	24.9	5.6	4	2.869	0.85	0.034	1
2677	2	24.9	5.6	4	2.872	0.85	0.034	1
2656	2	24.9	5.6	4	2.927	0.87	0.034	1
2660	2	24.9	5.6	4	3.123	0.86	0.034	1
2649	2	24.9	5.6	4	3.226	0.87	0.034	1
2653	2	24.9	5.6	4	3.284	0.85	0.034	1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
283	2	21.8	4.9	1	3.991	0.93	0.037	1
260	2	21.8	4.9	1	4.096	0.94	0.037	1
268	2	21.8	4.9	1	4.096	0.89	0.035	1
293	2	21.8	4.9	1	4.138	0.91	0.036	1
218	2	21.8	4.9	1	4.139	0.85	0.034	1
241	2	21.8	4.9	1	4.490	0.89	0.035	1
2271	2	21.8	4.9	2	1.571	0.94	0.037	2
2210	2	21.8	4.9	2	1.703	0.89	0.035	2
2213	2	21.8	4.9	2	1.925	0.91	0.036	2
2225	2	21.8	4.9	2	2.101	0.93	0.037	2
2235	2	21.8	4.9	2	2.413	0.92	0.036	2
2268	2	21.8	4.9	2	2.512	0.94	0.037	2
2224	2	21.8	4.9	2	2.754	0.93	0.037	2
2229	2	21.8	4.9	2	3.341	0.89	0.035	2
2411	2	21.8	4.9	3	2.071	0.92	0.036	1
2405	2	21.8	4.9	3	2.472	0.90	0.036	1
2464	2	21.8	4.9	3	3.122	0.89	0.035	1
2429	2	21.8	4.9	3	3.138	0.93	0.037	1
2444	2	21.8	4.9	3	3.272	0.88	0.035	1
2401	2	21.8	4.9	3	3.534	0.88	0.035	1
2477	2	21.8	4.9	3	4.432	0.93	0.037	1
2451	2	21.8	4.9	3	5.432	0.92	0.036	1
2624	2	21.8	4.9	4	4.295	0.86	0.034	1
2664	2	21.8	4.9	4	4.306	0.85	0.034	1
2636	2	21.8	4.9	4	4.514	0.88	0.035	1
2654	2	21.8	4.9	4	4.547	0.85	0.034	1
2608	2	21.8	4.9	4	4.552	0.87	0.034	1
2680	2	21.8	4.9	4	4.599	0.87	0.034	1
2639	2	21.8	4.9	4	4.731	0.88	0.035	1
2606	2	21.8	4.9	4	5.093	0.88	0.035	1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
232	2	18.7	4.2	1	5.908	0.97	0.038	1
274	2	18.7	4.2	1	6.530	0.97	0.038	1
240	2	18.7	4.2	1	6.732	0.94	0.037	1
26	2	18.7	4.2	1	7.168	0.96	0.038	1
210	2	18.7	4.2	1	7.223	0.93	0.037	1
288	2	18.7	4.2	1	7.306	0.96	0.038	1
249	2	18.7	4.2	1	7.508	0.94	0.037	1
27	2	18.7	4.2	1	8.390	0.97	0.038	1
2208	2	18.7	4.2	2	2.471	0.90	0.035	2
2259	2	18.7	4.2	2	2.630	0.90	0.036	2
2251	2	18.7	4.2	2	3.853	0.90	0.036	2
2214	2	18.7	4.2	2	4.688	0.93	0.037	2
2258	2	18.7	4.2	2	4.837	0.87	0.034	2
2302	2	18.7	4.2	2	5.034	0.92	0.036	2
2272	2	18.7	4.2	2	5.103	0.92	0.036	2
2297	2	18.7	4.2	2	5.682	0.97	0.038	2
2432	2	18.7	4.2	3	5.837	0.94	0.037	1
2454	2	18.7	4.2	3	6.006	0.92	0.036	1
2502	2	18.7	4.2	3	6.194	0.95	0.038	1
2437	2	18.7	4.2	3	6.811	0.91	0.036	1
2461	2	18.7	4.2	3	7.113	0.91	0.036	1
2498	2	18.7	4.2	3	7.821	0.90	0.036	1
2483	2	18.7	4.2	3	8.546	0.92	0.036	1
2478	2	18.7	4.2	3	8.992	0.91	0.036	1
2670	2	18.7	4.2	4	6.293	0.88	0.035	1
2646	2	18.7	4.2	4	7.151	0.85	0.034	1
2630	2	18.7	4.2	4	7.244	0.87	0.034	1
2667	2	18.7	4.2	4	7.456	0.84	0.033	1
2604	2	18.7	4.2	4	8.114	0.89	0.035	1
2620	2	18.7	4.2	4	8.331	0.89	0.035	1
2666	2	18.7	4.2	4	8.971	0.87	0.034	1
2674	2	18.7	4.2	4	9.686	0.89	0.035	1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
25	2	15.6	3.5	1	11.373	0.90	0.035	1
282	2	15.6	3.5	1	11.604	0.98	0.039	1
279	2	15.6	3.5	1	11.674	0.91	0.036	1
29	2	15.6	3.5	1	11.821	0.96	0.038	1
296	2	15.6	3.5	1	12.103	0.86	0.034	1
215	2	15.6	3.5	1	12.232	0.94	0.037	1
295	2	15.6	3.5	1	12.485	0.90	0.036	1
214	2	15.6	3.5	1	12.809	0.92	0.036	1
2255	2	15.6	3.5	2	5.973	0.91	0.036	2
2287	2	15.6	3.5	2	6.980	0.91	0.036	2
2296	2	15.6	3.5	2	7.842	0.87	0.034	2
2298	2	15.6	3.5	2	8.033	0.93	0.037	2
2283	2	15.6	3.5	2	8.251	0.89	0.035	2
2220	2	15.6	3.5	2	9.216	0.90	0.035	2
2240	2	15.6	3.5	2	9.443	0.94	0.037	2
2217	2	15.6	3.5	2	10.989	0.91	0.036	2
2403	2	15.6	3.5	3	9.857	0.89	0.035	1
2486	2	15.6	3.5	3	10.692	0.93	0.037	1
2407	2	15.6	3.5	3	11.042	0.90	0.036	1
2450	2	15.6	3.5	3	11.567	0.93	0.037	1
2421	2	15.6	3.5	3	12.300	0.89	0.035	1
2466	2	15.6	3.5	3	13.145	0.90	0.035	1
2485	2	15.6	3.5	3	13.989	0.88	0.035	1
2489	2	15.6	3.5	3	14.989	0.93	0.037	1
2637	2	15.6	3.5	4	10.073	0.85	0.034	1
2631	2	15.6	3.5	4	12.337	0.89	0.035	1
2613	2	15.6	3.5	4	13.146	0.87	0.034	1
2640	2	15.6	3.5	4	13.666	0.86	0.034	1
2675	2	15.6	3.5	4	14.891	0.88	0.035	1
2661	2	15.6	3.5	4	15.066	0.85	0.034	1
2619	2	15.6	3.5	4	16.641	0.88	0.035	1
2648	2	15.6	3.5	4	26.267	0.86	0.034	1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
2104	2	12.5	2.8	1	22.976	0.89	0.035	1
255	2	12.5	2.8	1	23.448	0.91	0.036	1
28	2	12.5	2.8	1	24.119	0.96	0.038	1
263	2	12.5	2.8	1	26.666	0.90	0.036	1
2103	2	12.5	2.8	1	26.726	0.89	0.035	1
277	2	12.5	2.8	1	27.500	0.89	0.035	1
265	2	12.5	2.8	1	28.655	0.91	0.036	1
235	2	12.5	2.8	1	31.284	0.93	0.037	1
2241	2	12.5	2.8	2	15.344	0.89	0.035	2
2281	2	12.5	2.8	2	15.962	0.89	0.035	2
2300	2	12.5	2.8	2	16.381	0.93	0.037	2
2304	2	12.5	2.8	2	17.345	0.89	0.035	2
2221	2	12.5	2.8	2	19.312	0.89	0.035	2
2252	2	12.5	2.8	2	19.813	0.89	0.035	2
2216	2	12.5	2.8	2	20.924	0.91	0.036	2
2202	2	12.5	2.8	2	21.402	0.89	0.035	2
2457	2	12.5	2.8	3	25.118	0.90	0.035	1
2413	2	12.5	2.8	3	26.029	0.95	0.038	1
2496	2	12.5	2.8	3	27.535	0.90	0.035	1
2414	2	12.5	2.8	3	28.263	0.91	0.036	1
2409	2	12.5	2.8	3	32.432	0.93	0.037	1
2628	2	12.5	2.8	4	24.414	0.88	0.035	1
2663	2	12.5	2.8	4	24.460	0.89	0.035	1
2672	2	12.5	2.8	4	29.448	0.90	0.036	1
2665	2	12.5	2.8	4	29.637	0.89	0.035	1
2658	2	12.5	2.8	4	29.778	0.90	0.036	1
2652	2	12.5	2.8	4	29.807	0.90	0.035	1
2657	2	12.5	2.8	4	30.655	0.89	0.035	1
2629	2	12.5	2.8	4	36.030	0.90	0.036	1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
21	2	9.3	2.1	1	73.507	0.92	0.036	1
248	2	9.3	2.1	1	77.706	0.89	0.035	1
2100	2	9.3	2.1	1	91.396	0.92	0.036	1
253	2	9.3	2.1	1	95.709	0.91	0.036	1
237	2	9.3	2.1	1	96.624	0.89	0.035	1
247	2	9.3	2.1	1	99.992	0.95	0.038	1
290	2	9.3	2.1	1	107.796	0.90	0.036	1
242	2	9.3	2.1	1	114.663	0.86	0.034	1
2277	2	9.3	2.1	2	43.140	0.88	0.035	2
2280	2	9.3	2.1	2	43.915	0.90	0.036	2
2266	2	9.3	2.1	2	47.489	0.90	0.036	2
2253	2	9.3	2.1	2	51.548	0.87	0.034	2
2286	2	9.3	2.1	2	60.274	0.90	0.035	2
2262	2	9.3	2.1	2	70.539	0.94	0.037	2
2201	2	9.3	2.1	2	73.404	0.88	0.035	2
2285	2	9.3	2.1	2	89.524	0.86	0.034	2
2447	2	9.3	2.1	3	66.138	0.97	0.038	1
2493	2	9.3	2.1	3	81.501	0.90	0.036	1
2480	2	9.3	2.1	3	81.770	0.97	0.038	1
2453	2	9.3	2.1	3	100.324	0.92	0.036	1
2499	2	9.3	2.1	3	101.103	0.87	0.034	1
2431	2	9.3	2.1	3	105.141	0.93	0.037	1
2625	2	9.3	2.1	4	49.426	0.90	0.036	1
2622	2	9.3	2.1	4	96.633	0.87	0.034	1
2602	2	9.3	2.1	4	99.856	0.92	0.036	1
2611	2	9.3	2.1	4	105.306	0.92	0.036	1
2676	2	9.3	2.1	4	108.747	0.87	0.034	1
2614	2	9.3	2.1	4	108.777	0.90	0.035	1
2609	2	9.3	2.1	4	113.883	0.88	0.035	1
2601	2	9.3	2.1	4	133.130	0.90	0.036	1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
216	2	6.2	1.4	1	489.483	0.95	0.037	1
212	2	6.2	1.4	1	554.744	0.89	0.035	1
284	2	6.2	1.4	1	557.800	0.94	0.037	1
297	2	6.2	1.4	1	577.012	0.90	0.036	1
2102	2	6.2	1.4	1	599.976	0.89	0.035	1
225	2	6.2	1.4	1	603.769	0.92	0.036	1
244	2	6.2	1.4	1	604.112	0.94	0.037	1
220	2	6.2	1.4	1	606.252	0.96	0.038	1
269	2	6.2	1.4	1	610.013	0.93	0.037	1
275	2	6.2	1.4	1	631.827	0.91	0.036	1
286	2	6.2	1.4	1	664.580	0.95	0.037	1
246	2	6.2	1.4	1	683.425	0.98	0.039	1
299	2	6.2	1.4	1	688.883	0.97	0.038	1
281	2	6.2	1.4	1	1096.275	0.91	0.036	1
2290	2	6.2	1.4	2	355.264	0.85	0.033	1
2203	2	6.2	1.4	2	485.070	0.84	0.033	1
2260	2	6.2	1.4	2	485.070	0.85	0.033	1
2284	2	6.2	1.4	2	485.070	0.85	0.034	1
2233	2	6.2	1.4	2	606.227	0.89	0.035	1
2264	2	6.2	1.4	2	663.795	0.86	0.034	1
2305	2	6.2	1.4	2	698.749	0.88	0.035	1
2206	2	6.2	1.4	2	743.731	0.90	0.035	1
2449	2	6.2	1.4	3	431.905	0.88	0.035	1
2424	2	6.2	1.4	3	485.070	0.83	0.033	1
2425	2	6.2	1.4	3	485.070	0.84	0.033	1
2428	2	6.2	1.4	3	640.552	0.84	0.033	1
2422	2	6.2	1.4	3	688.329	0.88	0.035	1
2474	2	6.2	1.4	3	697.890	0.84	0.033	1
2417	2	6.2	1.4	3	716.818	0.89	0.035	1
2476	2	6.2	1.4	3	785.686	0.85	0.034	1

Sample No.	Adhesive No.	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
2651	2	6.2	1.4	4	747.210	0.81	0.032	1
2678	2	6.2	1.4	4	753.401	0.85	0.033	1
2616	2	6.2	1.4	4	786.994	0.86	0.034	1
2610	2	6.2	1.4	4	806.381	0.85	0.033	1
2623	2	6.2	1.4	4	862.765	0.86	0.034	1
2659	2	6.2	1.4	4	868.306	0.81	0.032	1
2647	2	6.2	1.4	4	938.894	0.85	0.033	1

Table A-3. Data for liquid adhesive system

Sample No.	Adhesive System	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
80LA	3	24.9	5.6	1	0.794	0.18	0.0071	1
36LA	3	24.9	5.6	1	0.960	0.20	0.0080	1
78LA	3	24.9	5.6	1	1.046	0.17	0.0066	1
72LA	3	24.9	5.6	1	1.692	0.17	0.0066	1
59LA	3	24.9	5.6	1	2.163	0.18	0.0071	1
21LA	3	24.9	5.6	1	2.373	0.20	0.0077	1
23LA	3	24.9	5.6	1	2.674	0.21	0.0081	1
22LA	3	24.9	5.6	1	2.696	0.20	0.0080	1
3232	3	24.9	5.6	2	0.671	0.18	0.0070	2
3224	3	24.9	5.6	2	0.904	0.19	0.0073	2
3292	3	24.9	5.6	2	0.910	0.19	0.0077	2
3243	3	24.9	5.6	2	0.974	0.20	0.0080	2
3201	3	24.9	5.6	2	0.990	0.19	0.0075	2
3291	3	24.9	5.6	2	1.019	0.18	0.0070	2
3236	3	24.9	5.6	2	1.087	0.19	0.0075	2
3301	3	24.9	5.6	2	1.121	0.19	0.0073	2
3524	3	24.9	5.6	3	0.381	0.19	0.0076	1
3539	3	24.9	5.6	3	0.460	0.20	0.0079	1
3544	3	24.9	5.6	3	0.490	0.19	0.0075	1
3549	3	24.9	5.6	3	0.497	0.20	0.0079	1
3556	3	24.9	5.6	3	0.498	0.20	0.0079	1
3537	3	24.9	5.6	3	0.505	0.19	0.0076	1
3512	3	24.9	5.6	3	0.564	0.19	0.0074	1
3515	3	24.9	5.6	3	0.635	0.20	0.0078	1
3611	3	24.9	5.6	4	0.715	0.17	0.0069	1
3311	3	24.9	5.6	4	0.719	0.17	0.0065	1
3031	3	24.9	5.6	4	0.812	0.17	0.0069	1
3021	3	24.9	5.6	4	0.830	0.19	0.0074	1
3761	3	24.9	5.6	4	0.832	0.17	0.0069	1
3451	3	24.9	5.6	4	0.861	0.19	0.0074	1
3381	3	24.9	5.6	4	0.870	0.17	0.0066	1

Sample No.	Adhesive System	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
3834	3	24.9	5.6	5	0.741	0.24	0.0096	1
3454	3	24.9	5.6	5	0.752	0.25	0.0097	1
3044	3	24.9	5.6	5	0.786	0.22	0.0086	1
3194	3	24.9	5.6	5	0.898	0.22	0.0087	1
3494	3	24.9	5.6	5	0.938	0.22	0.0085	1
3694	3	24.9	5.6	5	1.053	0.23	0.0092	1
3094	3	24.9	5.6	5	1.065	0.21	0.0081	1
3654	3	24.9	5.6	5	1.073	0.21	0.0083	1
46LA	3	21.8	4.9	1	1.641	0.21	0.0084	1
30LA	3	21.8	4.9	1	2.123	0.21	0.0081	1
11LA	3	21.8	4.9	1	2.300	0.21	0.0082	1
92LA	3	21.8	4.9	1	2.300	0.17	0.0066	1
73LA	3	21.8	4.9	1	2.338	0.20	0.0077	1
88LA	3	21.8	4.9	1	3.146	0.21	0.0084	1
7LA	3	21.8	4.9	1	3.357	0.21	0.0084	1
85LA	3	21.8	4.9	1	3.873	0.21	0.0084	1
3223	3	21.8	4.9	2	1.302	0.18	0.0072	2
3210	3	21.8	4.9	2	1.387	0.19	0.0073	2
3261	3	21.8	4.9	2	1.541	0.18	0.0070	2
3252	3	21.8	4.9	2	1.549	0.19	0.0077	2
3259	3	21.8	4.9	2	1.684	0.20	0.0080	2
3218	3	21.8	4.9	2	1.754	0.19	0.0075	2
3260	3	21.8	4.9	2	1.881	0.19	0.0075	2
3238	3	21.8	4.9	2	2.089	0.19	0.0073	2
3536	3	21.8	4.9	3	0.659	0.19	0.0075	1
3561	3	21.8	4.9	3	0.754	0.19	0.0075	1
3548	3	21.8	4.9	3	0.761	0.19	0.0074	1
3565	3	21.8	4.9	3	0.771	0.21	0.0081	1
3517	3	21.8	4.9	3	0.777	0.17	0.0068	1
3526	3	21.8	4.9	3	0.833	0.21	0.0083	1
3518	3	21.8	4.9	3	0.833	0.21	0.0084	1
3568	3	21.8	4.9	3	0.840	0.20	0.0080	1

Sample No.	Adhesive System	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
3081	3	21.8	4.9	4	0.937	0.18	0.0070	1
3711	3	21.8	4.9	4	0.965	0.20	0.0080	1
3771	3	21.8	4.9	4	0.996	0.19	0.0075	1
3681	3	21.8	4.9	4	1.074	0.17	0.0066	1
3471	3	21.8	4.9	4	1.101	0.20	0.0078	1
3301	3	21.8	4.9	4	1.102	0.18	0.0071	1
3491	3	21.8	4.9	4	1.119	0.17	0.0065	1
3691	3	21.8	4.9	4	1.182	0.19	0.0074	1
3584	3	21.8	4.9	5	1.227	0.25	0.0098	1
3214	3	21.8	4.9	5	1.235	0.25	0.0099	1
3474	3	21.8	4.9	5	1.277	0.25	0.0097	1
3504	3	21.8	4.9	5	1.277	0.25	0.0098	1
3394	3	21.8	4.9	5	1.279	0.25	0.0100	1
3424	3	21.8	4.9	5	1.367	0.24	0.0095	1
3384	3	21.8	4.9	5	1.396	0.27	0.0106	1
3464	3	21.8	4.9	5	1.458	0.25	0.0098	1
90LA	3	18.7	4.2	1	4.492	0.21	0.0084	1
75LA	3	18.7	4.2	1	6.301	0.21	0.0084	1
12LA	3	18.7	4.2	1	6.482	0.20	0.0077	1
45LA	3	18.7	4.2	1	6.908	0.17	0.0066	1
20LA	3	18.7	4.2	1	7.055	0.21	0.0084	1
83LA	3	18.7	4.2	1	7.427	0.21	0.0084	1
9LA	3	18.7	4.2	1	7.727	0.21	0.0082	1
13LA	3	18.7	4.2	1	8.225	0.21	0.0081	1
3226	3	18.7	4.2	2	2.431	0.18	0.0072	2
3262	3	18.7	4.2	2	2.522	0.19	0.0075	2
3273	3	18.7	4.2	2	2.645	0.18	0.0070	2
3303	3	18.7	4.2	2	2.868	0.18	0.0072	2
3284	3	18.7	4.2	2	3.006	0.19	0.0073	2
3290	3	18.7	4.2	2	3.106	0.18	0.0072	2
3202	3	18.7	4.2	2	3.198	0.20	0.0078	2
3257	3	18.7	4.2	2	3.277	0.20	0.0080	2

Sample No.	Adhesive System	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
3557	3	18.7	4.2	3	0.985	0.17	0.0068	1
3525	3	18.7	4.2	3	1.002	0.17	0.0068	1
3571	3	18.7	4.2	3	1.004	0.18	0.0070	1
3521	3	18.7	4.2	3	1.022	0.17	0.0069	1
3575	3	18.7	4.2	3	1.044	0.18	0.0071	1
3574	3	18.7	4.2	3	1.143	0.17	0.0069	1
3555	3	18.7	4.2	3	1.166	0.18	0.0070	1
3528	3	18.7	4.2	3	1.295	0.17	0.0066	1
3131	3	18.7	4.2	4	1.279	0.19	0.0074	1
3061	3	18.7	4.2	4	1.369	0.19	0.0075	1
3841	3	18.7	4.2	4	1.422	0.20	0.0080	1
3821	3	18.7	4.2	4	1.431	0.21	0.0084	1
3561	3	18.7	4.2	4	1.580	0.22	0.0085	1
3151	3	18.7	4.2	4	1.642	0.18	0.0070	1
3371	3	18.7	4.2	4	1.656	0.21	0.0081	1
3231	3	18.7	4.2	4	1.664	0.17	0.0068	1
3714	3	18.7	4.2	5	1.827	0.24	0.0095	1
3534	3	18.7	4.2	5	1.831	0.25	0.0099	1
3564	3	18.7	4.2	5	1.842	0.25	0.0097	1
3664	3	18.7	4.2	5	1.983	0.26	0.0104	1
3484	3	18.7	4.2	5	2.024	0.25	0.0099	1
3034	3	18.7	4.2	5	2.273	0.26	0.0104	1
3574	3	18.7	4.2	5	2.282	0.26	0.0103	1
3824	3	18.7	4.2	5	2.630	0.26	0.0103	1
28LA	3	15.6	3.5	1	12.225	0.17	0.0067	1
33LA	3	15.6	3.5	1	12.452	0.21	0.0084	1
27LA	3	15.6	3.5	1	12.991	0.18	0.0071	1
17LA	3	15.6	3.5	1	15.684	0.18	0.0071	1
18LA	3	15.6	3.5	1	15.822	0.21	0.0081	1
74LA	3	15.6	3.5	1	16.865	0.18	0.0070	1
96LA	3	15.6	3.5	1	18.574	0.18	0.0071	1
103LA	3	15.6	3.5	1	23.873	0.18	0.0070	1

Sample No.	Adhesive System	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
3244	3	15.6	3.5	2	5.495	0.20	0.0080	2
3240	3	15.6	3.5	2	5.565	0.19	0.0075	2
3289	3	15.6	3.5	2	6.123	0.18	0.0070	2
3268	3	15.6	3.5	2	6.164	0.10	0.0040	2
3266	3	15.6	3.5	2	6.232	0.19	0.0077	2
3305	3	15.6	3.5	2	6.881	0.19	0.0073	2
3258	3	15.6	3.5	2	7.290	0.20	0.0080	2
3265	3	15.6	3.5	2	7.859	0.20	0.0078	2
3542	3	15.6	3.5	3	1.767	0.18	0.0070	1
3507	3	15.6	3.5	3	1.780	0.17	0.0066	1
3559	3	15.6	3.5	3	1.842	0.17	0.0065	1
3501	3	15.6	3.5	3	1.932	0.17	0.0068	1
3516	3	15.6	3.5	3	2.073	0.17	0.0065	1
3506	3	15.6	3.5	3	2.296	0.20	0.0079	1
3540	3	15.6	3.5	3	2.469	0.17	0.0069	1
3541	3	15.6	3.5	4	1.780	0.21	0.0081	1
3291	3	15.6	3.5	4	2.175	0.18	0.0070	1
3041	3	15.6	3.5	4	2.225	0.17	0.0069	1
3241	3	15.6	3.5	4	2.283	0.17	0.0066	1
3111	3	15.6	3.5	4	2.296	0.20	0.0080	1
3581	3	15.6	3.5	4	2.431	0.17	0.0069	1
3011	3	15.6	3.5	4	2.434	0.19	0.0076	1
3731	3	15.6	3.5	4	2.551	0.17	0.0069	1
3434	3	15.6	3.5	5	2.651	0.23	0.0089	1
3204	3	15.6	3.5	5	3.003	0.25	0.0097	1
3084	3	15.6	3.5	5	3.122	0.23	0.0090	1
3054	3	15.6	3.5	5	3.217	0.25	0.0099	1
3354	3	15.6	3.5	5	3.341	0.25	0.0100	1
3744	3	15.6	3.5	5	3.388	0.25	0.0099	1
3764	3	15.6	3.5	5	3.613	0.24	0.0095	1
3404	3	15.6	3.5	5	3.992	0.24	0.0094	1

Sample No.	Adhesive System	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
50LA	3	12.5	2.8	1	84.737	0.21	0.0082	1
93LA	3	12.5	2.8	1	84.895	0.17	0.0066	1
63LA	3	12.5	2.8	1	87.908	0.19	0.0075	1
98LA	3	12.5	2.8	1	94.762	0.19	0.0074	1
105LA	3	12.5	2.8	1	105.088	0.17	0.0069	1
24LA	3	12.5	2.8	1	133.072	0.19	0.0074	1
70LA	3	12.5	2.8	1	134.996	0.17	0.0067	1
81LA	3	12.5	2.8	1	146.734	0.18	0.0071	1
3280	3	12.5	2.8	2	12.191	0.18	0.0070	2
3241	3	12.5	2.8	2	15.792	0.20	0.0078	2
3277	3	12.5	2.8	2	16.139	0.19	0.0075	2
3285	3	12.5	2.8	2	16.976	0.18	0.0070	2
3208	3	12.5	2.8	2	17.544	0.19	0.0077	2
3213	3	12.5	2.8	2	17.774	0.19	0.0077	2
3237	3	12.5	2.8	2	21.664	0.19	0.0073	2
3278	3	12.5	2.8	2	22.299	0.19	0.0075	2
3534	3	12.5	2.8	3	2.727	0.17	0.0065	1
3547	3	12.5	2.8	3	2.811	0.18	0.0071	1
3570	3	12.5	2.8	3	3.144	0.17	0.0066	1
3513	3	12.5	2.8	3	3.346	0.17	0.0066	1
3520	3	12.5	2.8	3	3.476	0.17	0.0066	1
3504	3	12.5	2.8	3	3.491	0.17	0.0069	1
3531	3	12.5	2.8	3	3.506	0.18	0.0071	1
3554	3	12.5	2.8	3	3.746	0.17	0.0068	1
3441	3	12.5	2.8	4	3.206	0.20	0.0079	1
3461	3	12.5	2.8	4	3.522	0.18	0.0070	1
3781	3	12.5	2.8	4	3.630	0.17	0.0068	1
3271	3	12.5	2.8	4	3.684	0.17	0.0068	1
3051	3	12.5	2.8	4	3.801	0.17	0.0069	1
3411	3	12.5	2.8	4	3.833	0.21	0.0083	1
3141	3	12.5	2.8	4	3.915	0.18	0.0073	1
3361	3	12.5	2.8	4	4.184	0.18	0.0073	1

Sample No.	Adhesive System	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
3144	3	12.5	2.8	5	3.763	0.25	0.0099	1
3154	3	12.5	2.8	5	4.458	0.24	0.0095	1
3324	3	12.5	2.8	5	4.477	0.22	0.0086	1
3364	3	12.5	2.8	5	4.784	0.23	0.0090	1
3374	3	12.5	2.8	5	5.113	0.23	0.0090	1
3644	3	12.5	2.8	5	5.680	0.25	0.0097	1
3794	3	12.5	2.8	5	5.864	0.25	0.0100	1
3704	3	12.5	2.8	5	6.242	0.24	0.0096	1
57LA	3	9.3	2.1	1	88.389	0.17	0.0066	1
66LA	3	9.3	2.1	1	330.580	0.20	0.0077	1
40LA	3	9.3	2.1	1	480.381	0.18	0.0070	1
64LA	3	9.3	2.1	1	550.987	0.20	0.0080	1
100LA	3	9.3	2.1	1	561.571	0.17	0.0066	1
1LA	3	9.3	2.1	1	574.250	0.18	0.0072	1
54LA	3	9.3	2.1	1	576.931	0.17	0.0067	1
3230	3	9.3	2.1	2	46.776	0.18	0.0072	2
3217	3	9.3	2.1	2	47.339	0.19	0.0073	2
3271	3	9.3	2.1	2	66.117	0.20	0.0080	2
3233	3	9.3	2.1	2	73.375	0.19	0.0075	2
3304	3	9.3	2.1	2	94.750	0.18	0.0072	2
3287	3	9.3	2.1	2	95.908	0.19	0.0077	2
3294	3	9.3	2.1	2	104.813	0.20	0.0080	2
3242	3	9.3	2.1	2	105.151	0.19	0.0075	2
3551	3	9.3	2.1	3	4.624	0.17	0.0066	1
3545	3	9.3	2.1	3	6.261	0.17	0.0066	1
3519	3	9.3	2.1	3	6.386	0.17	0.0066	1
3569	3	9.3	2.1	3	6.858	0.17	0.0066	1
3550	3	9.3	2.1	3	7.018	0.18	0.0071	1
3573	3	9.3	2.1	3	7.223	0.20	0.0078	1
3558	3	9.3	2.1	3	7.422	0.17	0.0065	1
3514	3	9.3	2.1	3	9.802	0.17	0.0069	1

Sample No.	Adhesive System	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
3791	3	9.3	2.1	4	5.641	0.19	0.0074	1
3601	3	9.3	2.1	4	5.717	0.17	0.0068	1
3621	3	9.3	2.1	4	6.611	0.22	0.0085	1
3701	3	9.3	2.1	4	6.876	0.18	0.0071	1
3091	3	9.3	2.1	4	6.884	0.18	0.0070	1
3351	3	9.3	2.1	4	6.957	0.19	0.0075	1
3221	3	9.3	2.1	4	7.479	0.21	0.0081	1
3331	3	9.3	2.1	4	8.035	0.20	0.0078	1
3294	3	9.3	2.1	5	6.388	0.24	0.0096	1
3784	3	9.3	2.1	5	7.594	0.22	0.0085	1
3164	3	9.3	2.1	5	8.344	0.25	0.0097	1
3754	3	9.3	2.1	5	8.903	0.25	0.0097	1
3624	3	9.3	2.1	5	9.239	0.23	0.0091	1
3684	3	9.3	2.1	5	9.761	0.24	0.0094	1
3554	3	9.3	2.1	5	9.809	0.24	0.0095	1
3674	3	9.3	2.1	5	10.267	0.25	0.0098	1
3562	3	6.2	1.4	3	17.659	0.17	0.0066	1
3546	3	6.2	1.4	3	24.888	0.16	0.0064	1
3527	3	6.2	1.4	3	25.303	0.17	0.0068	1
3505	3	6.2	1.4	3	27.600	0.18	0.0071	1
3502	3	6.2	1.4	3	28.703	0.19	0.0075	1
3541	3	6.2	1.4	3	28.896	0.16	0.0063	1
3552	3	6.2	1.4	3	31.547	0.17	0.0066	1
3560	3	6.2	1.4	3	35.157	0.19	0.0074	1
3641	3	6.2	1.4	4	18.796	0.23	0.0090	1
3511	3	6.2	1.4	4	19.395	0.23	0.0089	1
3521	3	6.2	1.4	4	21.862	0.22	0.0086	1
3651	3	6.2	1.4	4	22.611	0.21	0.0084	1
3171	3	6.2	1.4	4	25.186	0.23	0.0091	1
3211	3	6.2	1.4	4	25.502	0.22	0.0088	1
3261	3	6.2	1.4	4	27.916	0.21	0.0083	1
3181	3	6.2	1.4	4	28.536	0.21	0.0084	1

Sample No.	Adhesive System	Load		Rep. No.	TTF hours	Adhesive Thickness		Failure Mode
		N	lbf			mm	in	
3344	3	6.2	1.4	5	31.359	0.24	0.0096	1
3304	3	6.2	1.4	5	31.954	0.23	0.0091	1
3414	3	6.2	1.4	5	32.845	0.26	0.0103	1
3274	3	6.2	1.4	5	33.209	0.27	0.0105	1
3814	3	6.2	1.4	5	35.799	0.21	0.0082	1
3444	3	6.2	1.4	5	46.751	0.24	0.0096	1
3844	3	6.2	1.4	5	47.190	0.27	0.0106	1
3734	3	6.2	1.4	5	50.843	0.26	0.0104	1

APPENDIX B. VARIABILITY OF THE TIME-TO-FAILURE DATA

The most important conclusions from this paper follow from an analysis of mean time-to-failure. The variability in time-to-failure is considered in this appendix. In addition to being interesting in its own right, these results should prove useful in future data-modeling efforts.

It is clear from the summary statistics in Table B-1 that the standard deviation of time-to-failure tends to increase with mean time-to-failure, and might also differ systemically among adhesive systems and replicate sets. An investigation was therefore undertaken of how the standard deviation of time-to-failure depends on load, adhesive system, and replicate. Before proceeding to the discussion of this analysis, it is important to keep in mind that standard deviations (and coefficients of variation) are much more difficult to estimate than means with small data sets. Consequently, the differences in the standard deviations and coefficients of variation in Table B-1, which at first might seem very large, are not necessarily inconsistent with a true coefficient of variation which is constant over load, adhesive system, and replicates. To see that this might be the case, consider Figure B-1. In this figure, the log of the standard deviation of time-to-failure is displayed against the log of the corresponding means of time-to-failure. The numbers on the plot distinguish the adhesive systems. If indeed the true coefficients of variation (for which the data provide noisy estimates) are constant over all of these sets of data, then one would expect to see points falling on a straight line with unit slope. A straight-line least-squares fit is also given on Figure B-1. It has a slope of 1.14, which is not significantly different from 1. There is a great deal of scatter about this line; this is a reflection of the scatter in the coefficients of variation in Table B-1. But overall, the fit to a line of unit slope is quite good. One might note that all of the loads have been combined here, and that this linear behavior could be an artifact due to changes in means and variance with load. However, separate plots made for each load also show nearly linear behavior with unit slope, so it is not misleading to combine load as has been done here.

Table B-1. Sample statistics for the creep-rupture data

Adhesive System ^a	Load N (lbf)	Rep. No.	Data Points	Time-to-Failure		
				Mean, h	sd, h	CoV, %
TS1 (TS1-1)	24.9 (5.6)	1	8	0.61	0.03	4.2
TS1 (TS1-1)		2	8	0.67	0.05	7.8
TS2 (TS2-1)		1	8	2.71	0.14	5.3
TS2 (TS2-1)		2	7	1.46	0.46	31.7
TS2 (TS2-2)		3	8	2.47	0.53	21.4
TS2 (TS2-3)		4	8	2.95	0.26	8.7
LA (LA-1)		1	8	1.80	0.79	43.7
LA (LA-2)		2	8	0.96	0.14	14.5
LA (LA-2)		3	8	0.50	0.07	14.7
LA (LA-3)		4	7	0.81	0.06	7.9
LA (LA-3)	5	8	0.91	0.14	15.5	
TS1 (TS1-1)	21.8 (4.9)	1	8	1.00	0.08	7.9
TS1 (TS1-1)		2	8	1.22	0.07	5.9
TS2 (TS2-1)		1	6	4.16	0.17	4.1
TS2 (TS2-1)		2	8	2.29	0.59	25.6
TS2 (TS2-2)		3	8	3.43	1.07	31.1
TS2 (TS2-3)		4	8	4.58	0.25	5.5
LA (LA-1)		1	8	2.64	0.74	28.2
LA (LA-2)		2	8	1.65	0.26	15.7
LA (LA-2)		3	8	0.79	0.06	7.7
LA (LA-3)		4	8	1.06	0.08	8.0
LA (LA-3)	5	8	1.32	0.08	6.3	
TS1 (TS1-1)	18.7 (4.2)	1	8	1.67	0.11	6.6
TS1 (TS1-1)		2	8	2.00	0.19	9.6
TS2 (TS2-1)		1	8	7.10	0.73	10.3
TS2 (TS2-1)		2	8	4.29	1.19	27.7
TS2 (TS2-2)		3	8	7.17	1.19	16.6
TS2 (TS2-3)		4	8	7.91	1.09	13.8
LA (LA-1)		1	8	6.83	1.14	16.6
LA (LA-2)		2	8	2.88	0.32	11.1
LA (LA-2)		3	8	1.08	0.11	10.1
LA (LA-3)		4	8	1.51	0.15	9.9
LA (LA-3)	5	8	2.09	0.29	13.7	
TS1 (TS1-1)	15.6 (3.5)	1	8	3.19	0.30	9.5
TS1 (TS1-1)		2	8	3.82	0.33	8.7
TS2 (TS2-1)		1	8	12.01	0.48	4.0
TS2 (TS2-1)		2	8	8.34	1.55	18.6
TS2 (TS2-2)		3	8	12.20	1.75	14.3
TS2 (TS2-3)		4	8	15.26	4.87	31.9
LA (LA-1)		1	8	16.06	3.88	24.2
LA (LA-2)		2	8	6.45	0.83	12.8
LA (LA-2)		3	7	2.02	0.27	13.4
LA (LA-3)		4	8	2.27	0.23	10.3
LA (LA-3)	5	8	3.29	0.40	12.2	

^aThe 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).

Adhesive System	Load N (lbf)	Rep. No.	Data Points	Time-to-Failure		
				Mean, h	sd, h	CoV, %
TS1 (TS1-1)	12.5 (2.8)	1	8	7.07	0.96	13.6
TS1 (TS1-1)		2	8	10.45	1.63	15.6
TS2 (TS2-1)		1	8	26.42	2.83	10.7
TS2 (TS2-1)		2	8	18.31	2.35	12.8
TS2 (TS2-2)		3	5	27.88	2.83	10.2
TS2 (TS2-3)		4	8	29.28	3.68	12.6
LA (LA-1)		1	8	109.0	25.39	23.3
LA (LA-2)		2	8	17.55	3.24	18.5
LA (LA-2)		3	8	3.28	0.36	0.9
LA (LA-3)		4	8	3.72	0.29	7.8
LA (LA-3)		5	8	5.05	0.84	16.6
TS1 (TS1-1)	9.3 (2.1)	1	8	28.51	3.87	13.6
TS1 (TS1-1)		2	8	44.43	6.50	14.6
TS2 (TS2-1)		1	8	94.67	13.89	14.7
TS2 (TS2-1)		2	8	59.98	16.62	27.7
TS2 (TS2-2)		3	6	89.33	15.27	17.1
TS2 (TS2-3)		4	8	101.97	23.93	23.5
LA (LA-1)		1	8	516.63	249.29	48.3
LA (LA-2)		2	8	79.28	24.27	30.6
LA (LA-2)		3	8	6.95	1.45	20.8
LA (LA-3)		4	8	6.78	0.81	11.9
LA (LA-3)		5	8	8.79	1.30	14.8
TS1 (TS1-1)	6.2 (1.4)	1	14	237.2	43.14	18.2
TS1 (TS1-1)		2	8	302.0	32.25	10.7
TS2 (TS2-1)		1	14	640.6	141.4	22.1
TS2 (TS2-1)		2	8	565.4	126.9	23.6
TS2 (TS2-2)		3	8	616.4	124.3	21.2
TS2 (TS2-3)		4	7	823.4	66.55	8.5
LA (LA-1)		1	14	NF ^b	----	----
LA (LA-2)		3	8	27.47	5.16	18.8
LA (LA-3)		4	8	23.73	3.66	15.4
LA (LA-3)		5	8	38.74	8.08	20.8
TS1 (TS1-1)	3.1 (0.7)	1	14	NF	----	----
TS2 (TS2-1)		1	14	NF	----	----
LA (LA-1)		1	14	NF	----	----

^aThe 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).

^bNF indicates no failure; the elapsed time when this report was issued was over 8600 hours.

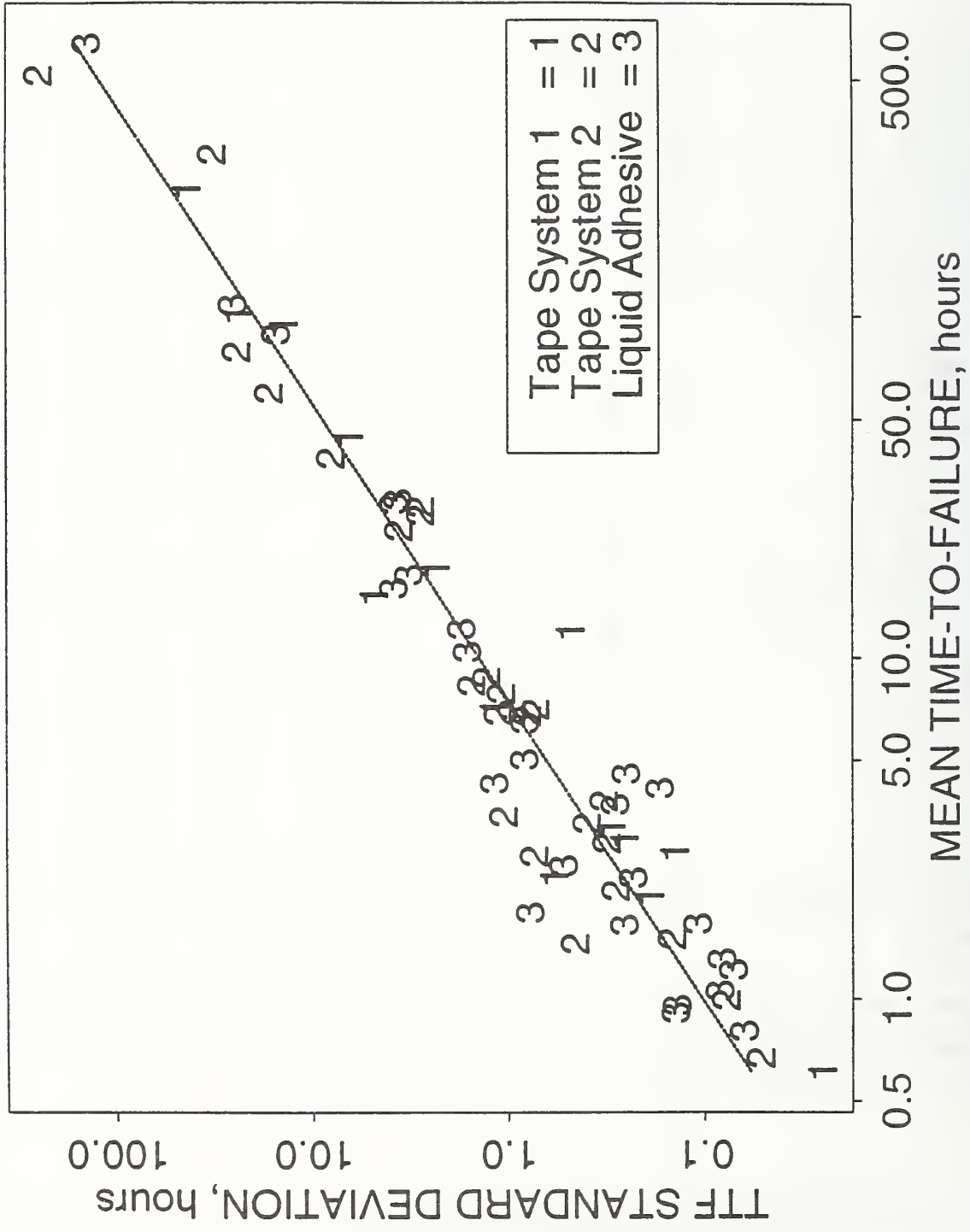


Figure B-1. Mean Versus Standard Deviation of Times-to-Failure for the Three Adhesive Systems; the Slope and the Intercept of the Least Squares Line are 1.14 and - 2.28, Respectively.

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