NAT'L INST. OF STAND & TECH R.I.C.
A11105 486829

NIST
PUBLICATIONS

NIST
United States Department of Commerce
Technology Administration
National Institute of Standards and Technology

NIST BUILDING SCIENCE SERIES 177

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

Walter J. Rossiter, Jr., Mark G. Vangel, Kevin M. Kraft

TA
435
U58
NO.177
1998

The National Institute of Standards and Technology was established in 1988 by Congress to "assist industry in the development of technology . . . needed to improve product quality, to modernize manufacturing processes, to ensure product reliability . . . and to facilitate rapid commercialization . . . of products based on new scientific discoveries."

NIST, originally founded as the National Bureau of Standards in 1901, works to strengthen U.S. industry's competitiveness; advance science and engineering; and improve public health, safety, and the environment. One of the agency's basic functions is to develop, maintain, and retain custody of the national standards of measurement, and provide the means and methods for comparing standards used in science, engineering, manufacturing, commerce, industry, and education with the standards adopted or recognized by the Federal Government.

As an agency of the U.S. Commerce Department's Technology Administration, NIST conducts basic and applied research in the physical sciences and engineering, and develops measurement techniques, test methods, standards, and related services. The Institute does generic and precompetitive work on new and advanced technologies. NIST's research facilities are located at Gaithersburg, MD 20899, and at Boulder, CO 80303. Major technical operating units and their principal activities are listed below. For more information contact the Publications and Program Inquiries Desk, 301-975-3058.

Office of the Director
- National Quality Program
- International and Academic Affairs

Technology Services
- Standards Services
- Technology Partnerships
- Measurement Services
- Technology Innovation
- Information Services

Advanced Technology Program
- Economic Assessment
- Information Technology and Applications
- Chemical and Biomedical Technology
- Materials and Manufacturing Technology
- Electronics and Photonics Technology

Manufacturing Extension Partnership Program
- Regional Programs
- National Programs
- Program Development

Electronics and Electrical Engineering Laboratory
- Microelectronics
- Law Enforcement Standards
- Electricity
- Semiconductor Electronics
- Electromagnetic Fields[1]
- Electromagnetic Technology[1]
- Optoelectronics[1]

Chemical Science and Technology Laboratory
- Biotechnology
- Physical and Chemical Properties[2]
- Analytical Chemistry
- Process Measurements
- Surface and Microanalysis Science

Physics Laboratory
- Electron and Optical Physics
- Atomic Physics
- Optical Technology
- Ionizing Radiation
- Time and Frequency[1]
- Quantum Physics[1]

Materials Science and Engineering Laboratory
- Intelligent Processing of Materials
- Ceramics
- Materials Reliability[1]
- Polymers
- Metallurgy
- NIST Center for Neutron Research

Manufacturing Engineering Laboratory
- Precision Engineering
- Automated Production Technology
- Intelligent Systems
- Fabrication Technology
- Manufacturing Systems Integration

Building and Fire Research Laboratory
- Structures
- Building Materials
- Building Environment
- Fire Safety Engineering
- Fire Science

Information Technology Laboratory
- Mathematical and Computational Sciences[2]
- Advanced Network Technologies
- Computer Security
- Information Access and User Interfaces
- High Performance Systems and Services
- Distributed Computing and Information Services
- Software Diagnostics and Conformance Testing

[1]At Boulder, CO 80303.
[2]Some elements at Boulder, CO.

NIST BUILDING SCIENCE SERIES 177

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

Walter J. Rossiter, Jr.

Building and Fire Research Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899-0001

Mark G. Vangel

Statistical Engineering Division
National Institute of Standards and Technology
Gaithersburg, MD 20899-0001

Kevin M. Kraft

Building and Fire Research Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899-0001

July 1998

U.S. Department of Commerce
William M. Daley, *Secretary*

Technology Administration
Gary R. Bachula, *Under Secretary for Technology*

National Institute of Standards and Technology
Raymond G. Kammer, *Director*

National Institute of Standards and Technology Building Science Series 177
Natl. Inst. Stand. Technol. Bldg. Sci. Ser. 177, 54 pages (July 1998)
CODEN: NBSSES

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 1998

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402–9325

ABSTRACT

This report presents the results of the Phase III research of an industry-government consortium study to develop nonproprietary data on the performance of tape-bonded seams of EPDM (ethylene propylene diene terpolymer) roof membranes. Four Phase III tasks investigated the effects of: (I) elevated test temperatures, (II) elevated temperature exposure prior to loading, (III) exposure to industry-developed protocols (i.e., Rubber Manufacturers Association and SPRI), and (IV) cold temperature preparation on peel creep-rupture response and on peel strength. Task (V) examined shear testing. Two commercial tape systems (i.e., tape and primer) and one liquid adhesive were applied to well-cleaned EPDM rubber in fabricating specimens. For each task, comparisons of the creep-rupture responses and strengths of tape-bonded and liquid-adhesive-bonded samples were made. Conclusions regarding the significant comparisons of the creep-rupture responses are as follows:

- In Task I, as the temperature and load of the creep test increased, peel times-to-failure of the three adhesive systems decreased. For any combination of temperature and load, the tape-bonded sample sets had longer mean peel times-to-failure than did the liquid-adhesive-bonded sample sets.
- In Task II, when exposed to elevated temperatures for varying times, peel times-to-failure of liquid-adhesive-bonded samples were either unaffected or increased versus that of the room temperature control. In comparison, peel times-to-failure of one tape-bonded system increased, and that of the other tape-bonded system decreased or were unaffected depending upon the exposure.
- In Task III under the RMA protocol, mean peel times-to-failure of liquid-adhesive-bonded samples increased upon exposure. In comparison, one tape system showed increased peel times-to-failure, and the other exhibited decreased peel times-to-failure. In the case of the SPRI protocol, mean peel times-to-failure of liquid-adhesive-bonded samples increased upon exposure. In comparison, one tape system was unchanged, whereas the other decreased.
- In Tasks II and III, where increased peel times-to-failure were observed, under some conditions, they were quite substantial; however, no evidence is available indicating that tape-bonded field seams (i.e., those sampled from roofs) have shown such prolonged peel times-to-failure. Also, where decreases in peel times-to-failure were observed for laboratory-exposed samples, the resultant mean times-to-failure were not atypical of values measured for some field seams.
- In Task IV, in the case of laboratory-prepared samples, mean peel times-to-failure of the liquid-adhesive-bonded system decreased upon cold temperature preparation. In comparison, mean peel times-to-failure of one tape system also decreased; those of the other tape-system were unaffected. Where decreases occurred at the coldest laboratory preparation temperature, the mean peel time-to-failure of the tape-bonded specimens was greater than that of the liquid-adhesive-bonded specimens. In the case of field-prepared samples, the tape-bonded samples and the liquid-adhesive-bonded sample sets had comparable mean peel times-to-failure. For both adhesive types, these values for the field samples were less than those of the samples prepared in the laboratory at cold temperatures.
- In Task V, many shear creep-rupture tests, particularly those at room temperature, produced few failures within the allotted test time. At 70 °C (158 °F) and loads of 24.9 kN/m and 28.0 kN/m (5.6 lbf/in and 6.3 lbf/in), both tape systems had shorter shear-creep lifetimes than the liquid adhesive system.

Key words: adhesive tape; adhesive testing; building technology; creep-rupture; EPDM membrane; heat exposure; roofing; seam; strength; time-to-failure

TABLE OF CONTENTS

LIST OF TABLES

Page

LIST OF FIGURES

1. INTRODUCTION

1.1 Background

Since the early 1990s, the use of preformed tapes for fabricating field seams of EPDM membranes has increased such that they are supplanting contact-type liquid adhesives for many applications [1]. This report presents the results of Phase III of an industry-government consortium study undertaken to: (1) compare the creep-rupture performance of tape-bonded and liquid-adhesive-bonded seams of EPDM membranes, and (2) recommend a test protocol and criteria for evaluating creep-rupture performance of such seams. The consortium study was initiated at the National Institute of Standards and Technology (NIST) in late 1994 in response to industry requests that independent evaluations be conducted and that nonproprietary data be developed on the performance of tape-bonded seams [2].

Two EPDM membrane manufacturers (Carlisle and Firestone), and two tape-system manufacturers (Adco and Ashland) along with two trade associations (NRCA and RCI)* joined with NIST through a Cooperative Research and Development Agreement (CRADA) to design and conduct Phase III of the study. The experimental program has consisted of three 1-year phases. A summary of the objective of each phase is as follows:

- In Phase I, the peel creep-rupture response (time-to-failure) of tape-bonded seam specimens subjected to various loads under ambient conditions was compared to that of liquid-adhesive-bonded specimens (Section 1.2).
- In Phase II, the peel creep-rupture response and peel strength of tape-bonded seam specimens were investigated under ambient conditions for a number of material and application variables (Section 1.3).
- In Phase III, which is the subject of this report, the peel creep-rupture response and peel strength of tape-bonded seam specimens were investigated as functions of test temperature, exposure of the specimens, and cold temperature application. In addition, shear-creep and shear-strength tests were conducted to complement tests conducted in peel, as seams in service experience both peel and shear stresses.

In the creep-rupture experiments, seam specimens of a fixed length are stressed under a constant load and the time over which they sustain the load until total separation (i.e., the time-to-failure) is recorded. Results from previous NIST studies indicate that creep-rupture tests provide a sensitive procedure for evaluating the effects of a multiplicity of application and environmental factors for both tape-bonded and liquid-adhesive-bonded seams. Such factors include EPDM surface condition, use of tape primer, adhesive thickness, temperature, and the effect of ozone on the capability of seams to sustain loads over time [3-10].

1.2 Phase I Findings

The results of Phase I were published in NIST Building Science Series (BSS) 175, "Performance of Tape-Bonded Seams of EPDM Membranes: Comparison of the Peel Creep-Rupture Response of Tape-Bonded and Liquid-Adhesive-Bonded Seams" [3]. Peel specimens were prepared at room temperature, 23 °C ± 2 °C (73 °F ± 4 °F),** using two commercial tape systems (i.e., tape and

---

*The National Roofing Contractors Association (NRCA) and the Roof Consultants Institute (RCI).

**Temperature variations in the report are absolute bounds.

primer) and one commercial liquid adhesive. In all cases, the EPDM rubber was well cleaned and, in the case of the tape-bonded specimens, a primer was applied. Seam specimens were tested for peel strength and for peel creep-rupture resistance (i.e., times-to-failure) under loads ranging from 3.1 N to 24.9 N (0.7 lbf to 5.6 lbf)* in increments of 3.1 N (0.7 lbf). Figure 1 shows a plot of mean time-to-failure versus load for the Phase I experiments [3]. No data points are shown for the 3.1 N (0.7 lbf) load, because no specimen failures were observed at this load. The tape-bonded sample sets had times-to-failure that were, in most cases, comparable to or greater than those of the liquid-adhesive-bonded sample sets.

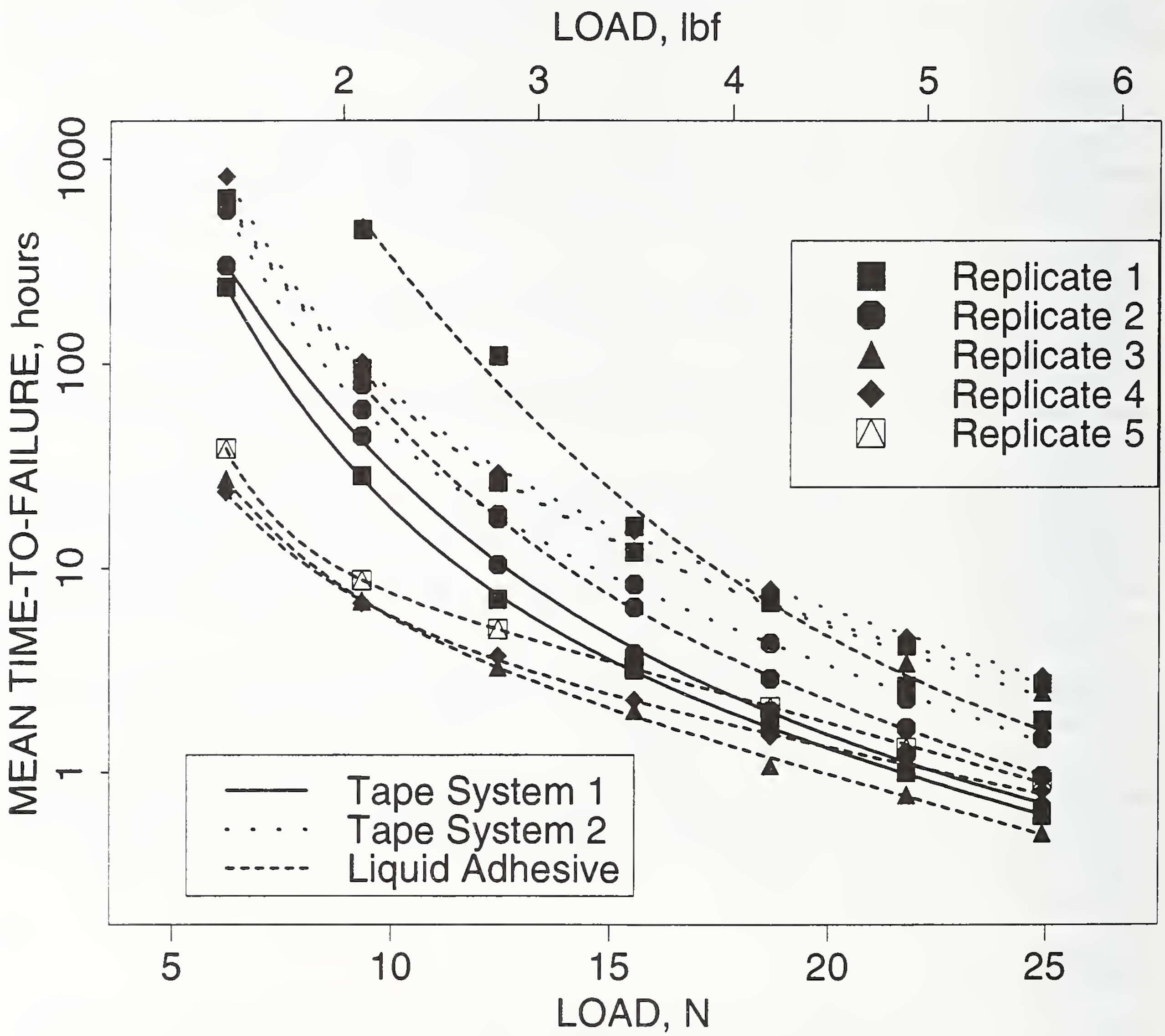

Figure 1. Mean Peel Time-to-Failure Versus Load for the Tape-Bonded and Liquid-Adhesive-Bonded Specimens Investigated in Phase I [3].

---

*These loads correspond to stresses of 0.12 kN/m^2 to 0.98 kN/m^2 (0.7 lbf/in^2 to 5.6 lbf/in^2).

1.3 Phase II Findings

The results of Phase II were published in NIST Building Science Series (BSS) 176, "Performance of Tape-Bonded Seams of EPDM Membranes: Effect of Material and Application Factors on Peel Creep-Rupture Response" [4]. In the Phase II study, seam specimens were prepared using the same commercial tape systems that were used in Phase II. The study was statistically designed to examine the effect of two material factors (tape system and tape thickness, i.e., "standard"* and thin) and five application factors (EPDM surface condition, primer, application temperature and pressure, and time-at-temperature). Figure 2 is a plot of mean time-to-failure for the combinations of application factors [4]. The plot characters identify the combinations of material factors. The horizontal axis specifies the levels of the five application factors. The application factor combinations are ordered in increasing mean response. The mean for the four sample sets (i.e., TS1-thin, TS1-"standard", TS2-thin, and TS2-"standard") prepared at each combination of application factors is indicated by the dotted line. Also included in Figure 2 is a horizontal dashed line that represents the average time-to failure for the three sets of liquid-adhesive-bonded specimens, fabricated with industry-recommended adhesive thickness and clean EPDM, that had the lowest average times-to-failure at 9.3 N (2.1 lbf) in Phase I (fig. 1). From the Phase II experimentation, it was concluded that, for the tape-bonded samples, primed, clean EPDM provided the longest times-to-failure (and highest peel strengths). In addition, primed, clean EPDM and "standard thickness tape" afforded times-to-failure that were statistically higher than minimum mean times-to-failure of well prepared liquid-adhesive-bonded specimens investigated in Phase I. Also, application temperatures and application pressures used in the investigation did not affect the times-to-failure of tape-bonded specimens prepared with primed, clean EPDM.

1.4 Objective and Scope of Phase III

In Phase III, five research tasks were selected for study by the Consortium Oversight Committee members. The first four tasks investigated effects on peel creep-rupture response and on peel strength due to: (I) elevated test temperatures, (II) elevated temperature exposure prior to loading, (III) exposure to industry-developed protocols, and (IV) cold temperature preparation. Task (V) examined shear testing. Each task was judged to be important to the broad characterization of the creep-rupture behavior of tape-bonded seams and to complement the creep-rupture and strength data developed in Phases I and II. Moreover, the results from these five Phase III investigations would provide guidance as to factors to be incorporated in a test protocol for evaluating the creep resistance of EPDM adhesive-bonded seams. Table 1 lists the five tasks with comments as to why each was selected for study. This report treats each task separately in presenting and discussing the data obtained (Sections 3-7). To compare the creep-rupture performance of tape-bonded and liquid-adhesive-bonded seams, each task incorporated tests of seam specimens fabricated using both tape and liquid adhesives.

---

*The thicker tapes had thicknesses typical of those commercially available at the time of the Phase II study; thus, they were designated as having "standard" thickness.

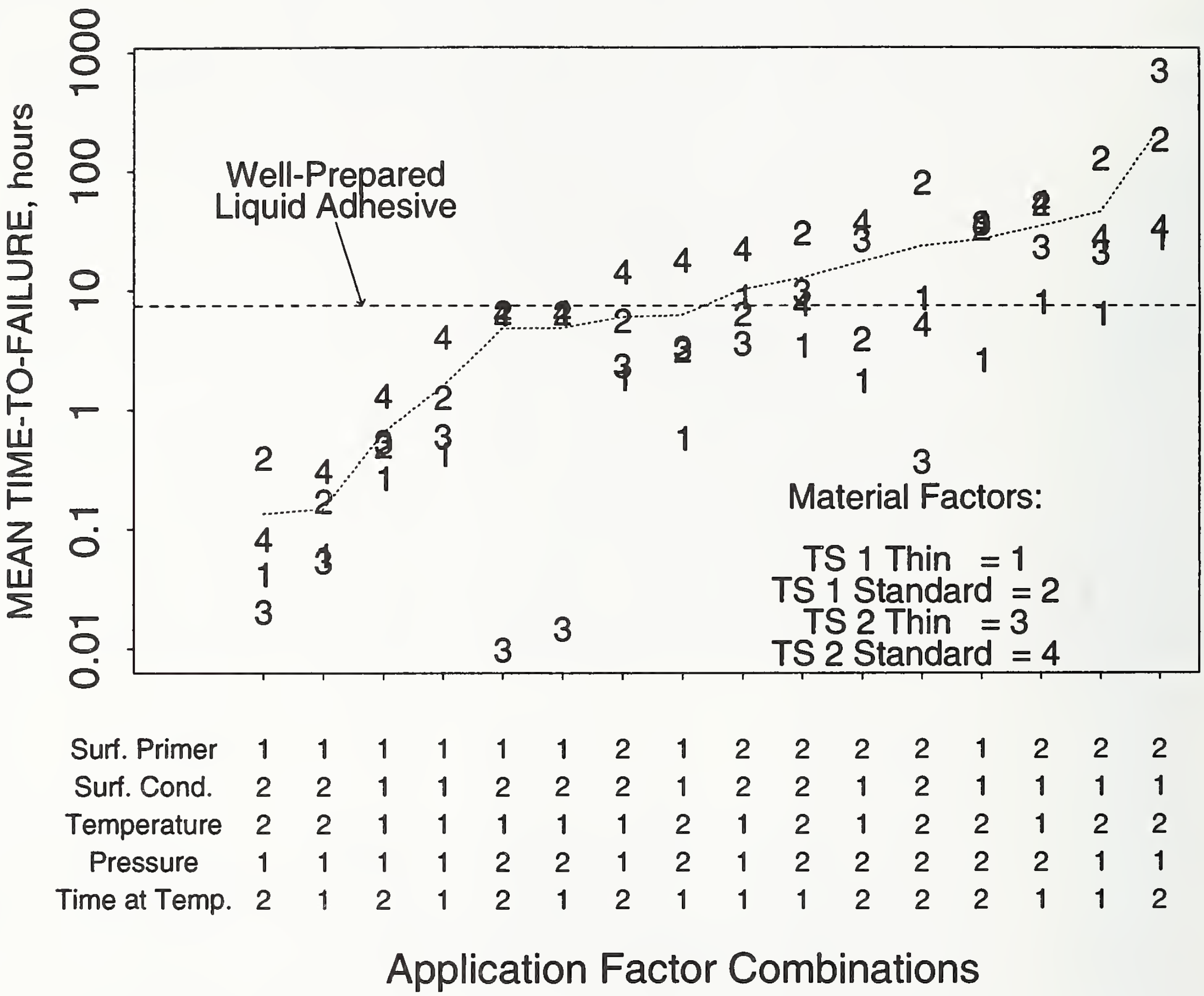

Figure 2. Mean Peel Time-to-Failure Versus Combinations of Application Factors Investigated in Phase II [4]. (Primer: Unprimed = 1, Primed = 2; EPDM Surface Condition: Clean = 1, Contaminated = 2; Application Temperature: Low = 1, High = 2; Application Pressure: Low = 1, High = 2; Time-at-Temperature: Short = 1, Long = 2.) The horizontal dashed line represents the mean time-to-failure of the three sets of "well prepared," liquid-adhesive-bonded specimens (i.e., those fabricated with industry-recommended adhesive thickness and clean EPDM) that had the lowest mean times-to-failure at the 9.3 N (2.1 lbf) load in Phase I (see fig. 1). The dotted line represents the mean for the four sample sets of tape-bonded specimens prepared at each combination of application factors.

Table 1. Tasks selected for study in the Phase III research

Task No.	Research Task	Selection of the Research Task
I	Effect of Elevated Test Temperatures on Peel Time-to-Failure	In Phases I and II, peel times-to-failure and peel-strength measurements were performed at room temperature. Characterization of seams stressed at elevated temperatures is important because, in service, EPDM membranes are subjected to elevated temperatures that can exceed 70 °C (158 °F).
II	Effect of Elevated Temperature Exposure Prior to Loading on Peel Time-to-Failure	In Phases I and II, the peel times-to-failure and peel-strength measurements were performed on specimens that, in general, were not exposed to conditions such as elevated temperatures that might adversely affect their peel creep-resistance and peel strength. Characterization of seams after such exposure is important because EPDM membranes in service may reach temperatures of 70 °C (158 °F) or more for prolonged periods.
III	Effect of Exposure to Industry-Developed Protocols on Peel Time-to-Failure	As indicated for Task II, in Phases I and II, the peel times-to-failure and peel-strength measurements were performed on specimens that, in general, were not exposed to elevated temperatures or other weather-related conditions. Characterization of seams after exposure to factors such as water and temperature cycling is important because seams are exposed to such factors in service. Two laboratory exposure protocols for seam specimens have been developed by the EPDM roofing industry, and are referred to as the RMA [11] and SPRI [12] procedures.[a] The two protocols (see Table 9) are similar in that they expose specimens to dry heat in an oven, heat in water, and freeze-thaw cycling. A difference between the two is that the SPRI protocol subjects the specimen to a mechanical load in shear during some exposures.
IV	Effect of Cold Temperature Preparation on Peel Time-to-Failure	In Phase II, peel times-to-failure and peel-strength measurements were performed on specimen sets that were prepared at 5 °C (41 °F) [4]. Phase II experimental limitations did not allow for sample preparation in the NIST laboratory at lower temperatures. Although 5 °C (41 °F) is relatively low, field experience has shown that EPDM membranes are at times installed in cold weather at lower temperatures, even those below 0 °C (32 °F). Characterization of seams prepared at low temperatures is important to quantify the effect of "cold weather" application on peel creep-rupture resistance and peel strength.
V	Shear Testing	In Phases I and II, times-to-failure and strength measurements were performed in peel. Characterization of seams stressed in shear is important because EPDM membranes in service may be subjected to shear stresses. Experience with earlier-generation, non-cured tape-bonded seams has shown that failures sometimes occurred due to poor creep-resistance in shear [13].

[a]RMA indicates the Rubber Manufacturers Association; SPRI is an association of sheet membrane and component suppliers to the commercial roofing industry.

2. EXPERIMENTAL

2.1 Materials and Specimen Preparation

Two commercial tape adhesive 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 for seam sample preparation. The EPDM sheet was a commercial product having a thickness of about 1.5 mm (0.060 in). The adhesives, tape primers, and EPDM were the same brand name products used in Phases I and II. However, the adhesives and primers were from different lots. The surface of the EPDM was well cleaned before bonding. The specimen preparation procedures have been previously described [2,3,5]. For Tasks I through IV, peel specimens had dimensions of 25 mm by 125 mm (1 in by 5 in) with a 75 mm (3 in) bond beginning from one end of the long dimension. For Task V, shear specimens had dimensions of 25 mm by 113 mm (1 in by 4.5 in) with a 25 mm (1 in) bond located at the specimen center. For each research task, a sufficient number of specimens was prepared to conduct all planned creep-rupture and strength tests. All specimens had a minimum age of 28 days when tested.

2.2 Creep-Rupture Tests

For each creep-rupture test at a given load and test temperature, eight replicates were randomly selected from the specimen set prepared for the research task. Details on the selected loads and test temperatures are given in the report sections that describe the five tasks. The creep-rupture tests were conducted in laboratory-constructed chambers according to the general procedure described in Martin, Embree, Stutzman, and Lechner [6]. For tests at room temperature, 23 °C ± 2 °C (73 °F ± 4 °F), the relative humidity was maintained between 40 % and 45 % using a saturated potassium carbonate solution [14]. Specimens were conditioned for a minimum of 16 h at the above-stated condition before applying the creep load. For tests at elevated temperatures, 40 °C ± 1 °C and 70 °C ± 1 °C (104 °F ± 2 °F and 158 °F ± 2 °F),* humidity was not controlled. Specimens were heated from room temperature to these test temperatures in approximately 0.5 h and 1.5 h, respectively. They were held for an additional 0.5 h at the test temperature before the creep load was applied. After testing, each specimen was visually examined and the mode of failure, adhesive, cohesive, or mixed, noted.**

2.3 Peel-Strength Tests

Four specimens were randomly selected from each sample set for each research task, and the peel or shear strengths were determined at a crosshead rate of 50 mm/min (2 in/min). Depending on the task, three test temperatures were used: 23 °C, 40 °C, and 70 °C (73 °F, 104 °F, and 158 °F). The Instron Model 1125 universal testing machine*** was equipped with hardware and software for

---

*For clarity, the test temperatures are generally referred to as 23 °C, 40 °C, and 70 °C (73 °F, 104 °F, and 158 °F) in the report.

**Failure was classified as either adhesive or cohesive according to definitions given in ASTM D 907. Adhesive failure: rupture of an adhesive bond such that the separation appears to be at the adhesive-adherend interface. Cohesive failure: rupture of an adhesive bond such that the separation appears to be within the adhesive.

***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.

recording and calculating strength data. Similar to the creep-rupture tests, each failed specimen was visually examined and the mode of failure was noted.

2.4 Data Presentation

In presenting strength data in Sections 3 through 7, the means in the summary tables are arithmetic means, and they are presented on linear axes in the plots. The error bars in the plots represent 95 % confidence intervals on the means. In presenting the creep-rupture data, the mean times-to-failure in the tables are also arithmetic means. This allows comparison of the Phase III peel creep-rupture data with the peel creep-rupture results tabulated in the Phase I and Phase II reports [3,4] (which were also presented as arithmetic means). However, the 95 % confidence levels for the peel creep-rupture data in the present report were calculated on a (natural) logarithmic scale and, consequently, the mean peel times-to-failure indicated in the plots are geometric means. Recall that the geometric mean ($\bar{x}_g$) of n-data points is the n-th root of the product of the n values (x_n) (eq (1)). Typically, the geometric mean of a sample set is close to the arithmetic mean. However, when the time-to-failure values are highly variable, or are close to zero, these two means can differ substantially. The arithmetic mean sometimes does not provide a reasonable estimate of a "typical" lifetime in cases where the creep data vary over several orders of magnitude, since this mean can be highly influenced by the longest lifetimes in the sample set. The geometric mean usually provides a more reasonable estimate. Calculating confidence intervals on a logarithmic scale (as was done in Sections 3 through 6) has the advantage of providing non-negative intervals of nearly equal width. This aids in data interpretation and presentation, particularly in making comparisons among data sets.

$$\bar{x}_g = (x_1 \cdot x_2 \cdots x_n)^{1/n} \qquad (1)$$

3. EFFECT OF ELEVATED TEST TEMPERATURES ON PEEL TIME-TO-FAILURE (TASK I)

3.1 Objective and Scope of Task I

The Task I investigation was conducted to evaluate the effect of elevated test temperature on peel-creep resistance and peel strength, and to compare the performance of tape-bonded and liquid-adhesive-bonded sample sets tested at the elevated temperatures. Peel strengths of tape-bonded and liquid-adhesive-bonded specimens were measured at 23 °C, 40 °C, and 70 °C (73 °F, 104 °F and 158 °F). Peel creep-rupture tests were performed at these temperatures under each of three loads, 9.3 N, 12.5 N, and 15.6 N (2.1 lbf, 2.8 lbf, and 3.5 lbf). Additionally, at 70 °C (158 °F), a creep-rupture test was performed under a 6.2 N (1.4 lbf) load. Table 2 summarizes the Task I test conditions and lists the number of specimens tested at each condition. Tests conducted at 23 °C (73 °F) were considered to be controls.

3.2 Task I Results and Discussion

Tables 3 and 4 summarize the peel-strength and peel creep-rupture data for Task I, respectively. Figure 3 is a plot of mean peel strength versus adhesive system for the three test temperatures. Figure 4 (A, B, and C) is a plot of the mean time-to-failure results for the three adhesive systems. Polynomial models with load and temperature as variables were fit to summarize the time-to-failure results. The models were fit separately to data for the three adhesive systems. The simplest model fitting the time-to-failure (ttf) data was:

$$\log(ttf) = C_o + C_1 \cdot L + C_2 \cdot L^2 + C_3 \cdot T + C_4 \cdot T^2 + C_5 \cdot T \cdot L \qquad (2)$$

where L denotes load, T is temperature, and C_0 through C_5 are coefficients (Table 5) estimated by least squares. The fitting of this model was used to produce the curves and error bars in Figure 4.

Table 2. Summary of test conditions and number of specimens for Task I

Adhesive System	Test Temp. °C (°F)	Peel-Strength Tests No. of Specimens	Peel Creep-Rupture Tests: No. of Specimens — Load, N (lbf) 15.6 (3.5)	12.5 (2.8)	9.3 (2.1)	6.2 (1.4)
TS1	23 (73)	4	8	8	8	---[a]
	40 (104)	4	8	8	8	---
	70 (158)	4	8	8	8	8
TS2	23 (73)	4	8	8	8	---
	40 (104)	4	8	8	8	---
	70 (158)	4	8	8	8	8
LA	23 (73)	4	8	8	8	---
	40 (104)	4	8	8	8	---
	70 (158)	4	8	8	8	8

[a]The dash indicates that tests were not conducted.

Both the peel-strength and peel creep-rupture specimens generally failed cohesively in Task I. An exception was TS2 subjected to creep tests at 23 °C (73 °F), wherein some failures were mixed. In these cases, the majority of the bond area failed cohesively.

3.2.1 Effect of Elevated Test Temperature on the Adhesive Systems. From Table 3 and Figure 3, the following observations on peel strength are noted:

- For the three adhesive systems, mean peel strengths determined at 23 °C (73 °F) were comparable to those measured in Phase I. For typical TS1, TS2, and LA sample sets in Phase I, the mean values were 1.86 kN/m, 2.32 kN/m, and 1.87 kN/m, respectively [3]. In comparison, in Task I, they were 1.74 kN/m, 2.14 kN/m, and 1.77 kN/m, respectively. This comparison indicates that the peel-strength data for the three adhesive systems were reproducible when different batches of samples were fabricated about two years apart using different batches of adhesives and primers.
- For the three adhesive systems, the mean peel strength tended to decrease linearly with increasing temperature. The lines in Figure 3 are least-squares fits to the data. The r^2-values for the fits were: 0.97, 0.93, and 0.96, respectively. The effect of temperature on peel strength was not unexpected because viscoelastic materials are known to display a strong temperature dependence [15].

From Table 4 and Figure 4, the following observations on peel creep-rupture behavior are noted:

- For the three adhesive systems, the mean time-to-failure decreased with an increase in temperature and load, although the decrease was slight for TS2 between 23°C and 40°C (73°F and 104°F).
- The quadratic model (eq (2)) fit the data well, as evidenced by the closeness of the data points to the curves. Note also that curvature is present in all curves in Figures 4A, 4B, and 4C.

Table 3. Summary of peel-strength data for Task I

Adhesive System	Temp. °C	Temp. °F	Peel Strength, kN/m min	max	mean[a]	sd[b]	Peel Strength, lbf/in min	max	mean[a]	sd[b]	CoV[c] %	Failure Mode[d]
TS1	23	73	1.73	1.76	1.74	0.01	9.9	10.0	9.9	0.06	0.6	1
	40	104	1.15	1.22	1.19	0.03	6.5	7.0	6.8	0.19	2.7	1
	70	158	0.66	0.68	0.67	0.01	3.8	3.9	3.8	0.04	1.0	1
TS2	23	73	2.06	2.19	2.14	0.06	11.8	12.5	12.2	0.32	2.7	1
	40	104	1.42	1.85	1.70	0.20	8.1	10.6	9.7	1.14	11.7	1
	70	158	1.07	1.16	1.13	0.04	6.1	6.6	6.4	0.21	3.3	1
LA	23	73	1.70	1.92	1.77	0.10	9.7	11.0	10.1	0.59	5.9	1
	40	104	1.03	1.08	1.06	0.02	5.91	6.15	6.05	0.10	1.70	1
	70	158	0.31	0.41	0.36	0.04	1.76	2.32	2.05	0.24	11.86	1

[a]Mean of four determinations. [b]sd indicates standard deviation. [c]CoV indicates coefficient of variation.
[d]Failure mode: 1 = cohesive.

Table 4. Summary of peel time-to-failure data for Task I

Adhesive System	Temp. °C	Temp. °F	Load N	Load (lbf)	Time-To-Failure, h min	max	mean[a]	sd[b]	CoV[c] %	Failure Mode[d]
TS1	23	73	9.3	(2.1)	83.66	95.05	90.34	3.76	4.2	1
			12.4	(2.8)	17.33	25.64	20.94	2.70	12.9	1
			15.6	(3.5)	5.02	7.46	6.02	0.85	14.1	1
	40	104	9.3	(2.1)	8.74	9.81	9.25	0.38	4.1	1
			12.4	(2.8)	1.93	2.29	2.14	0.13	6.1	1
			15.6	(3.5)	0.71	0.82	0.76	0.05	6.5	1
	70	158	6.2	(1.4)	3.78	4.94	4.10	0.38	9.2	1
			9.3	(2.1)	0.58	0.70	0.62	0.05	7.4	1
			12.4	(2.8)	0.16	0.20	0.18	0.01	7.4	1
			15.6	(3.5)	0.05	0.10	0.07	0.01	17.7	1
TS2	23	73	9.3	(2.1)	47.52	94.96	63.40	14.61	23.0	1(6), 3(2)
			12.4	(2.8)	15.47	26.52	19.31	3.53	18.3	1(7), 3 (1)
			15.6	(3.5)	7.98	10.75	9.08	0.94	10.3	1(7), 3(1)
	40	104	9.3	(2.1)	21.20	47.99	30.26	10.81	35.7	1
			12.4	(2.8)	11.72	22.17	14.28	3.37	23.6	1
			15.6	(3.5)	6.70	8.97	7.31	0.72	9.8	1
	70	158	6.2	(1.4)	7.13	10.30	8.33	1.12	13.4	1
			9.3	(2.1)	2.74	4.76	3.35	0.68	20.2	1
			12.4	(2.8)	1.48	1.99	1.71	0.15	8.6	1
			15.6	(3.5)	0.35	1.27	0.96	0.27	28.5	1
LA	23	73	9.3	(2.1)	5.37	20.38	12.04	4.43	36.8	1
			12.4	(2.8)	3.19	6.56	4.89	1.27	26.1	1
			15.6	(3.5)	1.73	2.79	2.34	0.33	14.2	1
	40	104	9.3	(2.1)	0.96	2.17	1.59	0.45	28.5	1
			12.4	(2.8)	0.39	0.86	0.65	0.168	25.9	1
			15.6	(3.5)	0.23	0.40	0.30	0.05	17.2	1
	70	158	6.2	(1.4)	0.22	5.33	1.00	1.76	177	1
			9.3	(2.1)	0.03	0.08	0.06	0.01	25.3	1
			12.4	(2.8)	0.03	0.06	0.04	0.01	33.5	1
			15.6	(3.5)	0.01	0.04	0.02	0.01	37.0	1

[a]Mean of eight determinations. [b]sd indicates standard deviation. [c]CoV indicates coefficient of variation.
[d]Failure mode: 1 = cohesive; 3 = mixed cohesive/adhesive; numbers in parentheses indicate the number of specimens in the sample set that experienced the given failure mode; where there are no parentheses, all specimens failed by the given mode.

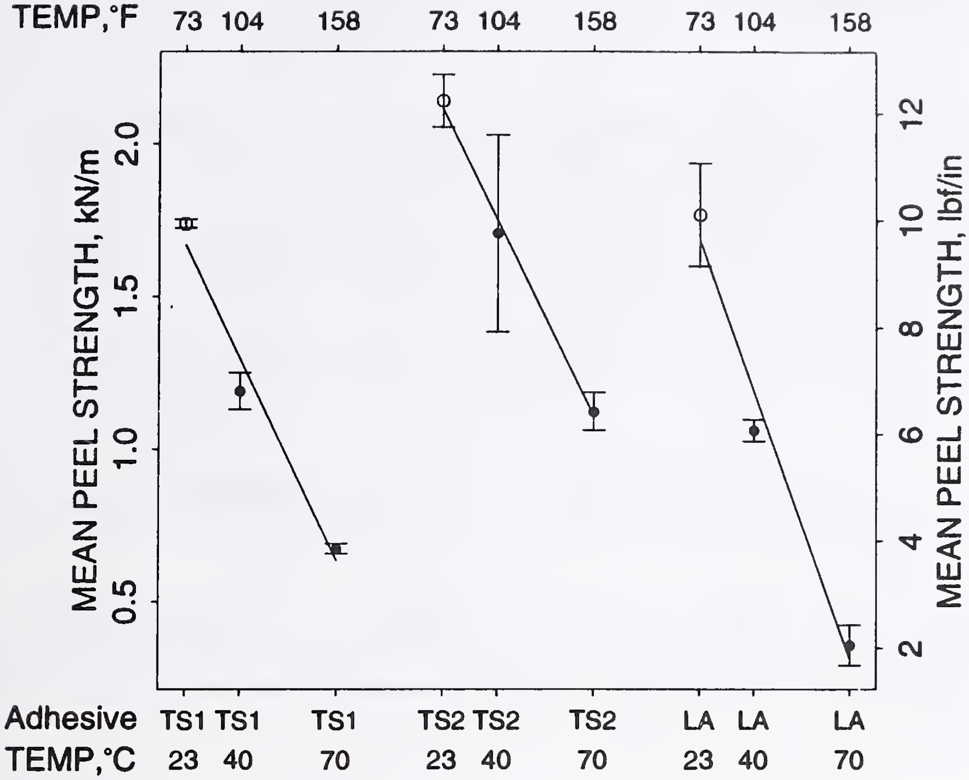

Figure 3. Mean Peel Strength as a Function of Temperature for Each Adhesive System.

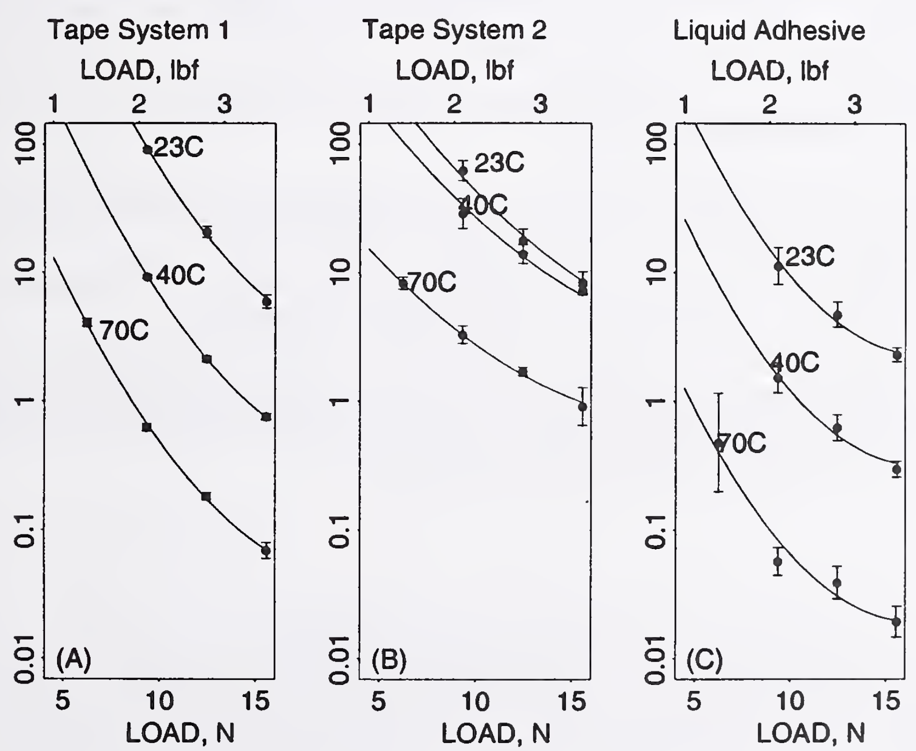

Figure 4. Mean Peel Time-to-Failure as a Function of Temperature and Load for Each Adhesive System.

Table 5. Coefficients for equation 1

Units	Adhesive Type	Coefficients[a,b]					
		C_0	C_1	C_2	C_3	C_4	C_5
SI	TS1	16.090 (0.3285)	-1.0150 (0.04483)	0.02201 (0.001602)	-0.2107 (0.005713)	0.000988 (0.000051)	0.001479 (0.000230)
	TS2	8.596 (0.7758)	-0.6258 (0.1058)	0.01114 (0.003781)	0.01511 (0.01349)	-0.001030 (0.000120)	0.002215 (0.000538)
	LA	12.540 (1.455)	-0.9977 (0.1993)	0.02898 (0.007124)	-0.1579 (0.02537)	0.000495 (0.000227)	0.000612 (0.001019)
Inch-Pounds	TS1	20.150 (0.4074)	-4.631 (0.2125)	0.4354 (0.03169)	-0.1366 (0.004075)	0.0003052 (0.000015)	0.003656 (0.000568)
	TS2	8.002 (0.9622)	-2.959 (0.5017)	0.2203 (0.07482)	0.02873 (0.009615)	-0.0003178 (0.000037)	0.005474 (0.001330)
	LA	15.510 (1.8030)	-4.486 (0.9448)	0.5734 (0.1410)	-0.09751 (0.01811)	0.0001528 (0.000070)	0.001514 (0.002517)

[a]Numbers in parentheses are one standard deviation. [b]The units of the coefficients, C_0, C_1, C_2, C_3, C_4, and C_5, are unitless, reciprocal load (in N or lbf), reciprocal load squared (in N or lbf), reciprocal temperature (in °C or °F), reciprocal temperature squared (in °C or °F), and reciprocal temperature times load (in °C·N or °F·lbf), respectively.

3.2.2 Comparison of Task I Tape-Adhesive and Liquid-Adhesive Results. From Table 3 and Figure 3, the following observation comparing the Task I peel-strength results for the tape-bonded and liquid-adhesive-bonded sample sets systems is noted:

- As the temperature increased, LA decreased in peel strength to a greater extent than did either TS1 or TS2, which performed about the same.

From Table 4 and Figure 4, the following observations comparing the Task I peel creep-rupture results for the tape-bonded and liquid-adhesive-bonded sample sets are noted:

- For any combination of test temperature and load, the times-to-failure of TS1 and TS2 were greater than that of LA. The differences were statistically significant.
- When temperature increased from 23 °C to 40 °C (73 °F to 104 °F) for a given load, TS2 showed less decrease in time-to-failure than LA; TS1 and LA had about the same decrease. When temperature further increased from 40 °C to 70 °C (104 °F to 158 °F) for a given load, both TS1 and TS2 experienced less decrease in time-to-failure than LA.

4. EFFECT OF ELEVATED TEMPERATURE EXPOSURE PRIOR TO LOADING ON PEEL TIME-TO-FAILURE (TASK II)

4.1 Objective and Scope of Task II

The Task II investigation was conducted to determine peel-creep resistance and peel strength after heat exposure in the laboratory, and to compare the performance of tape-bonded and liquid-adhesive-bonded sample sets after the heat exposures. Tape-bonded and liquid-adhesive-bonded samples were heated in forced air ovens at temperatures of 60 °C, 75 °C, and 90 °C (140 °F, 167 °F, and 194 °F) for 30 days, 90 days, and 150 days (± 1 day). After exposure, peel strength and peel time-to-failure were measured at 23 °C (73 °F). The creep load was 9.3 N (2.1 lbf). Peel strength and peel time-to-failure were also determined for room temperature control sample sets. Table 6 summarizes the Task II test conditions and lists the number of specimens tested.

Table 6. Summary of test conditions and number of specimens for Task II

Adhesive System	Exposure Temp.		Peel-Strength Tests: No. of Specimens Heat Exposure Period				Creep-Rupture Tests: No. of Specimens Heat Exposure Period			
	°C	°F	control[a]	30 days	90 days	150 days	control[a]	30 days	90 days	150 days
TS1	23	73	4	---[b]	---	---	8	---	---	---
	60	140	---	4	4	4	---	8	8	8
	75	167	---	4	4	4	---	8	8	8
	90	194	---	4	4	4	---	8	8	8
TS2	23	73	4	---	---	---	8	---	---	---
	60	140	---	4	4	4	---	8	8	8
	75	167	---	4	4	4	---	8	8	8
	90	194	---	4	4	4	---	8	8	8
LA	23	73	4	---	---	---	8	---	---	---
	60	140	---	4	4	4	---	8	8	8
	75	167	---	4	4	4	---	8	8	8
	90	194	---	4	4	4	---	8	8	8

[a]Control specimens were kept at room temperature. [b]The dash indicates that exposures were not performed.

4.2 Task II Results and Discussion

Tables 7 and 8 summarize the peel-strength and peel creep-rupture data for Task II, respectively. Figures 5 and 6 are plots of mean peel strength and mean peel time-to-failure for the three adhesive systems as a function of heat-exposure time and temperature. In both, the open plot character represents controls; the closed plot character represents heat-exposed specimens. Both the controls and heat-exposed specimens generally failed cohesively in the peel-strength and creep-rupture tests. Two exceptions were observed during the creep-rupture tests. One TS1 specimen and one LA specimen exposed for 90 days at 90 °C (194 °F) failed in mixed and adhesive modes, respectively.

Table 7. Summary of peel-strength data for Task II

Adhesive System	Heat Exposure			Peel Strength, kN/m				Peel Strength, lbf/in				CoV[c]	Failure Mode[d]
	Period	°C	°F	min	max	mean[a]	sd[b]	min	max	mean[a]	sd[b]	%	
TS1	control	23	73	1.74	1.92	1.81	0.08	9.92	10.96	10.31	0.46	4.4	1
	30 days	60	140	0.79	1.84	1.28	0.49	4.52	10.50	7.34	2.77	37.7	1
		75	167	2.08	2.15	2.11	0.03	11.86	12.28	12.05	0.18	1.5	1
		90	194	2.61	2.68	2.65	0.03	14.88	15.32	15.10	0.18	1.2	1
	90 days	60	140	2.05	2.07	2.06	0.01	11.71	11.84	11.77	0.06	0.5	1
		75	167	2.51	2.60	2.54	0.04	14.31	14.84	14.50	0.24	1.6	1
		90	194	2.65	2.73	2.69	0.03	15.15	15.57	15.35	0.18	1.2	1
	150 days	60	140	2.20	2.26	2.23	0.02	12.57	12.88	12.72	0.13	1.0	1
		75	167	2.72	2.78	2.74	0.03	15.52	15.85	15.65	0.16	1.0	1
		90	194	2.57	2.61	2.59	0.02	14.66	14.88	14.77	0.09	0.6	1
TS2	control	23	73	2.02	2.18	2.09	0.07	11.51	12.46	11.93	0.40	3.3	1
	30 days	60	140	2.00	2.10	2.06	0.04	11.44	11.97	11.79	0.23	2.0	1
		75	167	2.17	2.24	2.21	0.04	12.37	12.79	12.60	0.21	1.7	1
		90	194	1.61	1.83	1.72	0.11	9.22	10.45	9.80	0.61	6.2	1
	90 days	60	140	1.80	2.08	1.97	0.12	10.30	11.88	11.26	0.68	6.0	1
		75	167	2.05	2.42	2.29	0.17	11.69	13.80	13.10	0.96	7.3	1
		90	194	1.62	1.80	1.71	0.07	9.26	10.30	9.79	0.43	4.3	1
	150 days	60	140	2.31	2.53	2.40	0.10	13.21	14.42	13.73	0.58	4.2	1
		75	167	1.68	1.99	1.88	0.14	9.59	11.38	10.75	0.81	7.5	1
		90	194	1.25	1.52	1.41	0.12	7.12	8.67	8.08	0.67	8.3	1
LA	control	23	73	1.43	1.90	1.72	0.22	8.18	10.83	9.82	1.26	12.8	1
	30 days	60	140	1.78	1.81	1.80	0.02	10.14	10.36	10.25	0.10	0.9	1
		75	167	1.00	1.29	1.10	0.13	5.73	7.39	6.26	0.77	12.2	1
		90	194	1.20	1.74	1.37	0.25	6.84	9.94	7.85	1.45	18.4	1
	90 days	60	140	0.63	1.21	0.96	0.24	3.59	6.90	5.47	1.40	25.5	1
		75	167	0.62	1.07	0.85	0.19	3.55	6.13	4.83	1.11	22.9	1
		90	194	0.72	1.66	1.17	0.43	4.12	9.46	6.71	2.43	36.2	1
	150 days	60	140	0.35	1.36	0.76	0.43	1.98	7.74	4.37	2.46	56.2	1
		75	167	0.59	0.96	0.72	0.17	3.37	5.49	4.10	0.97	23.8	1
		90	194	0.88	1.31	1.12	0.21	5.05	7.45	6.42	1.18	18.4	1

[a]Mean of four determinations. [b]sd indicates standard deviation. [c]CoV indicates coefficient of variation. [d]Failure mode: 1 = cohesive.

Table 8. Summary of peel time-to-failure data for Task II

Adhesive System	Exposure Days	°C	°F	Failed Specimens No.	Time-To-Failure, h min	max	mean[a]	sd[b]	CoV[c] %	Failure Mode[d]
TS1	control	23	73	8	64.21	83.20	74.22	6.92	9.3	1
	30 days	60	140	8	72.60	77.83	75.26	1.71	2.3	1
		75	167	8	196.12	290.40	244.69	34.56	14.1	1
		90	194	2	1099.8	36961[e]	6079.3[e]	12500	206	1
	90 days	60	140	8	374.86	405.20	389.86	11.14	2.9	1
		75	167	2	1502.1	452140[e]	78590[e]	159400	203	1
		90	194	1	825.20	11300[e]	6605.1[e]	3490	52.8	1(7), 3(1)
	150 days	60	140	8	754.16	939.97	853.35	76.71	9.0	1
		75	167	0	7320[e]	76225[e]	28410[e]	20480	72.1	1
		90	194	8	1164.2	1630.4	1367.1	171.5	12.5	1
TS2	control	23	73	8	54.19	86.92	71.77	11.73	16.3	1
	30 days	60	140	8	45.92	86.50	59.03	13.77	23.3	1
		75	167	8	37.16	59.12	49.65	7.49	15.1	1
		90	194	8	9.42	27.90	17.48	6.21	35.5	1
	90 days	60	140	8	89.91	197.00	118.22	33.90	28.7	1
		75	167	8	35.30	72.43	52.52	11.76	22.4	1
		90	194	8	9.619	23.55	15.68	4.55	29.0	1
	150 days	60	140	8	103.91	172.95	136.90	26.07	19.0	1
		75	167	8	26.82	45.18	34.91	6.11	17.5	1
		90	194	8	3.70	11.80	8.32	2.61	31.4	1
LA	control	23	73	8	9.48	19.85	13.12	3.38	25.7	1
	30 days	60	140	8	25.33	644.87	266.99	209.57	78.5	1
		75	167	6	0.64	1760[e]	540[e]	760	141	1
		90	194	2	18.83	2140[e]	700[e]	777	111	1
	90 days	60	140	8	7.00	375.43	219.35	146.81	66.9	1
		75	167	8	0.05	135.10	50.52	56.06	111.0	1
		90	194	5	16.03	2724[e]	810[e]	950	117	1(7), 2(1)
	150 days	60	140	8	0.70	111.35	21.38	37.71	176.4	1
		75	167	8	1.180	120.699	44.56	44.03	98.8	1
		90	194	8	7.847	770.195	156.76	256.53	163.6	1

[a]Mean of eight determinations. [b]sd indicates standard deviation. [c]CoV indicates coefficient of variation.
[d]Failure mode: 1 = cohesive; 2 = adhesive; 3 = mixed cohesive/adhesive; numbers in parentheses indicate the number of specimens in the sample set that experienced the given failure mode; where there are no parentheses, all specimens failed by the given mode. [e]Estimated values based on censored data; i.e., the test was terminated before all specimens failed; times-to-failure were estimated assuming a linear rate of delamination.

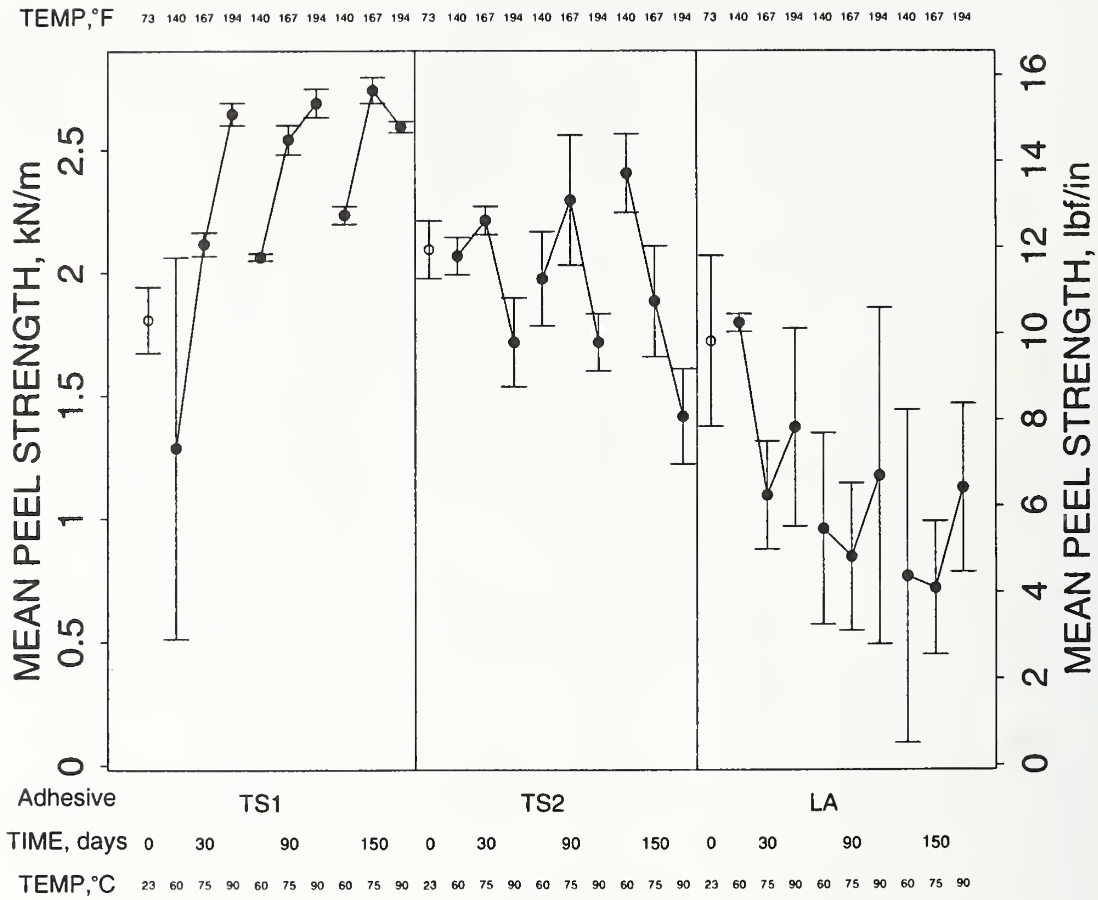

Figure 5. Mean Peel Strength for the Three Adhesive Systems as a Function of Heat-Exposure Time and Temperature. The Open Plot Characters Represent Controls; the Closed Plot Characters Represent Heat-Exposed Specimens.

4.2.1 Effect of Heat Exposure on the Adhesive Systems. From Table 7 and Figure 5, the following observations on peel strength are noted:

- For the three adhesive systems, the mean peel strengths of the controls were comparable to those of the Task I controls (compare Tables 3 and 7). For each adhesive system, the samples for Tasks I and II were prepared at different times using the same batch of adhesives and primers.
- For the three adhesive systems, the shortest exposure time and the lowest temperature, 30 days at 60 °C (140 °F), had no effect on peel strength. The mean peel strength of each control and that of these exposed samples were not significantly different.
- For TS1, with the exception of the exposure for 30 days at 60 °C (140 °F), heating resulted in increased peel strength—all heat-exposed sample sets had mean peel strengths significantly greater than that of the TS1 control. Moreover, mean peel strength generally increased with time and temperature, although a decrease was observed for the sample set exposed for 150 days at 90 °C (194 °F) versus that exposed for 150 days at 75 °C (167 °F).
- For TS2, mean peel strength was unaffected by exposure at 60 °C and 75 C° (140 °F and 167 °F) with the exception of the 150-day exposure at 60 °C (140 °F) for which a significant increase in strength was observed. All TS2 sample sets exposed at 90 °C (194 °F) had lower mean peel strengths than the control.

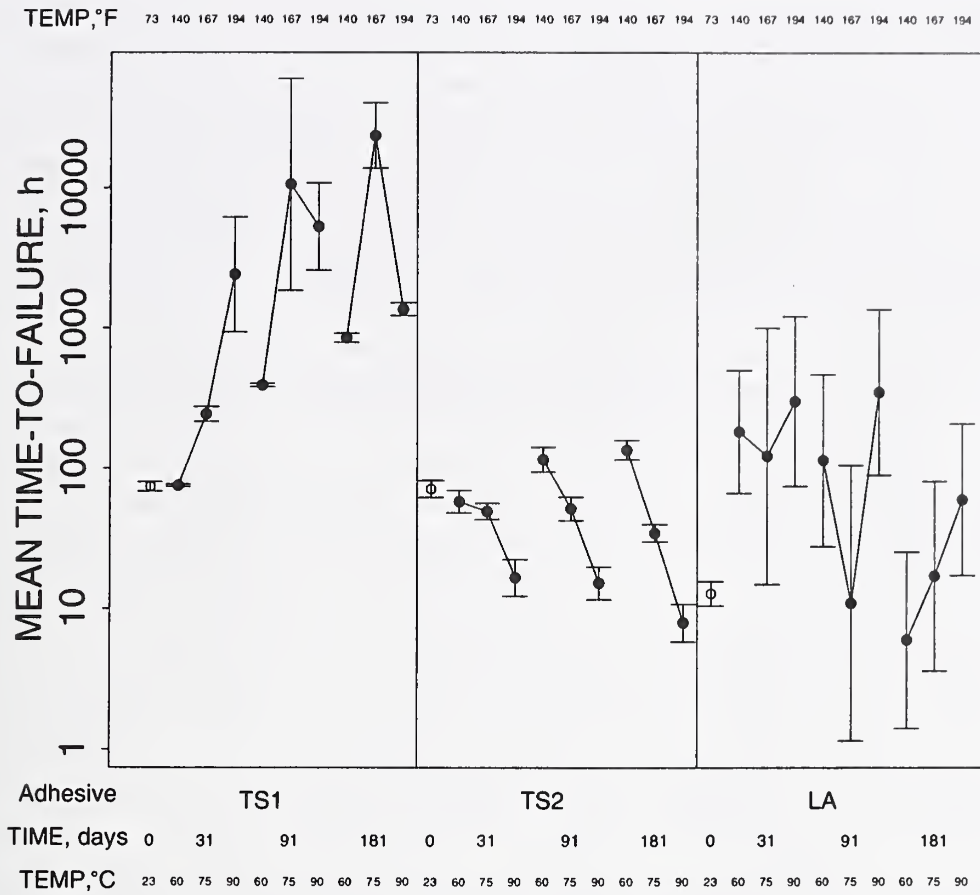

Figure 6. Mean Peel Time-to-Failure for the Three Adhesive Systems as a Function of Heat-Exposure Time and Temperature. The Open Plot Characters Represent Controls; the Closed Plot Characters Represent Heat-Exposed Specimens.

- For LA, mean peel strength generally decreased upon heat exposure. Analyses indicated that, for the 30-day and 90-day exposure periods, there was no significant difference between mean peel strength at 90 °C (194 °F) and that at 75 °F (167 °F). However, when exposed for 150 days, the 90 °C (194 °F) sample set had significantly greater strength than the 75 °F (167 °F) sample set. Reasons for this finding were not explored.

From Table 8 and Figure 6, the following observations on peel creep-rupture behavior are noted:

- For TS1, with the exception of the exposure for 30 days at 60 °C (140 °F) where no change occurred, all heat exposures resulted in longer times-to-failure than the control. The longest times-to-failure were found for the samples sets exposed at 75 °C (167 °F) for 90 days and 150 days.
- For TS2, the heat exposures at 60 °C and 75 °C (140 °F and 167 °F) had little effect on time-to-failure in comparison to the control. At 90 °C (194 °F), the mean times-to-failure were less than that of the control.
- For LA, the mean times-to-failure of the heat exposed sample sets were generally comparable to, or greater than, that of the control. However, for the 150-day at 60 °C (140 °F) exposure, the mean value was slightly less than that of the control; the difference was significant.

4.2.2 Comparison of Task II Tape-Adhesive and Liquid-Adhesive Results. From Table 7 and Figure 5, the following observations comparing the Task II peel-strength results for the tape-bonded and liquid-adhesive-bonded sample sets systems are noted:

- For LA with respect to the control, mean peel strength generally decreased due to the three temperature exposures. In comparison, decreasing strength upon temperature exposure was generally not found for the tape-bonded sample sets. For TS1 with respect to the control, mean peel strengths mostly increased at all temperatures. For TS2 with respect to the control, mean peel strength generally remained unchanged at 60 °C and 75 °C (140 °F and 167 °F); it decreased at 90 °C (194 °F).
- The laboratory heat exposures subjected samples to temperatures that EPDM membranes can experience in service. For example, Rosenfield measured EPDM membrane temperatures as high as 90 °C (194 °F) during a summer in Georgia [16]. Because the Task II peel-strength data indicated that heat exposure can increase peel strength above those determined for nonexposed (control) samples, it was of interest to compare the Task II peel-strength results with peel-strength data determined for field seams (i.e., those sampled from roofs). In particular, a question was raised as to whether tape-bonded field seams have shown peel strengths as high as those measured for laboratory heat-exposed samples. The range of mean peel strengths for all Task II laboratory heat exposed tape-bonded sample sets was 1.41 kN/m to 2.74 kN/m (8.08 lbf/in to 15.65 lbf/in). In comparison, in a field study, the range of mean peel strengths for tape-bonded field seams that failed cohesively was 0.34 kN/m to 2.97 kN/m (1.93 lbf/in to 16.97 lbf/in), with an average value of 1.76 kN/m (10.04 lbf/in) [17]. Thus, comparing the high end of these ranges, the field seams displayed comparable, if not slightly higher, peel strength than the heat exposed laboratory samples. This finding suggests that heat effects in the field might possibly contribute to an increase in peel strength. The comparison also shows that, at the lower end of the ranges, the laboratory heat exposed samples had considerably higher peel strengths than the field seams. Although laboratory heat exposures decreased the peel strength of some tape-bonded samples, the resultant strengths were, at the extreme, only about 20 % lower than the average value for the field seams.
- In a similar comparison, the mean peel strengths of the laboratory heat exposed liquid-adhesive-bonded samples ranged from 0.72 kN/m to 1.80 kN/m (4.10 lbf/in to 10.25 lbf/in); whereas for liquid-adhesive field seams, peel-strength values ranged from 0.23 kN/m to 1.2 kN/m (1.3 lbf/in to 6.9 lbf/in) with an average of about 0.5 kN/m (3 lbf/in) [18-20]. Although the LA laboratory samples generally lost peel strength upon heat exposure, the decreases did not result in values that were less than the average peel strength determined for the field seams.

From Table 8 and Figure 6, the following observations comparing the Task II peel creep-rupture results for the tape-bonded and liquid-adhesive-bonded sample sets are noted:

- For LA with respect to the control, mean times-to-failure generally were unaffected or increased due to the heat exposures. In comparison, TS1 mean times-to-failure increased, and the increases were greater for TS1 than for LA. For example, for the exposure of 90 days at 75 C (167 F), the TS1 exposed sample set had a mean time-to-failure that was estimated to be about 100 times greater than the control; in contrast, the mean time-to-failure of the LA exposed sample set was about 4 times greater than its control. In comparison to LA behavior, TS2 mean times-to-failure were unaffected or decreased upon heat exposure. The greatest decrease was observed for the 150-day exposure at 90 °C (194 °F). In this case, the exposed sample set had a mean value of 8.32 h, which was about 8.5 times less than the control. The 8.32 h time-to-failure was slightly less than the 13.12 h mean time-to-failure of the LA control; the difference was significant.

- As was the case for the peel-strength data, it was of interest to compare the Task II time-to-failure results with data determined for field seams. In Task II, many TS1 heat exposed specimens were found, vis-a-vis the control, to be very creep resistant with estimated times-to-failure greater than 10,000 h. The shortest mean time-to-failure for a TS2 heat exposed sample set was about 8 h. These extreme values may be compared with the time-to-failure results obtained from 10 field seams that failed cohesively during creep testing. For these field seams, mean times-to-failure ranged from 10.9 h to 173.7 h [17]. Thus, in this comparison, no evidence is found that the long times-to-failure produced in the laboratory upon heat exposure have occurred in the field. The comparison also indicates that, although some tape-bonded laboratory samples underwent a decrease in time-to-failure upon heat aging, the resultant values were not appreciably less than mean times-to-failure that have been obtained for field seams.

5. EFFECT OF EXPOSURE TO INDUSTRY-DEVELOPED PROTOCOLS ON PEEL TIME-TO-FAILURE (TASK III)

5.1 Objective and Scope of Task III

The Task III investigation was conducted to determine the peel-creep resistance and peel strength after laboratory exposure to the protocols developed by RMA [11] and SPRI [12] (see Table 9), and to compare the performance of tape-bonded and liquid-adhesive-bonded sample sets after such exposures. Both protocols subject specimens to dry heat in an oven, heat in water, and freeze-thaw cycling. In contrast to the RMA protocol, the SPRI protocol requires that the specimens be elongated in shear at 20 % while being subjected to the freeze-thaw cycling and to heat cycling (conducted in an ultraviolet test chamber).

The RMA protocol stipulates cycling for 28 days with an option to conduct it for 56 days (Table 9). In the Task III experiment, samples were cycled for 28 days and for 56 days so that data would be available on an RMA exposure period that was the same length as the SPRI exposure period, which is stipulated to be 56 days.

Table 9. Summary of the RMA and SPRI laboratory exposure protocols for seam specimens

Exposure Protocol	Step No.	Time days	Specimen Elongation[a]	Exposure Condition
RMA	1	1	None	80°C (176°F) in a forced air oven
	2	3	None	80°C (176°F) in water
	3	1/3	None	-18°C (0°F) in a freezer
	4	2 & 2/3	None	80°C (176°F) in water
	5	21	None	Repeat steps 1 - 4 for a total of 28 days (four 7-day cycles)
	6	28	None	Repeat steps 1 - 4 for a total of 56 days (eight 7-day cycles); note: it is optional as to whether the four cycles in step 6 are conducted.
SPRI	1	28	None	115°C (240°F) in a forced air oven
	2	14	20 %	70°C (158°F) in water
	3	2/3	20 %	-18°C (0°F) in a freezer
	4	1/3	20 %	21°C (70°F) in water
	5	6	20 %	Repeat steps 3 & 4 for a total of 7 days (seven 1-day cycles)
	6	1/6	20 %	4 h of UV in a QUV chamber[b] with a black body temperature of 70°C (158°F)
	7	1/3	20 %	8 h in the QUV chamber under condensation conditions without irradiation at a temperature of 50°C (122°F).
	8	6 & 1/2	20 %	Repeat steps 6 & 7 for a total of 7 days (14 light on/off cycles)

[a]Specimen in tension with bond in shear; the grips for the elongation jig are set 6 mm (0.25 in) from the specimen ends.
[b]Certain company products are mentioned in the text to specify adequately the experimental procedure and equipment uscd. In no case does such identification imply recommendation or endorsement by thc National Institute of Standards and Technology, nor does it imply that the equipment is necessarily the best available for the purpose.

After the RMA and SPRI exposures were completed, peel strength and peel time-to-failure of the tape-bonded and liquid-adhesive-bonded specimens were determined at 23 °C (73 °F). The creep load was 9.3 N (2.1 lbf). Table 10 summarizes the Task III test conditions and lists the number of specimens tested at each condition.

Table 10. Summary of test conditions and number of specimens for Task III

Exposure Protocol	Adhesive System	Peel-Strength Tests: No. of Specimens Exposure Period			Creep-Rupture Tests: No. of Specimens Exposure Period		
		control[a]	28 days	56 days	control[a]	28 days	56 days
RMA	TS1	4	4	4	8	8	8
	TS2	4	4	4	8	8	8
	LA	4	4	4	8	8	8
SPRI	TS1	4	---[b]	4	8	---[b]	8
	TS2	4	---	4	8	---	8
	LA	4	---	4	8	---	8

[a]Control samples were not exposed.
[b]The SPRI protocol does not include a 28-day exposure period.

5.2 Task III Results and Discussion

Tables 11 and 12 summarize the peel-strength and peel creep-rupture data for Task III, respectively. Figures 7 and 8 are plots of mean peel strength and mean peel time-to-failure, respectively, after subjecting specimens of each adhesive system to: (A) the RMA exposure protocol for 28 days and 56 days, and (B) the SPRI exposure protocol for 56 days. Data on room temperature control specimens are included. In general, the control and exposed specimens failed cohesively in the peel-strength and creep-rupture tests. An exception was the 56-day RMA exposed TS2 sample set, wherein three specimens failed in a mixed mode in the creep-rupture tests. In these cases, the majority of the bond area failed cohesively.

5.2.1 Effect of RMA and SPRI Exposures on the Adhesive Systems. From Table 11 and Figure 7, the following observations on peel strength are noted:

- For the three adhesive systems, the mean peel strengths determined for the RMA and SPRI controls were comparable to those measured for control sample sets in Tasks I and II (compare Tables 3, 7, and 11).
- When subjected to the RMA protocol, TS1 became stronger upon exposure, and the longer the exposure, the stronger were the specimens. TS2 became weaker upon exposure, and the longer the exposure, the weaker were the specimens. Considerable variability was observed for TS2 mean peel strength after the 56-day exposure. LA did not change strength upon exposure (no significant differences among mean peel strengths as a function of exposure were found). Considerable variability was seen for the LA mean peel strength for the 28-day and 56-day exposures.
- When subjected to the SPRI protocol, TS1 became stronger upon exposure; whereas TS2 became weaker. LA also became weaker upon exposure.

Table 11. Summary of peel-strength data for Task III

Exposure Protocol	Adhesive System	Exposure Period	Peel Strength, kN/m min	max	mean[a]	sd[b]	Peel Strength, lbf/in min	max	mean[a]	sd[b]	CoV[c] %	Failure Mode[d]
RMA	TS1	control[e]	1.64	1.86	1.75	0.10	9.4	10.6	10.0	0.56	5.6	1
		28 days	2.38	2.44	2.40	0.03	13.6	13.9	13.7	0.16	1.1	1
		56 days	2.50	2.63	2.57	0.06	14.3	15.0	14.7	0.32	2.2	1
	TS2	control	2.10	2.17	2.12	0.03	12.0	12.4	12.1	0.18	1.5	1
		28 days	1.94	2.01	1.97	0.03	11.1	11.5	11.3	0.17	1.5	1
		56 days	1.12	1.71	1.48	0.28	6.4	9.8	8.4	1.61	19.0	1
	LA	control	1.63	1.77	1.71	0.06	9.3	10.1	9.8	0.35	3.6	1
		28 days	1.51	1.90	1.71	0.21	8.6	10.9	9.7	1.22	12.5	1
		56 days	1.00	1.65	1.37	0.27	5.7	9.4	7.8	1.55	19.8	1
SPRI	TS1	control	1.69	1.92	1.80	0.10	9.7	11.0	10.3	0.56	5.4	1
		56 days	2.40	2.51	2.43	0.05	13.7	14.4	13.9	0.31	2.2	1
	TS2	control	1.86	2.14	1.98	0.12	10.6	12.2	11.3	0.71	6.3	1
		56 days	1.31	1.37	1.34	0.02	7.5	7.8	7.7	0.14	1.8	1
	LA	control	1.54	1.82	1.67	0.14	8.8	10.4	9.5	0.82	8.6	1
		56 days	0.93	1.12	1.03	0.08	5.3	6.4	5.9	0.43	7.4	1

[a]Mean of four determinations. [b]sd indicates standard deviation. [c]CoV indicates coefficient of variation.
[d]Failure mode: 1 = cohesive. [e]The control samples were not exposed.

- No statistically significant difference was found between the mean peel strength of the RMA and SPRI controls for each of the three adhesive systems. The 56-day RMA and SPRI exposures had the same effect on TS2 and LA mean peel strength, as the values after the two exposures were not significantly different. In the case of TS1, a significant difference between mean peel strengths after the 56-day RMA and SPRI exposures were found. From a practical viewpoint, the difference between the two peel strengths, 2.57 kN/m and 2.43 kN/m (14.7 lbf/in and 13.9 lbf/in), may not be important. Thus, the data suggest that, if the effect of the RMA and SPRI exposures on peel strength is the factor of interest, it may not be necessary to subject samples to both protocols.

From Table 12 and Figure 8, the following observations on peel creep-rupture behavior are noted:

- For each of the three adhesive systems, a statistically significant difference in peel time-to-failure was found between the RMA and SPRI controls. These differences were not considered to be practically important, but illustrate that some variability can occur when different sample batches are fabricated at different times using the same batches of adhesives and primers. Slight variability between replicate sets of seam samples was observed in Phase I [3].
- When subjected to the RMA protocol, TS1 had longer time-to-failure upon exposure, and the longer the exposure, the longer lived were the specimens. TS2 displayed shorter time-to-failure upon exposure with no difference observed between the 28-day and 56-day exposures. LA showed longer time-to-failure upon exposure with no effect observed between the 28-day and 56-day exposures.

Table 12. Summary of peel time-to-failure data for Task III

Exposure Protocol	Adhesive System	Exposure Days	Failed Specimens No.	Time-to-Failure, h min	max	mean[a]	sd[b]	CoV[c] %	Failure Mode[d]
RMA	TS1	control[e]	8	53.63	79.49	70.00	9.65	13.8	1
		28 days	4	731.92	3696[f]	1646[f]	936	56.9	1
		56 days	0	10765[f]	226069[f]	63361[f]	70541	111	1
	TS2	control	8	56.18	130.93	81.88	26.23	32.0	1
		28 days	8	29.60	47.45	39.04	6.98	17.9	1
		56 days	8	10.44	51.92	32.56	15.46	47.5	1 (5), 3 (3)
	LA	control	8	5.57	49.31	19.81	15.84	79.9	1
		28 days	1	511.74	3328[f]	2044[f]	781	38.2	1
		56 days	3	120.15	3351[f]	1495[f]	1134	75.9	1
SPRI	TS1	control	8	63.14	96.26	82.21	10.30	12.5	1
		56 days	8	54.52	93.16	74.90	13.86	18.5	1
	TS2	control	8	35.15	65.18	46.70	8.98	19.2	1
		56 days	8	4.15	7.35	5.83	1.16	19.9	1
	LA	control	8	2.67	9.55	4.87	2.24	45.9	1
		56 days	8	50.58	701.72	241.36	197.66	81.9	1

[a]Mean of eight determinations. [b]sd indicates standard deviation. [c]CoV indicates coefficient of variation.
[d]Failure mode: 1 = cohesive; 3 = mixed cohesive/adhesive; numbers in parentheses indicate the number of specimens in the sample set that experienced the given failure mode; where there are no parentheses, all specimens failed by the given mode. [e]The control samples were not exposed. [f]Estimated values based on censored data; i.e., the test was terminated before all specimens failed; times-to-failure were estimated assuming a linear rate of delamination.

- When subjected to the SPRI protocol, TS1 mean time-to-failure was unaffected by the exposure. TS2 showed shorter mean time-to-failure upon exposure. LA had longer mean time-to-failure upon exposure.
- For a given adhesive system, the RMA and SPRI exposures did not have the same effect. For example, TS1 had considerably longer time-to-failure after the RMA exposure than after the SPRI exposure. TS2 was somewhat longer lived after the RMA exposure than after the SPRI exposure. It is not known whether stressing the specimens during SPRI exposure contributes to these observations, but applied stress is a factor differentiating the RMA and SPRI exposures (Table 9).

5.2.2 Comparison of Task III Tape-Adhesive and Liquid-Adhesive Results. From Table 11 and Figure 7, the following observations comparing the Task III peel-strength results for the tape-bonded and liquid-adhesive-bonded sample sets are noted:

- Regarding the RMA protocol, the LA mean peel strength was unchanged upon exposure. In comparison, TS1 increased in mean peel strength, and TS2 decreased in mean peel strength. Although decreased, the TS2 56-day mean peel strength of 1.48 kN/m (8.4 lbf/in) was not statistically different than that of the LA control, 1.71 kN/m (9.8 lbf/in).
- Regarding the SPRI protocol, the LA mean peel strength decreased upon exposure. In comparison, as was the case with the RMA exposure, TS1 increased in mean peel strength, and

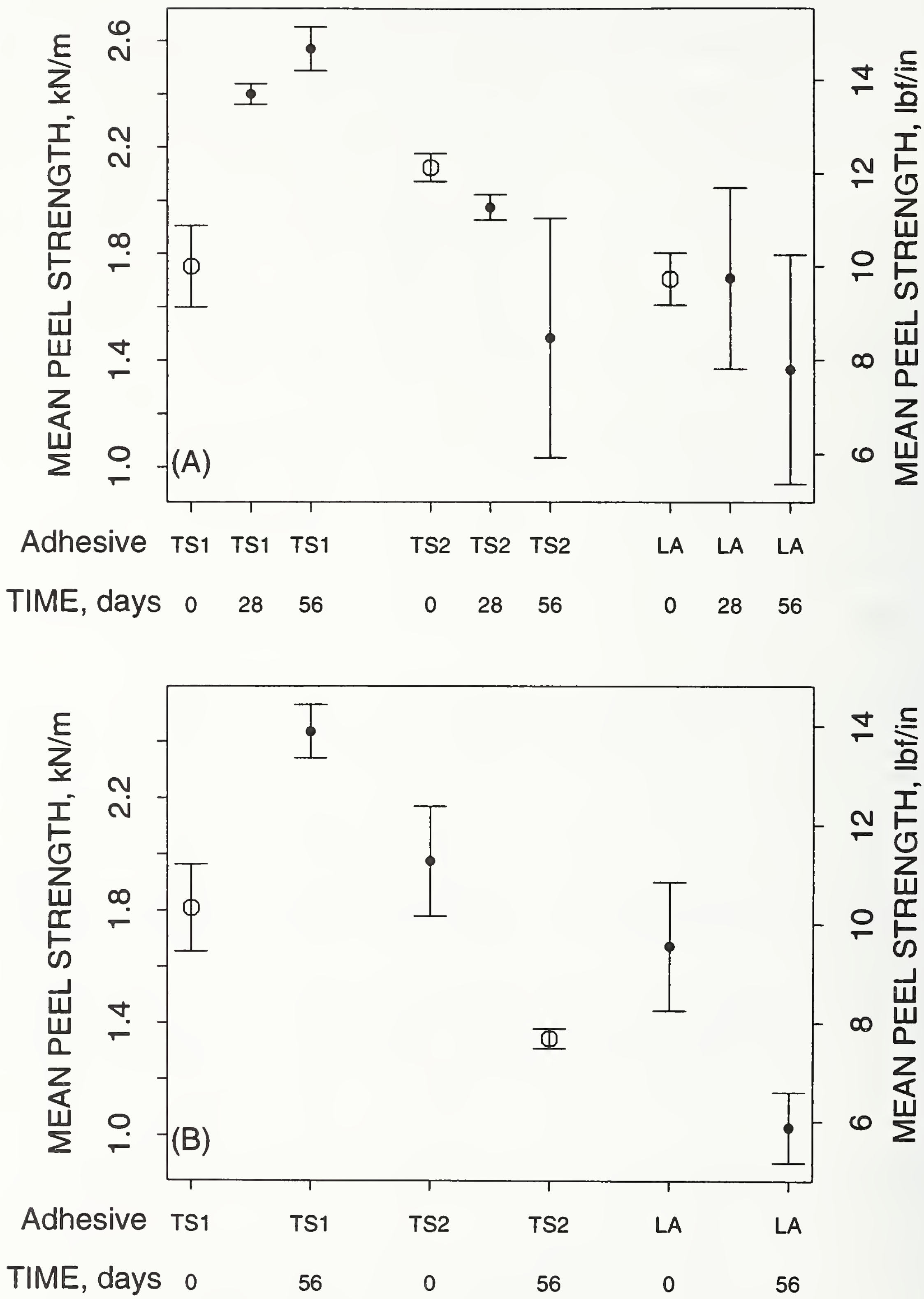

Figure 7. Mean Peel Strength after Subjecting Specimens to the: (A) RMA Exposure Protocol for 28 days and 56 days, and (B) SPRI Exposure Protocol for 56 days.

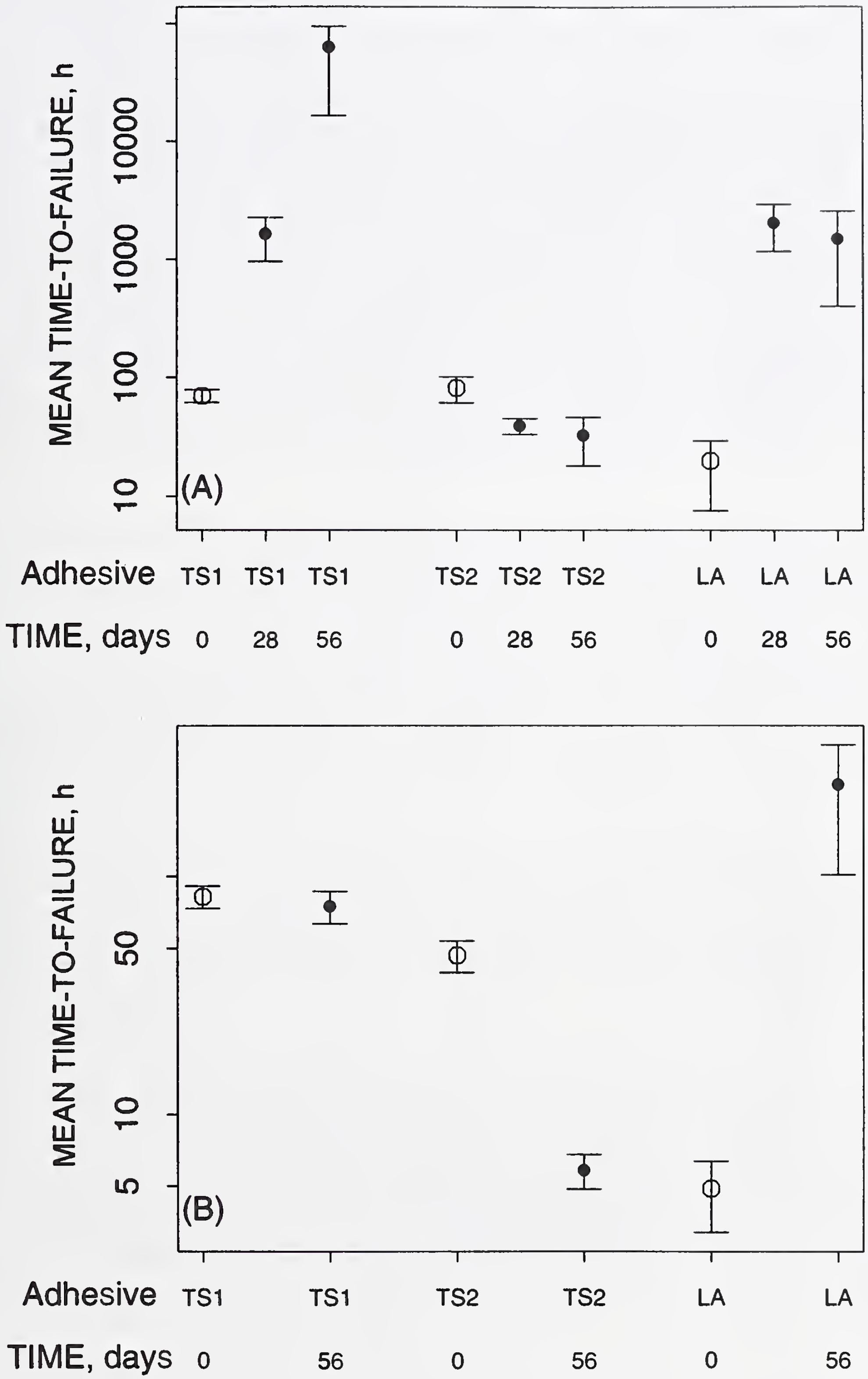

Figure 8. Mean Peel Time-to-Failure after Subjecting Specimens to the: (A) RMA Exposure Protocol for 28 days and 56 days, and (B) SPRI Exposure Protocol for 56 days.

TS2 decreased in mean peel strength. In this case, the TS2 mean peel strength after exposure was less than that of the LA control, but greater than that of LA exposed for 56 days.

- As in the case for Task II, it was of interest to compare the Task III peel-strength results with peel-strength data determined for field seams. The RMA and SPRI exposures subject samples to conditions incorporating factors (i.e., heat, moisture, and stress) that may result in changes in strength in service. As previously indicated, the range of mean peel strengths for tape-bonded field seams that failed cohesively was 0.34 kN/m to 2.97 kN/m (1.93 lbf/in to 16.97 lbf/in), with an average value of 1.76 kN/m (10.04 lbf/in) [17]. For the Task III samples, independent of exposure protocol and time, the range of mean peel strengths for the exposed tape-bonded sample sets was 1.34 kN/m to 2.57 kN/m (7.7 lbf/in to 14.7 lbf/in). Thus, it is seen that, at the high ends of the ranges, the peel strengths of the laboratory sample sets that gained strength during exposure and those of the field sample sets were comparable. At the low end of the ranges, the laboratory exposed samples displayed greater peel strength than the field samples. That is, although some tape-bonded samples lost peel strength due to the RMA and SPRI exposures, strength was not reduced below values displayed by some field samples.

From Table 12 and Figure 8, the following observations comparing the Task III peel creep-rupture results for the tape-bonded and liquid-adhesive-bonded sample sets are noted:

- Regarding the RMA protocol, LA mean time-to-failure increased upon exposure. In comparison, TS1 and TS2 had increased and decreased time-to-failure, respectively. TS2 mean time-to-failure after 56-day exposure was not statistically different than that of the LA control.
- Regarding the SPRI protocol, the LA mean time-to-failure increased upon exposure. In comparison, TS1 was unchanged, and TS2 decreased. Again, as was the case for the RMA exposure, TS2 mean time-to-failure after 56-day exposure was not statistically different than that of the LA control.
- Similar to the peel-strength discussions, it was of interest to compare the Task III peel time-to-failure results with data determined for tape-bonded field seams. As previously indicated, mean times-to-failure of field tape-bonded seams that failed cohesively in creep tests have ranged from 10.9 h to 173.7 h [17]. In comparison, for TS1 after SPRI exposure, the mean time-to-failure was 75 h, i.e., in the middle of the field-seam range. After 56-day RMA exposure which considerably increased the creep resistance of the TS1 laboratory sample sets, the minimum mean time-to-failure was estimated to be above 10,000 h. This value was substantially beyond the field seam range. That is, the comparison indicates that the relatively long TS1 times-to-failure, brought about by the RMA exposure, have not been observed for field seams. This finding parallels that of Task II, wherein it was found that prolonged times-to-failure due to laboratory heat exposure have not been evidenced for field seams. Comparing the TS2 results with field seams, the mean times-to-failure of the RMA and SPRI sample sets (about 33 h and 6 h, respectively) were comparable to the shorter times-to-failure determined for field seams. That is, although TS2 decreased in time-to-failure upon RMA and SPRI exposures, the resultant values were not atypical of those of some field seams.

6. EFFECT OF COLD TEMPERATURE PREPARATION ON PEEL TIME-TO-FAILURE (TASK IV)

6.1 Objective and Scope of Task IV

The Task IV investigation was conducted to characterize the peel-creep resistance and peel strength of seam specimens prepared either in the laboratory or in the field at temperatures of about 0 °C (32 °F) or less, and to compare the creep-rupture performance of tape-bonded and liquid-adhesive-bonded specimens after preparation at the cold temperatures. The availability of field-prepared sample sets allowed for a preliminary comparison between creep-rupture (and peel strength) results from laboratory specimens and those fabricated in practice. Nevertheless, it is must be remembered in the discussions that follow that the field data are limited and, strictly speaking, apply only to the field sample sets prepared for the study. Specifically, the materials and conditions under which each sample set was prepared were not described. The tapes, primers, liquid adhesives, and EPDM rubber sheets (or their combinations) used in the field may be different than those used at NIST in Phase III. Moreover, these materials may also vary among the different field sample sets.

Table 13 summarizes the Task IV preparation temperatures and lists the number of specimens tested. In the laboratory, tape-bonded and liquid-adhesive-bonded peel samples were prepared* in a temperature-humidity controlled glove box at temperatures of -5 °C to -2 °C (23 °F to 28 °F), and -1 °C to 1 °C (30 °F to 34 °F) with humidity between 10 % to 15 %. The samples were kept at about -4 °C (25 °F) for 28 days after preparation, and then shipped overnight to NIST. Room-temperature laboratory specimens, i.e., controls, were prepared at 21 °C to 23 °C (69 °F to 73°F) with a humidity of 45 % to 50 %, and kept under those conditions for a minimum of 28 days before testing.** This cold temperature preparation experiment was planned to include a temperature lower than -4 °C (25 °F). However, the available cold temperature-humidity box could not provide a lower temperature.

In the field, roofing mechanics using roof-top application procedures prepared seam samples outdoors at contractor facilities when the temperature was no higher than -8 °C (18 °F). After preparation, the samples were kept outdoors for a minimum of 28 days after which they were shipped overnight to NIST. Table 14 provides a summary of the general weather conditions for the month during which the field samples were fabricated.

After receipt at NIST, the laboratory and field cold-temperature-prepared samples were placed in a freezer at about -20 °C (-4 °F), where they were stored until warming to room temperature for peel specimen preparation, and for peel-strength and peel creep-rupture testing. The time elapsed between removal from the freezer and testing did not exceed 24 h. Where sufficient field-prepared samples were available, additional test specimens were kept at room temperature for a minimum of 28 days and then subjected to the peel strength and peel creep-rupture tests at 9.3 N (2.1 lbf). In the discussions which follow, samples tested within 24 h of removal from the freezer and those kept at room temperature for at least 28 days before testing are referred to as "cold" and "warm" samples, respectively.

---

*The specimens were prepared in the laboratories of consortium member, Firestone Building Products Co., because of the availability of a cold temperature-humidity box at that facility.

**For clarity, the Task IV preparation temperatures are referred to as -4 °C, 0 °C , and 23 °C (25 °F , 32 °F , and 73 °F) in the report.

Table 13. Summary of preparation temperatures and number of specimens for Task IV

Preparation Location	Adhesive Type	Prep. Temp. °C	Prep. Temp. °F	Peel-Strength Tests No. of Specimens "cold"	Peel-Strength Tests No. of Specimens "warm"	Creep-Rupture Tests No. of Specimens "cold"	Creep-Rupture Tests No. of Specimens "warm"
Laboratory	Tape—TS1	23	73	4	---	8	---
		0	32	4	---	8	---
		-4	25	4	---	8	---
	Tape—TS2	23	73	4	---	8	---
		0	32	4	---	8	---
		-4	25	4	---	8	---
	Liquid—LA	23	73	4	---	8	---
		0	32	4	---	8	---
		-4	25	4	---	8	---
Field—Alaska	Tape	-14	7	4	4	8	8
Field—Montana		-8	18	4	---	8	---
Field—New Hampshire		-8	18	4	4	8	8
Field—Wisconsin		-19	-3	4	4	8	8
Field—Alaska	Liquid	-14	7	4	4	8	8
Field—Montana		-8	18	4	4	8	8

6.2 Task IV Results and Discussion

Tables 15 and 16 summarize the peel-strength and peel creep-rupture data for Task IV, respectively. Figures 9 and 10 are plots of mean peel strength and mean peel time-to-failure, respectively, for the laboratory (plot A) and field (plot B) sample sets.

6.2.1 Effect of Cold Temperature Preparation on the Adhesive Systems. From Table 15 and Figure 9, the following observations on peel strength are noted:

- In the case of the tape-bonded sample sets, TS1 and TS2 controls had mean peel strengths typical of those of the controls in Tasks I, II, and III (compare Tables 3, 7, 11, and 15). In the case of the LA control sample set, the mean peel strength was about 20 % less that those of the sample sets prepared for the three previous tasks. All Task IV control specimens failed cohesively.
- For the TS1 laboratory samples, when prepared at -4 °C and 0 °C (25 °F and 32 °F), the mean peel strengths were 1.19 kN/m and 1.13 kN/m (6.81 lbf/in and 6.46 lbf/in), respectively. This represented a strength reduction of about 35 % in comparison to the control. The failure mode for the cold temperature prepared sample sets was adhesive with the locus of failure at the interface of the tape and the primer. The mean peel strengths for the two sample sets prepared at the cold temperatures were not statistically different, which may reflect the relatively small difference between the two preparation temperatures. These Task IV TS1 results may be compared with those of a similar cold temperature preparation experiment conducted in Phase II, but at temperatures not quite as cold. In Phase II, TS1 sample sets prepared at 5 °C (41 °F) had mean

Table 14. General weather conditions under which the Task IV field samples were prepared

Adhesive Type	Preparation Location	Weather Conditions[a]
Tape & Liquid	Alaska	The samples were prepared at -14 °C (7 °F) under partly cloudy skies in late November, 1997. The daily high temperatures for the month during which the samples remained outdoors ranged from -16 °C to 3 °C (4 °F to 38 °F); the daily low temperatures ranged from to -23 °C to -1 °C (-10 °F to 30 °F). Snow was always on the ground over the exposure, and reached a maximum depth of about 380 mm (15 in) late in the exposure period.
Tape & Liquid	Montana	The samples were prepared at -8 °C (18 °F) without sun in March, 1997. The daily high temperatures for the period during which the samples remained outdoors ranged from -7 °C to 26 °C (19 °F to 79 °F); the daily low temperatures ranged from to -21 °C to 7 °C (-6 °F to 45 °F). About 40 mm (1.6 in) of rain was received over the first two thirds of the exposure period. During seven of the last 10 days of exposure, about 575 mm (23 in) of snow was received.
Tape	New Hampshire	The sample was prepared at -8 °C (18 °F) under partly sunny conditions in March, 1997. The daily high temperatures for the period during which the sample remained outdoors ranged from -2 °C to 22 °C (29 °F to 71 °F); the daily low temperatures ranged from -18 °C to 4 °C (-1 °F to 39 °F). Seventy-five mm (3 in) of rain were received over the exposure period. Three hundred mm (12 in) of snow fell on one day at the middle of the period.
Tape	Wisconsin	The sample was prepared at -19 °C (-3 °F) without sun in January, 1997. Although the preparation was conducted in January, the samples did not arrive at NIST until after the end of the winter. The daily high temperatures for the period during which the sample remained outdoors ranged from -18 °C to 23 °C (0 °F to 73 °F); the daily low temperatures ranged from to -26 °C to 8 °C (-15 °F to 46 °F). It snowed two days after sample preparation and a snow-cover existed for about 2½ months thereafter.

[a]From the U.S. National Weather Service.

peel strengths of about 1.4 kN/m (8.0 lbf/in), and failed cohesively.* Thus, in Phase II, the peel-strength reduction due to cold temperature preparation was about 25 % when compared with the Phase III control. The colder temperatures in Task IV apparently provided greater decreases in mean peel strength (than found in Phase II), and a change in failure mode.

- For the TS2 laboratory samples, preparation at -4 °C and 0 °C (25 °F and 32 °F) resulted in mean peel strengths of 1.92 kN/m and 1.83 kN/m (10.94 lbf/in and 10.48 lbf/in), respectively; the failure modes were cohesive. These peel strengths were, on the average, about 11 % less than that of the control. The differences among the control and cold temperature prepared sample sets were statistically significant. By comparison, TS2 Phase II sample sets prepared at 5 °C (41 °F) had mean peel strengths of about 2.2 kN/m (12.5 lbf/in),** which was typical of that of the Task IV control. In contrast with Task IV, the Phase II TS2 cold temperature prepared sample sets failed in a mixed mode. However, the Phase II analysis indicated that specimens failing in a mixed mode had mean peel strengths comparable to those failing cohesively [4].

---

*See reference [4], Table 6A, Sample Sets Nos. 14 and 46.

**See reference [4], Table 6B, Sample Sets Nos. 30 and 62.

Table 15. Summary of peel-strength data for Task IV

	Adhesive Type	Prep. °C	Prep. °F	Conditioning After Field Prep.	Peel Strength, kN/m min	max	mean[a]	sd[b]	Peel Strength, lbf/in min	max	mean[a]	sd[b]	CoV[c] %	Fail. Mode[d]
LABORATORY	Tape—TS1	23	73	28 days min. at -4 °C (25 °F); RT[e] warm for specimen prep and test.	1.77	2.00	1.85	0.11	10.10	11.44	10.55	0.62	5.9	1
		0	32		1.09	1.20	1.13	0.06	6.20	6.88	6.46	0.31	4.9	2
		-4	25		1.03	1.34	1.19	0.12	5.91	7.63	6.81	0.71	10.4	2
	Tape—TS2	23	73		2.01	2.18	2.11	0.08	11.48	12.47	12.05	0.44	3.7	1
		0	32		1.83	1.86	1.83	0.02	10.43	10.63	10.48	0.10	0.9	1
		-4	25		1.86	2.02	1.92	0.07	10.60	11.53	10.94	0.41	3.7	1
	Liquid—LA	23	73		1.17	1.44	1.34	0.13	6.68	8.22	7.64	0.72	9.4	1
		0	32		1.12	1.54	1.24	0.20	6.39	8.78	7.09	1.13	15.9	1
		-4	25		0.71	1.11	0.91	0.17	4.04	6.35	5.21	0.95	18.2	1
FIELD	Tape—AK[f]	-14	7	"Cold" kept outdoors for 28 day min; RT warm for specimen prep and test.	1.07	1.23	1.13	0.07	6.09	7.03	6.48	0.40	6.2	2
	Tape—MT	-8	18		0.43	0.73	0.61	0.13	2.45	4.17	3.46	0.73	21.0	2
	Tape—NH	-8	18		0.85	1.13	0.99	0.16	4.87	6.46	5.65	0.90	15.9	2
	Tape—WI	-19	-3		0.30	0.55	0.42	0.11	1.70	3.13	2.41	0.63	26.2	2
	Liquid—AK	-14	7		0.51	0.65	0.55	0.07	2.89	3.73	3.13	0.40	12.8	2
	Liquid—MT	-8	18		0.84	1.12	0.96	0.12	4.81	6.39	5.51	0.69	12.5	3
FIELD	Tape—AK	-14	7	"Warm" kept outdoors 28 day min; RT warm for specimen prep; RT for 28 days min. before test.	0.81	1.67	1.26	0.38	4.63	9.53	7.20	2.14	30.2	1,2,3[g]
	Tape—NH	-8	18		1.57	1.60	1.59	0.02	8.95	9.15	9.07	0.10	1.1	3
	Tape—WI	-19	-3		0.18	0.56	0.38	0.16	1.01	3.21	2.15	0.91	42.4	2
	Liquid—AK	-14	7		0.45	0.58	0.52	0.06	2.56	3.31	2.97	0.31	10.5	2
	Liquid—MT	-8	18		0.71	0.87	0.80	0.07	4.05	4.97	4.58	0.42	9.3	3

[a]Mean of four determinations. [b]sd indicates standard deviation. [c]CoV indicates coefficient of variation.
[d]Failure mode: 1 = cohesive; 2 = adhesive; 3 = mixed. [e]RT indicates room temperature, about 23 °C (73 °F).
[f]The abbreviations are U.S. postal codes for states: AK = Alaska; MT = Montana, NH = New Hampshire, and WI = Wisconsin. [g]One, two, and one specimens failed cohesively, adhesively, and mixed, respectively.

- For the LA laboratory samples, preparation at -4 °C and 0 °C (25 °F and 32 °F) resulted in mean peel strengths of 0.91 kN/m and 1.24 kN/m (5.21 lbf/in and 7.09 lbf/in), respectively. The difference between these values and that of the control was only significant for the preparation at -4 °C (25 °F). At this temperature, the reduction in peel strength was about 32 % in comparison to the control. The failure mode for the LA sample sets prepared at the three temperatures was cohesive.*
- For the four "cold" tape-bonded samples prepared in the field, the mean peel strengths ranged from 0.42 kN/m to 1.13 kN/m (2.41 lbf/in to 6.48 lbf/in). All these "cold" sample sets failed adhesively. Examination of the failed specimens indicated that the EPDM rubber surface of the Wisconsin tape-bonded sample contained some dirt-like contamination. No observations of concern were

*The design of the Phase II experimentation did not include tests of liquid-adhesive-bonded samples and, consequently, no comparison of liquid-adhesive-bonded sample sets from Phase II and Task IV may be made.

Table 16. Summary of peel time-to-failure data for Task IV

	Adhesive Type	Temp. °C	Temp. °F	Conditioning After Field Prep.	Time-to-Failure, h min	max	mean[a]	sd[b]	CoV[c] %	Failure Mode[d]
LABORATORY	Tape—TS1	23	73	28 days min. at -4 °C (25 °F); RT[e] warm for specimen prep and test.	84.97	112.03	99.14	9.57	9.6	1
		0	32		1.79	2.54	2.18	0.30	13.8	2
		-4	25		1.81	6.46	3.47	1.52	44.0	2
	Tape—TS2	23	73		36.95	52.85	44.17	5.9	13.3	1
		0	32		32.73	68.70	43.11	11.1	25.9	1
		-4	25		45.38	73.70	56.18	10.0	17.8	1
	Liquid—LA	23	73		2.59	5.90	4.25	1.31	30.9	1
		0	32		1.42	5.65	3.39	1.28	37.7	1(2), 2(1), 3(5)
		-4	25		0.80	3.31	1.88	0.83	43.9	1(4), 2(1), 3(3)
FIELD	Tape—AK[f]	-14	7	"Cold" kept outdoors 28 day min; RT warm for specimen prep and test.	0.66	2.69	1.46	0.70	47.9	2
	Tape—MT	-8	18		0.10	1.04	0.40	0.35	87.2	2
	Tape—NH	-8	18		0.47	1.31	0.88	0.39	44.4	2
	Tape—WI	-19	-3		0.02	0.27	0.14	0.08	60.4	2
	Liquid—AK	-14	7		0.17	1.49	0.43	0.44	102	2
	Liquid—MT	-8	18		0.30	1.37	0.70	0.36	51.1	1
FIELD	Tape—AK	-14	7	"Warm" kept outdoors 28 day min; RT warm for specimen prep; RT for 28 days min. before test.	1.94	12.4	5.24	3.6	68.7	2
	Tape—NH	-8	18		0.82	3.03	1.78	0.67	37.5	2
	Tape—WI	-19	-3		0.04	0.32	0.17	0.11	61.4	2
	Liquid—AK	-14	7		0.36	1.14	0.62	0.26	41.9	2
	Liquid—MT	-8	18		0.28	1.69	0.63	0.45	70.6	1

[a]Mean of eight determinations. [b]sd indicates standard deviation. [c]CoV indicates coefficient of variation.
[d]Failure mode: 1 = cohesive; 2 = adhesive; 3 = mixed; numbers in parentheses indicate the number of specimens in the sample set that experienced the given failure mode; where there are no parentheses, all specimens failed by the given mode. [e]RT indicates room temperature, about 23 °C (73 °F). [f]The abbreviations are U.S. postal codes for states: AK = Alaska, MT = Montana, NH = New Hampshire, and WI = Wisconsin.

noted for Alaska, Montana, and New Hampshire samples, although the locus of failure with regard to the primer could not be ascertained. The 1.13 kN/m and 0.99 kN/m (6.48 lbf/in and 5.65 lbf/in) peel strengths for the Alaska and New Hampshire sample sets, respectively, were not statistically different from those of the TS1 tape-bonded samples prepared in the laboratory at -4 °C and 0 °C (25 °F and 32 °F). The Montana and Wisconsin tape-bonded sample sets were weaker than all tape-bonded samples prepared at cold temperatures in the laboratory.

- The peel strengths of the "cold" tape-bonded samples may be compared with those of adhesively-failing tape-bonded field seams, which have been found to range from 0.32 kN/m to 2.57 kN/m (1.83 lbf/in to 14.68 lbf/in) with an average of 0.97 kN/m (5.56 lbf/in) [17]. The peel strengths of the four "cold" sample sets were within this range—with those of Montana and Wisconsin being comparable to the low end values. For Alaska and New Hampshire, the mean peel strengths were about the same as the average value of the adhesively-failing field seams.

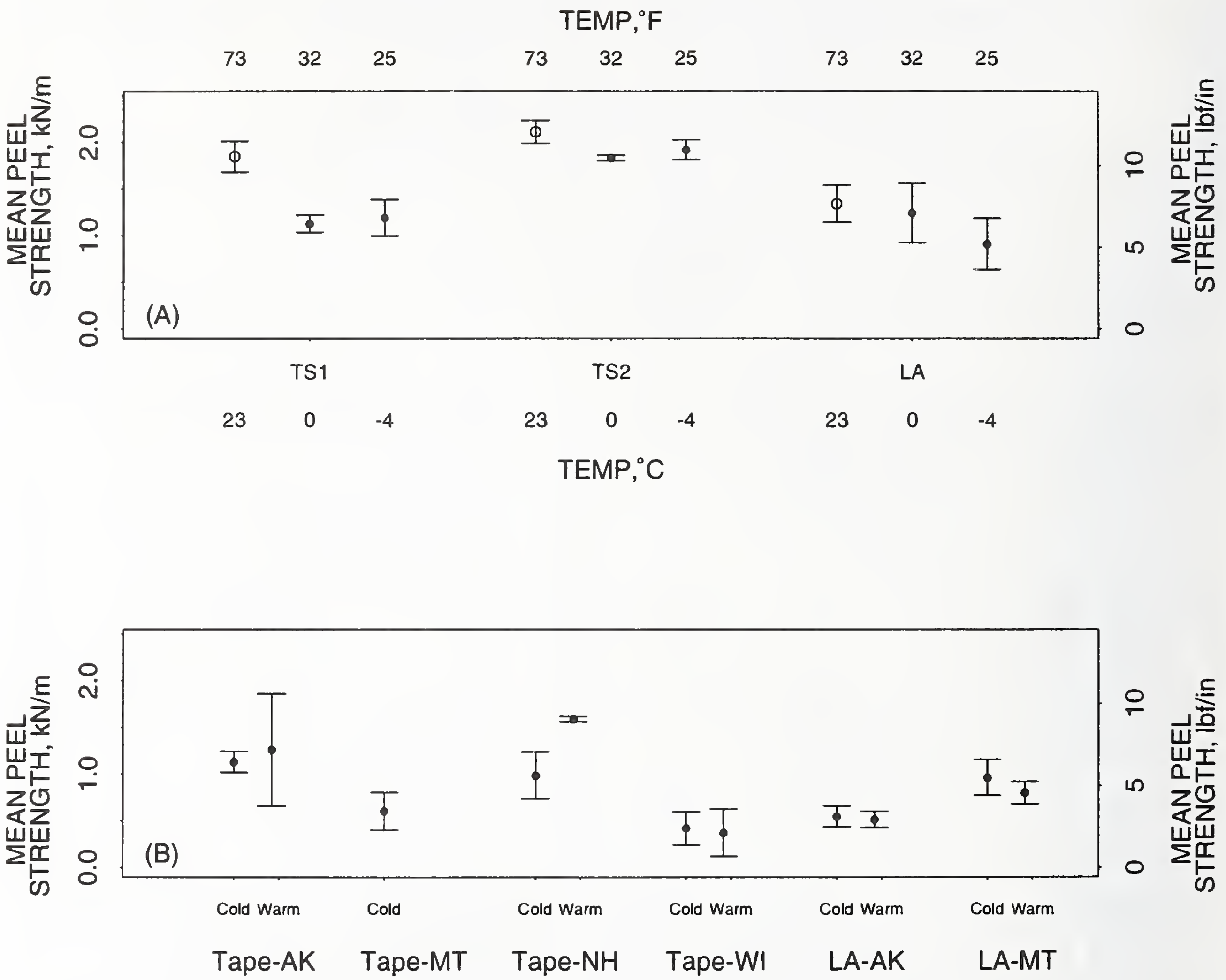

Figure 9. Mean Peel Strength of the Cold Temperature Prepared Samples: (A) Laboratory Prepared Including a Control at 23 °C (73 °F), and (B) Field Prepared. Tests Were Not Conducted On a "Warm" Set of Montana Tape-Bonded Samples.

- Peel strength tests were conducted on "warm" tape-bonded samples from Alaska, New Hampshire, and Wisconsin.* For the Alaska sample, no significant difference in mean peel strength was observed between the "cold" and "warm" sample sets. For the New Hampshire sample, keeping the specimens at 23 °C (73 °F) for 28 days before testing apparently resulted in increased peel strength. The mean peel strengths of the "cold" and "warm" sample sets were 0.99 kN/m and 1.59 kN/m (5.65 lbf/in and 9.07 lbf/in), respectively, with adhesive and mixed failure modes. It is known that tape-bonded seam samples prepared at room temperature increase in strength for about

*Tests were not conducted on the tape-bonded samples from Montana because of lack of specimens.

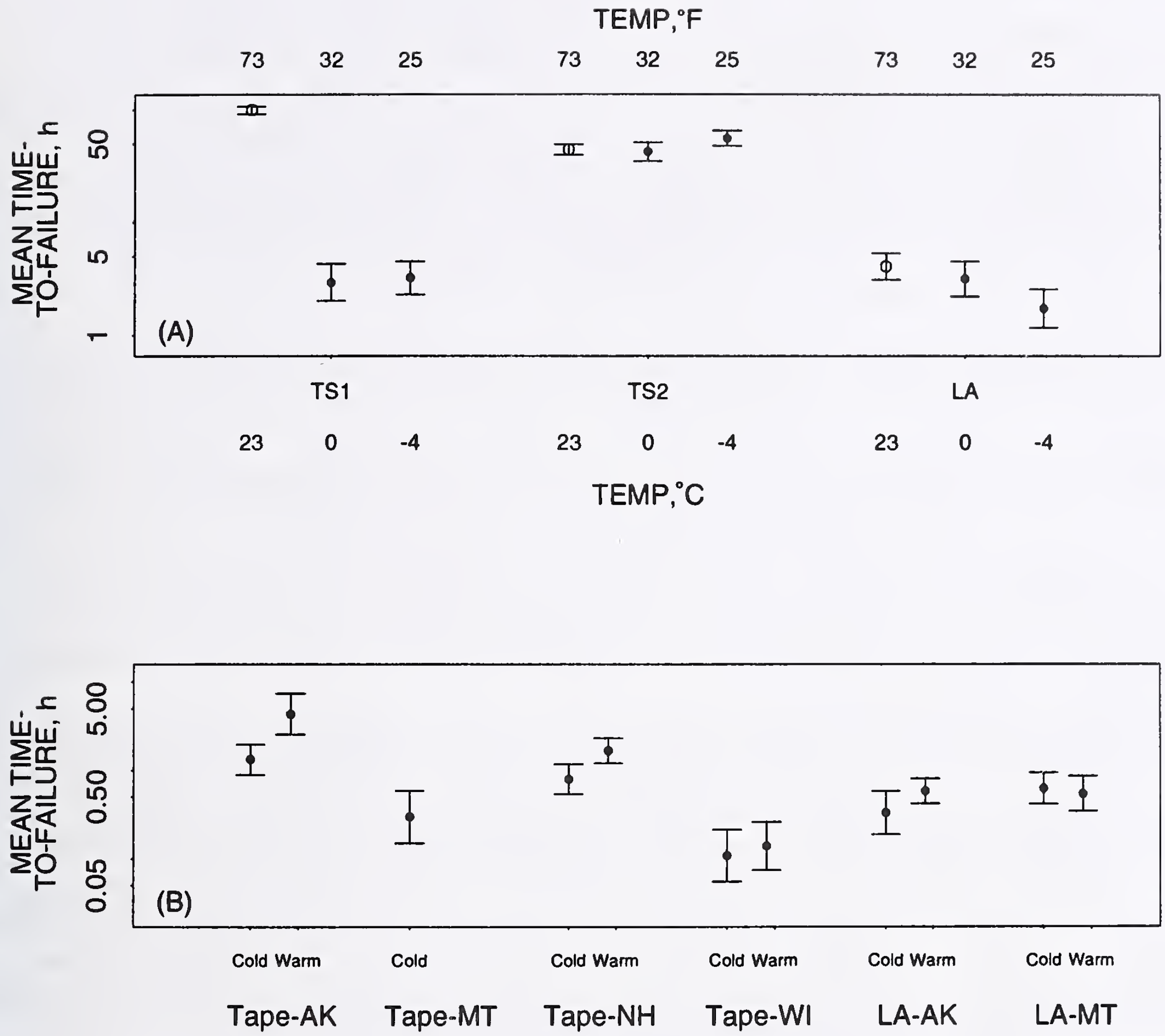

Figure 10. Mean Peel Time-to-Failure of the Cold Temperature Prepared Samples: (A) Laboratory Prepared Including a Control at 23 °C (73 °F), and (B) Field Prepared. Tests Were Not Conducted On a "Warm" Set of Montana Tape-Bonded Samples.

2 weeks to 3 weeks after fabrication [2]. The increase in strength between the "cold" and "warm" New Hampshire sample sets suggests that, at least in one case, the mechanism accounting for initial increased strength with time may be retarded if the samples remain cold after preparation, but that it may proceed if the samples are "warmed" to room temperature. In the case of the Wisconsin tape-bonded sample, no effect of "warming" to room temperature was observed. The "cold" and "warm" Wisconsin sample sets had mean peel strengths of 0.42 kN/m and 0.38 kN/m (2.41 lbf/in and 2.15 lbf/in), respectively, which were not significantly different. Similar to the "cold" Wisconsin sample set, the "warm" sample set had a dirt-like contaminated EPDM surface and

failed adhesively. It may be that the effect of EPDM surface contamination overrode any potential effect of "warming."

- In the case of the "cold" Alaska and Montana liquid-adhesive-bonded samples, the mean peel strengths were 0.55 kN/m and 0.96 kN/m (3.13 lbf/in and 5.51 lbf/in), respectively. In three previous studies [18-20], the peel strengths of such samples have ranged from 0.23 kN/m to 1.2 kN/m (1.3 lbf/in to 6.7 lbf/in) with an average of 0.5 kN/m (3 lbf/in). Thus, in comparison, the "cold" Montana sample set had a mean peel strength at the upper end of this range, whereas the Alaska sample set was average. Peel-strength measurements were also conducted on "warm" Alaska and Montana liquid-adhesive-bonded sample sets. The mean peel strengths were 0.52 kN/m and 0.80 kN/m (2.97 lbf/in and 4.58 lbf/in), which were not statistically different from those of the "cold" Alaska and Montana sample set, respectively.

From Table 16 and Figure 10, the following observations on peel creep-rupture behavior are noted:

- For the TS1 laboratory samples, the mean times-to-failure of the control sample set and of those prepared at -4 °C and 0 °C (25 °F and 32 °F) were 99.14 h, 3.47 h, and 2.18 h, respectively. The control sample set failed cohesively, whereas the cold temperature prepared sample sets failed adhesively at the interface of the tape and the primer. There was no statistical difference between the mean times-to-failure of the two cold temperature prepared sample sets.
- For the TS2 laboratory-prepared samples, there was no effect on time-to-failure due to cold temperature preparation. The mean times-to-failure of the control sample set and of those prepared at -4 °C and 0 °C (25 °F and 32 °F) were 44.17 h, 56.18 h, and 43.11 h, respectively. All sample sets failed cohesively.
- For the LA samples, the mean times-to-failure of the control sample set and of that prepared at 0 °C (32 °F) were 4.25 h and 3.39 h, respectively (no significant difference). In contrast, the 1.88 hour mean time-to-failure of the sample set prepared at -4 °C (25 °F) was significantly less than that of the control. The failure mode for the control sample set was cohesive; for the two cold temperature prepared sample sets, cohesive, adhesive, and mixed failures were observed.
- For the four "cold" tape-bonded samples prepared in the field, all sample sets failed adhesively with mean times-to-failure ranging from 0.14 h to 1.46 h. These times-to-failure were among the lowest determined in the Task IV studies. In comparison, in a limited set (5 roofs) of field samples that failed adhesively during creep-rupture testing, the three shortest mean times-to-failure were 0.05 h, 0.1 h, and 0.4 h [17]. Thus, although the Task IV cold temperature prepared field samples had low times-to-failure, the values were about the same as those found for adhesively failing seams sampled from roofs in service. As a final comment, it is noted that the Wisconsin sample set, which had the lowest mean time-to-failure of the "cold" tape-bonded sample sets, showed dirt-like EPDM surface contamination, as was the case for the Wisconsin peel-strength sample set.
- Peel creep-rupture tests were conducted on "warm" Alaska, New Hampshire, and Wisconsin tape-bonded sample sets. For Alaska and New Hampshire, the "warm" sample sets were significantly longer lived than the respective "cold" sample sets. In the Alaska case, the mean "cold" and "warm" times-to-failure were 1.46 h and 5.24 h, respectively. In the New Hampshire case, they were of 1.78 h and 0.88 h, respectively. For Wisconsin, the "warm" and "cold" sample sets were not statistically different.
- For the field liquid-adhesive samples from Alaska and Montana, the "cold" and "warm" mean times-to-failure were not significantly different. It is noted that the Alaska sample sets failed adhesively, whereas the Montana sample sets failed cohesively.

6.2.2 Comparison of Task IV Tape-Adhesive and Liquid-Adhesive Results. From Table 15 and Figure 9, the following observations comparing the Task IV peel-strength results for tape-bonded and liquid-adhesive-bonded systems are noted:

- For the laboratory samples, the tape-bonded and liquid-adhesive-bonded sample sets experienced, vis-a-vis the controls, decreased peel strength upon cold temperature preparation. The percent strength reductions for the TS1 and LA sample due to preparation at the colder temperature, -4 °C (25 °F), were about the same—approximately 35 % and to 30 %, respectively. At this temperature, TS1 was significantly stronger than LA—1.19 kN/m versus 0.91 kN/m (6.81 lbf/in versus 5.21 lbf/in). In the case of TS2, the 9 % reduction in peel strength versus its control was considerably less than that of the liquid-adhesive system.
- For the field samples, the four "cold" tape-bonded sample sets had mean peel strengths that were comparable to those of the "cold" LA sample sets. "Warming" the LA sample sets had no effect on peel strength. However, one "warm" tape-bonded sample set, New Hampshire, showed a significantly greater mean peel strength than its "cold" counterpart.

From Table 16 and Figure 10, the following observations comparing the Task IV peel creep-rupture results for the tape-bonded and liquid-adhesive-bonded systems are noted:

- For the laboratory samples, LA mean times-to-failure decreased upon cold temperature preparation. In comparison, TS1 mean time-to-failure also decreased, while that of TS2 was unaffected. The reduction in mean time-to-failure due to cold temperature preparation was greater for TS1 than for LA. This was evidenced in that the mean time-to-failure of the -4 °C (25 °F) TS1 sample set was a factor of approximately 28 less than that of its control, whereas the mean times-to-failure for the two LA sample sets differed by a factor of only 2.3. Nevertheless, although TS1 was more affected than LA by the cold temperature preparation, the mean time-to-failure of the -4 °C (25 °F) TS1 sample set was still greater than that of the corresponding LA sample set. The reason was that the mean time-to-failure of the TS1 control was considerably greater than that of the LA control.
- For the field samples, the "cold" tape-bonded sample sets and the "cold" liquid-adhesive-bonded sample sets had comparable times-to-failure. For both adhesive types, the mean times-to-failure for the "cold" field sample sets were less than those of the cold temperature prepared laboratory sample sets. Less control is likely applied when preparing samples in the field than in the laboratory and, consequently, a factor(s) other than application temperature may contribute to the creep-rupture behavior of field prepared samples.

7. SHEAR TESTING (TASK V)

7.1 Objective and Scope of Task V

The Task V investigation was conducted to characterize shear-creep resistance and shear strength, and to compare the performance of tape-bonded and liquid-adhesive-bonded sample sets under shear loading. Shear strengths of tape-bonded and liquid-adhesive-bonded specimens were measured at 23 °C, 40 °C, and 70 °C (73 °F, 104 °F and 158 °F). Shear creep-rupture tests were performed at 23 °C (73 °F) under loads of 28.0 N, 31.1 N, and 34.2 N, (6.3 lbf, 7.0 lbf, and 7.7 lbf), and at 70 °C (158 °F) under loads of 24.9 N and 28.0 N (5.6 lbf and 6.3 lbf). Table 17 summarizes the Task V test conditions and lists the number of specimens tested at each condition.

Loading greater than 34.2 N (7.7 lbf) was not performed because the creep-rupture chambers were not designed for such loads. A 34.2 N (7.7 lbf) shear creep-rupture load was approximately 20 % to 25 % of the shear strengths of the seam samples determined at 23 °C (73 °F).

Past experience in conducting shear creep-rupture tests on liquid-adhesive-bonded sample sets at 23 °C (73 °F) under loads representing about 20 % of the shear strength has shown that the failures did not occur for many months [5,7]. In designing the Task V experiment, it was planned to conduct the shear creep-rupture tests for no longer than 1 month so that a chamber would not be dedicated to a single test for a long period. This design was followed for the tests at 23 °C (73 °F). Tests at 70 °C (158 °F) under loads of 24.9 N and 28.0 N (5.6 lbf and 6.3 lbf) were conducted for 2 months and 2.5 months, respectively.

7.2 Task V Results and Discussion

Tables 18 and 19 summarize the shear-strength and shear creep-rupture data for Task V, respectively. Figure 11 is a plot of mean shear strength versus adhesive system for the three test temperatures. The shear time-to-failure data in Table 19 are not plotted because few, if any, of the specimens in a sample set failed under some test conditions.

Table 17. Summary of test conditions and number of specimens for Task V

Adhesive System	Test Temp. °C	Test Temp. °F	Shear Strength Tests No. of Specimens	Shear Creep-Rupture Tests: No. of Specimens, Load, N (lbf) 24.9 (5.6)	28.0 (6.3)	31.1 (7.0)	34.2 (7.7)
TS1	23	73	4	---[a]	8	8	8
	40	104	4	---	---	---	---
	70	158	4	8	8	---	---
TS2	23	73	4	---	8	8	8
	40	104	4	---	---	---	---
	70	158	4	8	8	---	---
LA	23	73	4	---	8	8	8
	40	104	4	---	---	---	---
	70	158	4	8	8	---	---

[a]The dash indicates that tests were not conducted.

For the shear-strength tests, the failure modes were cohesive. For the creep-rupture tests, the failure modes were generally cohesive with the exception of some samples of TS2 at both 23 °C and 70 °C (73 °F and 158 °F). In this case, five specimens failed adhesively and one failed in a mixed mode. It was also apparent that, in shear, the creep-rupture tape-bonded specimens, and particularly TS2, underwent two types of cohesive failure at the elevated temperatures. Some underwent a slip-stick [15] type of failure, whereby shearing apparently occurred stepwise over sections of the bond rather than by a somewhat constant-shearing phenomenon. Figure 12 illustrates these two types of failure. Note the uneven adhesive surface for the slip-stick failure in comparison to the relatively smooth surface for the constant rate failure. Failed liquid-adhesive-bonded shear specimens did not display slip-stick behavior. Also, it is noted that slip-stick behavior of peel specimens under creep-rupture testing has not been observed during any phase of the study.

7.2.1 Effect of Shear Testing on the Adhesive Systems. From Table 18 and Figure 11, the following observations on shear strength are noted:

- The TS1 and TS2 mean shear strengths at 23 °C (73 °F) were 5.59 kN/m and 7.46 kN/m (31.9 lbf/in and 42.6 lbf/in), respectively. This is the first nonproprietary report of shear-strength data on tape-bonded seam samples wherein the measurements were made 28 days after the specimens were fabricated. Dupuis [13] reported shear-strength data on specimens that were 7 days old. His values ranged from about 3.5 kN/m to 5.8 kN/m (20 lbf/in to 33 lbf/in). They are lower than those reported in the present study, perhaps due to the age of the samples. It is known that peel strength of tape-bonded samples increases at 23 °C (73 °F) for about 2 weeks to 3 weeks after sample fabrication [2].
- For the LA sample set, the mean shear strength at 23 °C (73 °F) was 6.95 kN/m (39.7 lbf/in). This LA shear strength was the same as the value, 6.90 kN/m (39.4 lbf/in), reported by Rossiter, Martin, Lechner, Embree, and Seiler in an earlier NIST study [5].
- Similar to the peel-strength findings in Task I, mean shear strength of the three adhesive systems decreased with increasing temperature. For the range of test temperatures, the strength-temperature relationships tended to be linear. The lines in Figure 11 represent least squares-fits to the data. The r^2-values for TS1, TS2, and LA are 0.91, 0.96, and 0.97, respectively.

Table 18. Summary of shear-strength data for Task V

Adhesive System	Temp. °C	Temp. °F	Shear Strength, kN/m min	max	mean[a]	sd[b]	Shear Strength, lbf/in min	max	mean[a]	sd[b]	CoV[c] %	Failure Mode[d]
TS1	23	73	5.33	5.75	5.59	0.21	30.4	32.9	31.9	1.17	3.7	1
	40	104	3.67	3.90	3.79	0.10	20.9	22.3	21.7	0.55	2.5	1
	70	158	2.47	2.74	2.63	0.11	14.1	15.7	15.0	0.65	4.3	1
TS2	23	73	6.68	7.80	7.46	0.52	38.1	44.5	42.6	2.99	7.0	1
	40	104	5.33	6.10	5.78	0.33	30.4	34.8	33.0	1.87	5.7	1
	70	158	2.70	3.48	3.20	0.34	15.4	19.8	18.2	1.93	10.6	1
LA	23	73	6.76	7.26	6.95	0.22	38.6	41.5	39.7	1.23	3.1	1
	40	104	4.48	4.98	4.71	0.21	25.58	28.44	26.90	1.21	4.5	1
	70	158	2.36	2.59	2.44	0.11	13.45	14.77	13.95	0.63	4.5	1

[a]Mean of four determinations. [b]sd indicates standard deviation. [c]CoV indicates coefficient of variation.
[d]Failure mode: 1 = cohesive.

Table 19. Summary of shear time-to-failure data for Task V

Adhesive System	Temp. °C	Temp. °F	Load N	Load lbf	Surviving Specimens Number	Surviving Specimens Test Period	Ordered Time-to-Failure Spec. No.	Ordered Time-to-Failure TTF, h	Ordered Time-to-Failure Mean TTF[a]	Failure Mode[b]
TS1	23	73	28.0	6.3	8	1 month	---	---	---	---
			31.1	7.0	8	1 month	---	---	---	---
			34.2	7.7	7	1 month	1	219.8	---	1
	70	158	24.9	5.6	0[c]	NA[d]	1	2.44	26.8[e]	1
							2	4.13		1
							3	7.43		1
							4	9.59		1
							5	13.16		1
							6	63.15		1
							7	87.82		1
			28.0	6.3	0	NA	1	1.62	3.35	1
							2	1.91		1
							3	3.03		1
							4	3.55		1
							5	3.58		1
							6	3.59		1
							7	3.60		1
							8	5.96		1
TS2	23	73	28.0	6.3	7	1 month	1	23.9	---	2
			31.1	7.0	2	1 month	1	10.9	---	2
							2	25.6		2
							3	297.5		1
							4	445.1		1
							5	470.6		3
							6	632.4		1
			34.2	7.7	0	NA	1	3.26	165.4	2
							2	134.9		1
							3	177.2		1
							4	184.5		1
							5	187.4		1
							6	200.1		1
							7	207.3		1
							8	228.4		1
	70	158	24.9	5.6	0	NA	1	2.32	27.0	2
							2	8.98		1
							3	13.16		1
							4	15.92		1
							5	16.45		1
							6	42.58		1
							7	43.15		1
							8	73.49		1
			28.0	6.3	0[c]	NA	1	4.68	7.63[e]	1
							2	5.73		1
							3	5.96		1
							4	6.31		1
							5	9.05		1
							6	10.18		1
							7	11.53		1
LA	23	73	28.0	6.3	8	1 month	---		---	---
			31.1	7.0	8	1 month	---		---	---
			34.2	7.7	8	1 month	---		---	---
	70	158	28.0	6.3	4[c]	2.5 months	1	14.01		1
							2	79.01	---	1
							3	96.04		1
			24.9	5.6	8	2 months	---	---	---	---

[a]The mean time-to-failure is given for those sample sets for which all specimens failed. [b]Failure Mode: 1 = cohesive; 2 = adhesive; 3 = mixed. [c]The time-to-failure of one specimen was not recorded due to an experimental problem. [d]NA indicates not applicable. [e]Mean is calculated for the recorded time-to-failures.

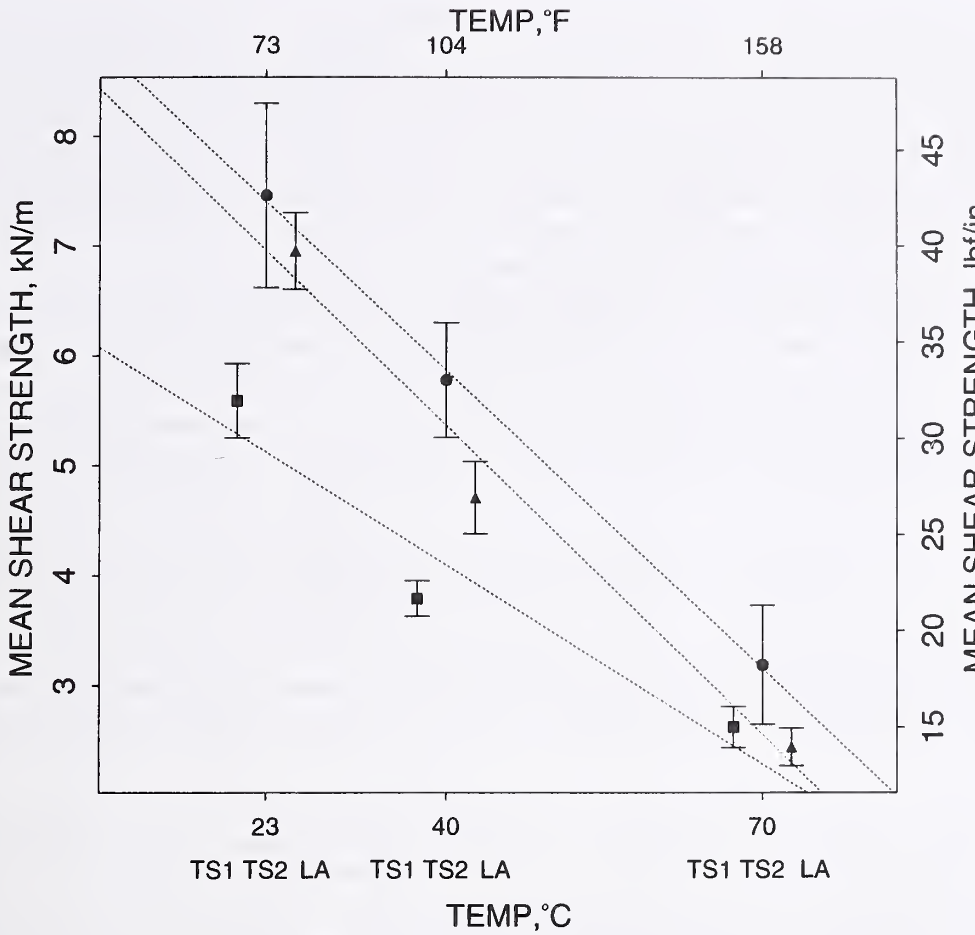

Figure 11. Mean Shear Strength as a Function of Temperature for Each Adhesive System.

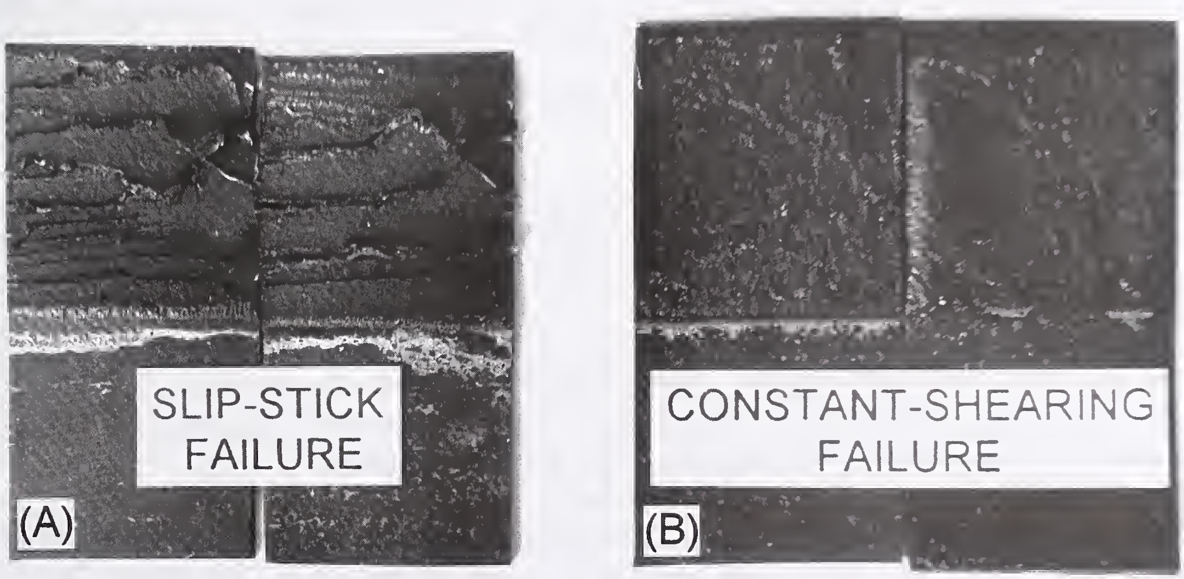

Figure 12. Photograph of Typical Failed Surfaces of Tape-Bonded Specimens Tested in Shear: (A) Slip-Stick Shear Propagation and (B) Constant-Shearing Propagation

From Table 19, the following observations on shear creep-rupture behavior are noted:

- For TS1, when the tests were conducted at 23 °C (73 °F), failures were not observed during the 1-month test period with the exception of one specimen of the sample set loaded at 34.2 N (7.7 lbf). Increasing test temperature resulted in decreased shear time-to-failure. Whereas at 23 °C (73 °F) under the 28.0 N (6.3 lbf) load, no specimens failed; at 70 °C (158 °F) and the same load, the entire sample set failed. Also, at 70 °C (158 °F), increasing the load resulted in decreased time-to-failure. At this temperature, the mean shear times-to-failure for the 24.9 N and 28.0 N (5.6 lbf and 6.3 lbf) loads were 26.8 h and 3.35 h, respectively.
- For TS2, failures were observed at 23 °C (73 °F) for the three loads. Qualitatively, shear time-to-failure increased with decreasing load. For example, all specimens in the sample set loaded at 34.2 N (7.7 lbf) failed with a mean time-to-failure of 165.4 h. In contrast, only one specimen failed in 1 month under the 28.0 N (6.3 lbf) load. At 70 °C (158 °F), all specimens failed with mean shear times-to-failure of 27.0 h and 7.63 h when loaded at 24.9 N and 28.0 N (5.6 lbf and 6.3 lbf), respectively.
- For LA, no failures were observed at 23 °C (73 °F) at any load. Also, no failures occurred at 70 °C (158 °F) at the 24.9 N (5.6 lbf) load. At 70 °C (158 °F) under the 28.0 N (6.3 lbf) load, four specimens failed. The 70 °C (158 °F) tests were conducted for at least 2 months. Based on the results of Task II, wherein it was found that the creep lifetime of LA peel samples can be prolonged due to heat exposure, it may possibly be that the longer the LA shear specimens survived in the creep chambers at 70 °C (158 °F), then the longer they would be expected to survive.

7.2.2 <u>Comparison of Task V Tape-Adhesive and Liquid-Adhesive Results</u>. From Table 18 and Figure 11, the following observation comparing the Task V shear-strength results for the tape-bonded and liquid-adhesive-bonded sample sets systems is noted:

- For tests conducted at room temperature, LA and TS2 had comparable shear strengths (the difference was not significant); TS1 had lower mean shear strength than LA. However, the effect of increasing temperature on decreasing shear strength was less for TS1 than for TS2 and LA, where the effect was about the same. This is evidenced by the slopes of the lines (fig. 11). Thus, at the highest temperature of 70 °C (158 °F), the mean shear strengths of TS1 and LA were comparable (no significant difference), while that of TS2 was greater than that of LA. The difference was significant.

From Table 19, the observations comparing the Task V shear creep-rupture results for the tape-bonded and liquid-adhesive-bonded sample sets systems are given below. Note that the comparisons are qualitative, because many of the shear tests, and in particular those at room temperature, produced few, if any, failures.

- At 23 °C (73 °F), no comparison between TS1 and LA may be made because specimens of either adhesive type did not generally fail. Qualitatively, TS2 was shorter lived than LA. Under the 31.1 N and 34.2 N (7.0 lbf and 7.7 lbf) loads, 6 TS2 specimens and 8 TS2 specimens failed, respectively; whereas again in comparison, no LA specimens failed.
- At 70 °C (158 °F), both TS1 and TS2 had shorter shear-creep lifetimes than LA. No tape-bonded specimens survived the testing, and the maximum mean time-to-failure for a sample set was less than 30 h. In contrast, most LA specimens survived for test periods of 2 months (i.e., about 720 h) or more.

8. STANDARD TEST PROTOCOL FOR TAPE-BONDED SEAMS

As indicated in Section 1, an objective of the industry-government consortium study of tape-bonded seams of EPDM membranes is to recommend a standard test protocol for evaluating creep-rupture performance. The availability of such a protocol is important, because the use of tape adhesives for EPDM membranes is expected to continue to increase and new tape products may appear on the market. A test protocol based on the present consortium study would allow creep-rupture data developed on new products to be compared with those reported herein and in previous reports [3,4]. Moreover, the availability of the test protocol would allow criteria for creep-rupture resistance to be incorporated in standard specifications developed for EPDM tape adhesives.

Table 20 provides a proposal for sample preparation temperatures, test parameters, and exposure conditions for incorporation in a test protocol. This proposal is based on the results of the Phase III study, and includes conducting creep-rupture tests on seven sets of samples—five under peel loading and two under shear loading. Table 20 can provide the basis of a voluntary consensus standard such as promulgated by the ASTM (American Society of Testing and Materials).

Regarding the proposal in Table 20, Sample Set No. 1 is a control for tests under peel loading, and differs from Sample Set No. 2 in that the test temperatures are 23 °C and 70 °C (73 °F and 158 °F), respectively. Sample Set No. 3 is prepared at -4 °C (25 °F), and tested at 23 °C (73 °F). The inclusion of Sample Sets Nos. 2 and 3 in the proposed protocol is based on the results of Task I and Task IV, respectively, which showed that test temperature and low temperature preparation affect peel time-to-failure.

Sample Set No. 4 is subjected to peel creep-rupture testing after exposure to dry heat; whereas Sample Set No. 5 is tested after exposure to dry heat, wet heat, and freeze-thaw cycling (i.e., the SPRI exposure). These sample sets are included in the proposed protocol because the Task II and Task III findings indicated that these respective exposures also affect peel time-to-failure.

In shear, Sample Set No. 6 is a control, and differs from Sample Set No. 7 in that the test temperatures are 23 °C and 70 °C (73 °F and 158 °F), respectively. The effects of shear loading were investigated in Task V.

Table 20. Sample preparation temperatures, test parameters, and exposure conditions proposed for incorporation in a test protocol

Sample Set	Sample Preparation Temperature		Test Parameters					Artificial Exposure Before Testing
			Temperature		Loading	Load[a]		
No.	°C	°F	°C	°F	Peel	N	lbf	Conditions
1	23	73	23	73	Peel	9.8	2.2	None
2	23	73	70	158	Peel	9.8	2.2	None
3	-4	25	23	73	Peel	9.8	2.2	None
4	23	73	23	73	Peel	9.8	2.2	Heat: 90 °C (194 °F) for 90 days
5	23	73	23	73	Peel	9.8	2.2	Dry heat, wet heat, and freeze-thaw cycling according to SPRI procedure (see Table 9)
6	23	73	23	73	Shear	29.4	6.6	None
7	23	73	70	158	Shear	29.4	6.6	None

[a]The peel and shear loads used in Phase III were 9.3 N and 28 N (2.1 lbf and 6.3 lbf), respectively. The proposal for the test protocol suggests conducting the peel tests at 9.8 N (2.2 lbf) and shear tests at 29.4 N (6.6 lbf) because these values correspond to 1 kg and 3 kg, respectively. Dead weights of 1 kg mass and 3 kg mass may be available through laboratory equipment supply houses.

9. SUMMARY AND CONCLUSIONS

The use of tape adhesive systems for fabricating field seams of EPDM membranes has significantly increased in the 1990s. A three-phased industry-government consortium study has been conducted to develop nonproprietary data on tape-bonded seam performance. In Phase I, the creep-rupture response (time-to-failure) of tape-bonded seam samples subjected to various peel loads under ambient conditions was compared to that of liquid-adhesive-bonded samples. In Phase II, two material factors (tape system and tape thickness) and five application factors (EPDM surface condition, primer, application temperature, application pressure, and time-at-application temperature) were investigated in a statistically designed experiment for their effects on peel creep-rupture response. In Phase III, four tasks investigated the effects of: (I) elevated test temperatures, (II) elevated temperature exposure prior to loading, (III) exposure to industry-developed protocols (i.e., Rubber Manufacturers Association and SPRI), and (IV) cold temperature preparation on peel creep-rupture response and on peel strength. Phase III Task (V) examined shear testing. The Consortium Oversight Committee members reasoned that each task was important for characterizing the general creep-rupture behavior of tape-bonded seams, and for complementing the data developed in Phases I and II. Moreover, the results from these five Phase III investigations would provide guidance as to factors to be incorporated in a test protocol for evaluating the creep resistance of EPDM adhesive-bonded seams. For each Phase III task, comparisons of the creep-rupture responses and strength of tape-bonded and liquid-adhesive-bonded samples were made.

Two commercial tape systems (i.e., tape and primer) and one liquid adhesive were applied to well-cleaned EPDM rubber in preparing the samples. Each of the Phase III tasks were performed in five independent investigations:

- In Task I, peel times-to-failure were determined at 23 °C, 40 °C, and 70 °C (73 °F, 104 °F and 158 °F) under each of three loads, 9.3 N, 12.5 N, and 15.6 N (2.1 lbf, 2.8 lbf, and 3.5 lbf). Additionally, at 70 °C (158 °F), a test was performed under a 6.2 N (1.4 lbf) load.
- In Task II, specimens were heated at 60 °C, 75 °C, and 90 °C (140 °F, 167 °F, and 194 °F) for 30 days, 90 days, and 150 days. After exposure, peel times-to-failure were measured at 23 °C (73 °F) under a 9.3 N (2.1 lbf) load. Peel time-to-failure was also determined for room temperature control specimens.
- In Task III, peel times-to-failure were determined on specimens after laboratory exposure to the protocols developed by the Rubber Manufacturers Association (RMA) and SPRI. Both protocols subject samples to dry heat in an oven, heat in water, and freeze-thaw cycling. The SPRI protocol also requires that the specimens be elongated in shear at 20 % under some of these exposure conditions.
- In Task IV, peel times-to-failure of specimens prepared either in the laboratory or in the field at temperatures of about 0 °C (32 °F) or less were determined. In the laboratory, sample preparation was performed in a temperature-humidity controlled glove box. In the field, roofing mechanics prepared samples outdoors using roof-top application procedures.
- In Task V, shear times-to-failure were measured at 23 °C (73 °F) under loads of 28.0 N, 31.1 N, and 34.2 N, (6.3 lbf, 7.0 lbf, and 7.7 lbf), and at 70 °C (158 °F) under loads of 24.9 N and 28.0 N (5.6 lbf and 6.3 lbf).

The conclusions regarding the comparisons of the creep-rupture responses of tape-bonded and liquid-adhesive-bonded samples from the Phase III experimentation were that:

- In Task I, as the temperature and load of the creep test increased, the peel time-to-failure of the three adhesive systems decreased. For any combination of temperature and load, the tape-bonded sample sets had longer mean peel time-to-failure than the liquid-adhesive-bonded sample sets.
- In Task II, when exposed to various elevated temperatures for varying times, peel time-to-failure of liquid-adhesive-bonded samples was either unaffected or increased versus that of the room temperature control sample. In comparison, peel time-to-failure of one tape-bonded system increased, and that of the other tape system decreased or was unaffected depending upon the exposure. Where increased peel times-to-failure were observed, under some conditions, they were quite substantial; however, no evidence is available indicating that tape-bonded field seams (i.e., those sampled from roofs) have shown such prolonged times-to failure. Also, in cases where laboratory samples experienced decreased peel time-to-failure, the resultant values were not less than peel times-to-failure determined for field seams.
- In Task III under the RMA protocol, mean peel time-to-failure of liquid-adhesive-bonded samples increased upon exposure. In comparison, one tape system showed substantially increased peel time-to-failure, and the other exhibited decreased peel time-to-failure. In the case of the SPRI protocol, mean peel time-to-failure of liquid-adhesive-bonded samples increased upon exposure. In comparison, one tape system was unchanged, whereas the other decreased. The results were compared with peel times-to-failure of field seams. This comparison indicated that substantially long peel times-to-failure of the magnitude of those observed for the laboratory heat exposed samples have not been evidenced for field seams. Also, where decreases in peel time-to-failure were observed for seams upon laboratory exposure, the resultant mean peel times-to-failure were not atypical of values measured for some field seams.
- In Task IV, in the case of laboratory samples, mean peel times-to-failure of the liquid-adhesive-bonded system decreased upon cold temperature preparation. In comparison, mean peel time-to-failure of one tape system also decreased; while that of the other tape system was unaffected. Where decreases occurred for the colder preparation temperature of -4 °C (25 °F), the mean peel time-to-failure of the tape system was greater than that of the liquid-adhesive-bonded system. In the case of field-prepared samples, the tape-bonded samples and the liquid-adhesive-bonded sample sets had comparable peel times-to-failure. For both adhesive types, these values for the field samples were less than those of the samples prepared in the laboratory at cold temperatures. The field samples were prepared at temperatures lower than those used in the laboratory.
- In Task V, many shear creep-rupture tests, particularly those at room temperature, produced few failures within the allotted 1-month test time. At 70 °C (158 °F) and loads of 24.9 kN/m and 28.0 kN/m (5.6 lbf/in and 6.3 lbf/in), both tape systems had shorter shear-creep lifetimes than the liquid adhesive system. The maximum mean shear time-to-failure for a tape-bonded sample set was less than 30 h; while, in contrast, most of the liquid-adhesive bonded specimens survived for 2 months or more.

10. ACKNOWLEDGMENTS

The Phase III research described in this report was jointly sponsored by NIST and the CRADA members. The authors acknowledge with thanks the support of the CRADA organizations and their representatives: Dennis Fisher (Adco), David Hatgas (Ashland), Daniel Cotsakis (Carlisle SynTec), Chester Chmiel (Firestone), Thomas Smith (NRCA), and Joe Hale (RCI). The authors also thank their NIST colleagues who contributed to the study. Jack Lee assisted with the creep-rupture and peel-strength measurements. Edward Embree and Philip Embree modified the creep chambers for elevated temperature experiments. Joannie Chin, Geoffrey Frohnsdorff, Jonathan Martin, Carl Schultheisz, and Shyam Sunder reviewed the report. Donald Hunston related his experiences in discussing adhesive testing.

Especially acknowledged is William Cullen who retired as the NRCA Research Associate in September 1997. Prior to his retirement, he was a member of the Consortium Oversight Committee, and contributed significantly to all phases of the study. With sadness, the authors report that William Cullen passed away on November 5, 1997 as they were completing the Phase III research. The authors, as well as the industry members of the consortium Oversight Committee, remember his valuable contributions to the study including noteworthy suggestions on the study design, constructive comments on previous reports, and general enthusiasm on the benefits of the study towards improving the performance of EPDM roofing. The authors were privileged to have worked closely with him.

11. REFERENCES

[1] Hatgas, David J. and Spector, Richard C., "Tape Products Used in EPDM Roofing Systems," *Proceedings, ACS Rubber Division Symposium*, American Chemical Society, Philadelphia, PA (May 4 1995), 26 pages.

[2] Rossiter, Walter J., Jr., Lechner James A., Seiler, James F., Jr., and Embree, Edward, "Performance of Tape-Bonded Seams of EPDM Membranes: Initial Characterization," *Proceedings, 11th Conference on Roofing Technology*, U.S. National Roofing Contractors Association, Rosemont, IL (September 1995), pp. 78-89.

[3] Rossiter, Walter J., Jr., Vangel, Mark G., Embree, Edward, Kraft, Kevin M., and Seiler, James F., Jr., "Performance of Tape-Bonded Seams of EPDM Membranes: Comparison of the Peel Creep-Rupture Response of Tape-Bonded and Liquid-Adhesive-Bonded Seams," Building Science Series 175, National Institute of Standards and Technology (May 1996), 73 pages.

[4] Rossiter, Walter J., Jr., Vangel, Mark G., Kraft, Kevin M., and Filliben, James J., "Performance of Tape-Bonded Seams of EPDM Membranes: Effect of Material and Application Factors on Peel Creep-Rupture Response," Building Science Series 176, National Institute of Standards and Technology (May 1997), 61 pages.

[5] Rossiter, Walter J., Jr., Martin, Jonathan W., Lechner, James A., Embree, Edward, and Seiler, James F., Jr., "Effect of Adhesive Thickness and Surface Cleanness on Creep-Rupture Performance of EPDM Peel and Lap-Shear Joints," *Roofing Research and Standards Development: 3rd Volume*, ASTM STP 1224, American Society for Testing and Materials, West Conshohocken, PA (June 1994), pp. 123-138.

[6] Martin, Jonathan W., Embree, Edward, Stutzman, Paul E., and Lechner, James A., "Strength and Creep-Rupture Properties of Adhesive-Bonded EPDM Joints Stressed in Peel," Building Science Series 169, National Institute of Standards and Technology, Gaithersburg, MD (May 1990), 59 pages.

[7] Martin, Jonathan W., Rossiter, Walter J., Jr., and Embree, Edward, "Factors Affecting the Strength and Creep-Rupture Properties of EPDM Joints," *Proceedings, Third International Symposium on Roofing Technology*, U.S. National Roofing Contractors Association, Rosemont, IL (April 1991), pp. 63-71.

[8] Rossiter, Walter J., Jr., Martin, Jonathan W., Embree, Edward, Seiler, James F., Jr., Byrd, W. Eric, and Ream, Ed, "The Effect of Ozone on the Creep-Rupture of Butyl-Adhered EPDM Seam Specimens," *Proceedings, 10th Conference on Roofing Technology*, U.S. National Roofing Contractors Association, Rosemont, IL (April 1993), pp. 85-92.

[9] Martin, Jonathan W., Embree, Edward, and Bentz, Dale P., "Effect of Time and Stress on the Time-to-Failure of EPDM T-Peel Joints," *Proceedings, 8th Conference on Roofing Technology*, U.S. National Roofing Contractors Association, Rosemont, IL (April 1987), pp. 69-74.

[10] Rossiter, Walter J., Jr., Nguyen, Tinh, Byrd, W. Eric, Seiler, James F., Jr., Lechner, James A., and Bailey, David M., "Cleaning Aged EPDM Rubber Roofing Membrane Material for Patching: Laboratory Investigations and Recommendations," USACERL Technical Report FM-92/05, U.S. Army Construction Engineering Research Laboratory, Champaign, IL (August 1992), 58 pages.

[11] "Minimum Peel Strength Requirements for Adhesives Used in Seaming Black EPDM Membranes," RP-10, Rubber Manufacturers Association, Washington, DC (1989), 3 pages.

[12] "Recommended Laboratory Test Methods for Liquid and Tape Adhesives Used to Splice Single Ply Membranes," SPRI, Needham, MA (July 1995), 32 pages.

[13] Dupuis, R.M., "Splice Tape for Use in EPDM Roof Systems," Midwest Roofing Contractors Association 1994 Convention Program, Midwest Roofing Contractors Association, Kansas City, MO (October 1994), 8 pages.

[14] ASTM E 104 - 85 (Reapproved 1991), "Standard practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions," Annual Book of Standards, Vol. 08.03, American Society for Testing and Materials, West Conshohocken, PA (1995).

[15] Kaelble, D.H., "Theory and Analysis of Peel Adhesion: Rate-Temperature Dependence of Viscoelastic Interlayers," J. Colloid Science, Vol. 19 (1964), pp. 413-424.

[16] Rosenfield, Myer J., "Field Test Results of Experimental EPDM and PUT Roofing," U.S. Army Corps of Engineers Construction Engineering Research Laboratories, Technical Report M-357 (September 1984), p. 34.

[17] Rossiter, Jr.,Walter J., Vangel, Mark G., and Kraft, Kevin M., "Performance of Tape-Bonded Seams of EPDM Membranes: a Field Investigation," Proceedings of the Xth Congress of the International Waterproofing Association Held in Copenhagen, International Waterproofing Association, Basford, Nottingham, England (10-12 June 1998).

[18] Rossiter, Walter J., Jr., Seiler, James F., Jr., Spencer, William P., and Stutzman, Paul E., "A Field Study of the Performance of EPDM Roofing at Air Force Facilities," National Institute of Standards and Technology, NISTIR 4504 (January 1991), 77 pages.

[19] Rossiter, Walter J., Jr., Seiler, James F., Jr., Spencer, William P., Lechner, James A., and Stutzman, Paul E., "Characteristics of Adhesive-Bonded Seams Sampled From EPDM Roof Membranes," Proceedings of the Third International Symposium on Roofing Technology, U.S. National Roofing Contractors Association, Rosemont, IL (April 1991), pp. 167-179.

[20] Dupuis, Rene M., "What Contractors Should Know About the Seams of EPDM Roof Membranes," Contractors Guide, (October 1991), pp. 34-41.

NIST *Technical Publications*

Periodical

Journal of Research of the National Institute of Standards and Technology—Reports NIST research and development in those disciplines of the physical and engineering sciences in which the Institute is active. These include physics, chemistry, engineering, mathematics, and computer sciences. Papers cover a broad range of subjects, with major emphasis on measurement methodology and the basic technology underlying standardization. Also included from time to time are survey articles on topics closely related to the Institute's technical and scientific programs. Issued six times a year.

Nonperiodicals

Monographs—Major contributions to the technical literature on various subjects related to the Institute's scientific and technical activities.

Handbooks—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications—Include proceedings of conferences sponsored by NIST, NIST annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

National Standard Reference Data Series—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a worldwide program coordinated by NIST under the authority of the National Standard Data Act (Public Law 90-396). NOTE: The Journal of Physical and Chemical Reference Data (JPCRD) is published bimonthly for NIST by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements are available from ACS, 1155 Sixteenth St., NW, Washington, DC 20056.

Building Science Series—Disseminates technical information developed at the Institute on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

Technical Notes—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NIST under the sponsorship of other government agencies.

Voluntary Product Standards—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The standards establish nationally recognized requirements for products, and provide all concerned interests with a basis for common understanding of the characteristics of the products. NIST administers this program in support of the efforts of private-sector standardizing organizations.

Order the **following** *NIST publications—FIPS and NISTIRs—from the National Technical Information Service, Springfield, VA 22161.*

Federal Information Processing Standards Publications (FIPS PUB)—Publications in this series collectively constitute the Federal Information Processing Standards Register. The Register serves as the official source of information in the Federal Government regarding standards issued by NIST pursuant to the Federal Property and Administrative Services Act of 1949 as amended, Public Law 89-306 (79 Stat. 1127), and as implemented by Executive Order 11717 (38 FR 12315, dated May 11, 1973) and Part 6 of Title 15 CFR (Code of Federal Regulations).

NIST Interagency or Internal Reports (NISTIR)—The series includes interim or final reports on work performed by NIST for outside sponsors (both government and nongovernment). In general, initial distribution is handled by the sponsor; public distribution is handled by sales through the National Technical Information Service, Springfield, VA 22161, in hard copy, electronic media, or microfiche form. NISTIR's may also report results of NIST projects of transitory or limited interest, including those that will be published subsequently in more comprehensive form.

U.S. Department of Commerce
National Institute of Standards
and Technology
Gaithersburg, MD 20899–0001

Official Business
Penalty for Private Use $300