CHARACTERISTICS OF ADHESIVE-BONDED SEAMS SAMPLED FROM EPDM ROOF MEMBRANES

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This study reports on the characterization of seam samples cut from EPDM roof systems. Data on seam characteristics and failure analysis are needed to further the understanding of seam performance and to develop performance criteria for seams. Forty-eight samples were cut from EPDM roofs whose seams were generally rated as having provided satisfactory or unsatisfactory performance. The samples were subjected to laboratory tests which included identification of the adhesive, measurement of adhesive thickness, and determination of the strength and mode of failure in peel. Comparisons were made between the test results and performance ratings.

It was found that the majority of the unsatisfactorily performing seams had been bonded with neoprene-based adhesives and had been exposed on roofs for 45 months or more when sampled. The limited number of samples with butyl-based adhesives were rated as providing satisfactory performance(or not rated). The butyl- based seams were relatively young, and had peel strengths only slightly greater than those of the satisfactorily performing neoprene-based samples. No relation between performance and either peel strength or adhesive thickness was found. The majority of the samples contained relatively thin adhesive layers (less than 0.005 in. or 0.13 mm). Of the samples subjected to SEM analysis, the majority showed evidence of release agent on the rubber and adhesive surfaces analyzed. This finding provided evidence that a field method to judge rubber surface cleanness before application of the adhesive is needed.

KEYWORDS

Adhesives, analysis, characterization, EPDM, low-sloped roofing, membranes, peel strength, performance, roofs, scanning electron microscopy, seams.

INTRODUCTION

In September 1987, approximately 50 individuals representing a cross segment of the U.S. roofing industry participated in a roundtable meeting to assess the need for research to improve the performance of low-sloped roofs.[1] A major need identified was the determination of the characteristics of the new roofing materials, components and systems as related to in-service exposure. The generic types of materials included elastomers, thermoplastics and polymer-modified bitumens, which composed about 60 percent of the membrane material installed in the United States in 1989.[2]

In spite of the rapid increase in the use of the new membrane systems in the United States since the mid-1970s, relatively few reports have been published concerning in-service performance; but some examples from the U.S. literature may be cited.[3-10] The majority of the field reports on the new membrane materials has centered on ethylene-propylene-diene terpolymer (EPDM). This is not surprising, considering that this rubber comprises the largest share of the new products used in the United States.[2] However, considering that seam performance has been the major concern with this system,[2,11] it is interesting that only limited field data have been published on the characterization of seams sampled from roofs. Reports of failure analyses of problem seams and factors affecting their in-service performance are not available in the technical literature. The availability of data on seam characteristics and failure analysis would be beneficial in furthering the understanding of seam performance and in developing performance criteria for seams.

This paper reports on a study of the characterization of field seam samples cut from low-sloped EPDM roofing systems. Most of the samples were made available to NIST research staff with the assistance of U.S. industry associations, particularly the Roof Consultants Institute (RCI) and the National Roofing Contractors Association (NRCA). In a few instances, roofing practitioners who were not members of these associations sent samples for inclusion in the study. The individuals supplying the samples provided, in most cases, a value judgment as to whether the seams had performed satisfactorily or unsatisfactorily. Satisfactory seams were described as those which were performing well and did not require excessive maintenance or repair. If leaks had occurred, they were generally described as minor, localized, and not continually recurring. In contrast, unsatisfactory seams had required considerable maintenance and repair. These seams had experienced many leaks that had, in some cases, recurred often over a large portion of the roof. Considerable patching of these seams had been carried out. However, samples from these roofs were cut from well-adhered sections of the seams.

SEAM SAMPLES

Table 1 presents, for each of the seam samples, the location of the building, membrane manufacturer, type of membrane attachment, membrane age and the number of specimens. Forty-eight seam samples were examined. These included four patch samples (nos. 10P, 13P, 21P and 30P), and one cover strip sample (no. 19C). The patch samples were taken from roofs where the original seams had been overlaid with a strip of EPDM rubber as a repair procedure. The cover strip was placed over the original seam at the time of its fabrication. Sample 31 was a new roof from which the seams were sampled at three different times. Because the strength of a newly-formed seam increases with time,[12] these samples were given different designations (i.e., 31-1, 31-2 and 31-3).

The dimensions of the samples were about 450 mm X 300 mm (18 in. by 12 in.) with the seam oriented parallel to the long dimension. The width of the seams ranged from about 75 mm to 150 mm (3 in. to 6 in.). In many cases, the NIST research staff was present when the seam samples were taken from the roofs. It was planned to obtain a minimum of three specimens for each sample. However, in some cases, practical constraints precluded cutting three specimens per sample.

Table 2 is a summary of the data set describing the seam samples. As is evident, the samples represented a variety of products from a number of states, and had ages ranging from 1 to 105 months.

LABORATORY TESTS AND OBSERVATIONS

The test procedures used in the study are described in the Appendix. For the bonded seam samples, the adhesive thickness and peel strength were measured, and the mode of failure during peel testing was noted. Three failure modes were apparent: adhesive (interfacial), cohesive (within the bulk of the adhesive) or through small void areas present in the adhesive layer. The surface of the voids was shiny as if little or no contact of the adhesive had occurred in these areas. Although the voids produced a "cohesive-like" failure, they represented a distinct failure mode and were distinguished from cohesive failure. For many samples, the failure during peel testing was a combination of two or three of the failure modes. A rough estimate (± 10 percent) was made of the percent of the seam surface area experiencing each of the failure modes. After peel testing, the surfaces of the delaminated strips were visually examined to assess whether any contamination could be seen on the exposed rubber or adhesive. A number of delaminated strips were examined using scanning electron microscopy (SEM).

All adhesives were subjected to a "Beilstein" flame test and thermogravimetry (TG) for initial identification of the generic type. If the identification was not conclusive (see discussion below), a solubility test and a Fourier Transform Infrared Spectroscopy (FTIR) analysis were conducted. The color of all adhesives was also noted.

RESULTS AND ANALYSIS

Table 3 summarizes the data and observations obtained during testing of the seam samples, and also includes the value judgment provided to NIST researchers concerning the performance of the seam samples. Table 4 presents the results of the tests conducted on the adhesives. Data were recorded in a computer file and analyzed using a graphics program called "DATAPLOT."[13] For each specimen cut from a roof, five 100 mm X 25 mm (4 in. by 1 in.) strips were used in determining adhesive thickness and peel strength. For each sample, the values for these properties (Table 3) are the averages of all determinations (i.e., number of specimens times five). Average values were used for simplicity in reporting and graphically illustrating the results. In the case of the adhesive thickness, the coefficient of variation within any one set of five strips was 40 percent or less. For the peel strength, the coefficient of variation within any one set of five strips was 20 percent or less.

DISCUSSION

In addition to providing data on seams sampled from roofs,the intent of the study was to examine whether any of the characteristics measured showed a relation to performance (i.e., satisfactory or unsatisfactory) assigned to the seams. It should be remembered that the judgments were subjective and dependent on the individual providing the sample. The sample set was quite limited in relation to the amount of EPDM roofing that has been installed in the United States over the last decade. Also, the samples were selected primarily on the basis of opportunity; i.e., someone in practice was willing to allow the cutting of the roofs in question. This precluded using an experimental design for sampling seams on a statistical basis.

The sections that follow present the analysis of the seam characteristics as a function of the assigned performance judgments. Note in Table 3 that about half the samples (including patches) were described as having performed satisfactorily, while the other half was rated as unsatisfactory.

Adhesive Flame Test, TG Analysis and FTIR Analysis

Initial flame and TG tests were conducted on five EPDM seam adhesive products available in the NIST laboratories. These products served as controls to provide base line data for comparison with the results obtained when the tests were conducted on the adhesives in the field samples. The generic polymeric types of adhesive controls were: two neoprene (polychloroprene)-based,* two butyl-based, and one styrene/ethylene-butylene (SEB)-based. These controls represented the major types of generic polymers that have been used in practice to date to formulate adhesives for EPDM membrane seams.

In initiating the study, it was considered that the flame test and TG analysis could be more readily conducted than the FTIR analysis, because the later procedure necessitated having solutions of the adhesives. Thus, the former tests were first performed as a means of identification. When the flame test was performed on the five controls, two distinct colors were seen. The neoprene-based adhesives displayed a bright green flame, whereas the butyl-based and SEB-based adhesives gave an orange flame. In the case of the TG analysis, two distinct shapes of curves were apparent (Figure 1), clearly distinguishing the neoprene-based controls from the butyl-based and SEB-based controls. The latter two types of products showed comparable curves.** The finding that the butyl-based and the SEB-based controls displayed similar TG curves and flame test results meant that additional tests were needed to distinguish between them. Consequently, these

**Neoprene is the common name used in the roofing industry (and others) to describe polychloroprene rubbers, and is the nomenclature used in this report.*

***As a consequence of the TG results on the controls, for simplicity, the thermogravimetry curves of the seam samples were described as "neoprene-like," or "butyl-like."*

controls were subjected to a solubility test in toluene and FTIR analysis for further identification.

The SEB-based control was found to be readily soluble in toluene, while the butyl-based controls were not. Moreover, the FTIR spectra of the two butyl-based controls, which were extremely similar (although not identical), were distinct from that of SEB- based control. Thus, these two tests readily distinguished butyl-based adhesives from SEB-based adhesives. Figure 2 shows the results of the flame and TG tests of the adhesives in the field seams as a function of sample age and performance rating. The flame test showed one of three results: (1) a green flame, typical of neoprene-based controls, (2) an orange flame (that often was not strong), typical of butyl-based or SEB-based controls, and (3) a flame whose color was difficult to interpret because it was primarily orange but included an occasional tinge of green. The latter observation was attributed to the possible presence of trace amounts of a halide component. With regard to the TG analysis, with one exception, the curves were readily categorized as either neoprene-like or butyl-like. In the case of sample no. 18, the TG curve was reasonably similar to a butyl-like product. However, the mass loss on heating in nitrogen was slightly less, while that in air was slightly greater than the mass losses experienced by the butyl-based controls in those two atmospheres.

It is evident from Figure 2 that, whenever the "Beilstein test" gave a green flame, the TG results indicated that the adhesive was a neoprene-based material (and vice versa). On the other hand, when the flame was either orange or difficult to interpret, the TG analysis showed "butyl-like" curves. Thus, about three-quarters of the seam samples were characterized as neoprene-based adhesives.

The 15 samples that displayed butyl-like TG curves were subjected to the solubility test and FTIR analysis. Three (nos. 1, 5 and 6) were readily soluble in toluene and gave FTIR spectra identical to that of the SEB-based control. The other 12 samples were not readily soluble in toluene and gave FTIR spectra identical to those of the butyl-based controls. Included in this group was sample no. 18 whose TG curve was not readily identified as butyl-like.

A conclusion drawn from the above-described analysis of the adhesives was that FTIR was the preferred method for identification. This finding was consistent with classical organic analysis in that IR spectroscopy is used to "fingerprint" compounds. It requires available spectra of known controls for positive identification, but differentiation between unknown compounds can be made positively in the absence of known controls. On a qualitative basis, a bright green flame was indicative of a neoprene adhesive for all samples in the study. A marked advantage of this test is the rapidity with which it can be conducted.

From Figure 2, it may be seen that the samples with unsatisfactory performance ratings were all 45 months older in age. These samples contained either neoprene-based or SEB-based (nos. 1, 5 and 6) adhesives. The observation that the majority of the unsatisfactory samples were relatively old and the adhesives were neoprene-based was consistent with field experiences reported[14-16] for EPDM roofing systems. Seams with neoprene-based adhesives have, in some cases, deteriorated after some years in service.[15] Technical reports describing the problem, its extent, mechanism, and factors contributing to its occurrence have not been published in the literature. One short review[16] on the effect of moisture on roof system performance stated that "neoprene cements that joined individual sheets and were used as flashing cements were sensitive to elevated temperature and water."

The fact that none of the younger seams were described as unsatisfactory cannot be taken as an indicator that seam problems only arise for aged roofs. The ages of the seams given here were those at the time of sampling, and the ages at which problems first arose were not known. Moreover, it should be remembered that the seams rated as satisfactory were not necessarily leak-free. Thus, the study results emphasize the long-term problem associated with neoprene-based adhesives, while not addressing short-term problems that may have caused leaks.

Of the 12 adhesives that were identified as butyl-based, only seven were given a performance rating, but all were described as satisfactory. Samples having a satisfactory rating were generally 60 months old or less, although two neoprene-based samples were 96 and 105 months old.

Color

Figure 3 plots the color of the adhesive versus age and performance rating. The majority of the satisfactorily performing samples were black and relatively young; whereas the majority of the unsatisfactorily-performing samples were yellow-brown and relatively old. However, in spite of these observations, color should not be used as a general performance indicator. First, it is very subjective. Second, the observed relation between color and performance is considered to be an artifact of the change of adhesives over the years by the EPDM roofing industry. In recent years, the adhesives have generally been black and butyl-based, while the older adhesives were yellow and neoprene-based. Many organic materials that are yellow when new turn yellow-brown or a darker color upon deterioration.

Strength Versus Age and Performance

Figure 4 shows the results of the T-peel tests as a function of sample age and performance rating. No relation was found between peel strength and performance rating. An implication of the data in Figure 4 is that relatively high strength is not a requisite for satisfactory performance. Note in Figure 4 that two samples (nos. 9 and 12) had peel strengths of about 0.26 kN/m (1.5 lbf/in.), and they reportedly performed satisfactorily for 8 years or more. Conversely, four samples (nos. 1, 5, 6 and 15) having unsatisfactory performance displayed the greatest strengths.

Relationships between strength and age (Figure 4) were investigated for the satisfactory neoprene-based samples and the satisfactory butyl-based samples separately. This approach was necessary because seams having butyl-based adhesives are generally stronger (when tested in the laboratory) and younger than those with neoprene-based adhesives.[9] For each of these two types of adhesives in the satisfactory-performance group, no significant relation between peel strength and age was found.

In the case of the unsatisfactory samples (Figure 4), a straight line fit to the data showed a statistically significant (5 percent level) negative slope of -0.0098 kN/m (-0.056 lbf/in.). This slope was influenced considerably by the SEB-based adhesives (nos. 1, 5 and 6). If these samples were not included in the analysis, the slope was -0.0067 kN/m(-0.038 lbf/in.). A possible reason for the negative slope is that, in cases where seams experience problems, their peel strengths decrease with age. Another explanation might be that, for the limited data set, the initial strengths of the younger samples were greater than those of the older seams (e.g., maybe more primers were used in recent years). However, this study cannot choose which reason applies, because the initial peel strengths (time zero) of the samples were not known.

When sample no. 5, which had the greatest strength of the sample set, was delaminated, the adhesive layer was found to contain numerous voids (Figure 5). The individual supplying this sample reported that the roof sporadically leaked over the five years the EPDM system was in place. Inspection of the seams at the time of sampling indicated that they were apparently tightly adhered—an observation that was consistent with their measured peel strength. Based on the observations of the delaminated sample, a possible explanation of the sporadic leaking was the formation of continuous channels connecting the voids in the adhesive layer. The finding illustrates the importance of having a continuous and relatively uniform layer of adhesive to reduce the risk of water transmission through pathways that may be created by voids and skips.

Figure 6 presents the peel strength data as related to the generic type of adhesive. Most of the neoprene-based adhesives had peel strengths less than 0.53 kN/m (3.0 lbf/in.), which were typical for these products applied to unprimed EPDM rubber.[9] The neoprene-based adhesive seams with strengths above this value may have been fabricated using a primer.[17] For the neoprene-based samples, the average peel strengths for the satisfactory and unsatisfactory data sets were 0.39 and 0.35 kN/m (2.2 and 2.0 lbf/in.), respectively. The difference was not statistically significant.

The peel strengths of the limited number of butyl-based products ranged from 0.39 to 0.95 kN/m (2.2 to 5.4 lbf/in.), with an average of 0.63 kN/m (3.6 lbf/in.). Although this average value was statistically different (1.3 percent level) from the average strength of the satisfactory neoprene-based samples, it was only about 60 percent greater. The values for peel strength of the butyl-based samples were comparable to those reported for other such seams taken from roofs in service.[9,10,18,19] As has been previously noted,[10,18] peel strength values of the order of the range found in this study are much less than those of seams prepared and cured in the laboratory. In the latter case, the peel strengths have been 1.4 to 1.6 kN/m (8 to 9 lbf/in.) or more.[10,12] Reasons why the peel strength values of field samples have been less than those of laboratory specimens have not been investigated, although a question has been raised as to the role played by moisture, in addition to surface cleanness of the rubber.[18] Butyl-based adhesives cure by a moisture-induced mechanism, and thus, differences in moisture conditions between the lab and the field might result in differences in strength. Another explanation, based on the results of this study, is the presence of micro-cavities (honeycomb structure) in the adhesive layer (see discussion below on SEM analysis). These micro-cavities represent defects in the adhesive layer which could contribute to lower-than-expected strength when peel failure is cohesive (in comparison to that obtained if the micro-cavities were not present).

Strength Versus Thickness and Performance

In a controlled laboratory experiment, Martin et al.[20] have shown that the times-to-failure of EPDM seam specimens, loaded in T-peel in creep-rupture experiments, are significantly lengthened by increased thicknesses of the adhesive layer. Also, Watanabe and Rossiter[21] have shown that the peel strengths increase with an increase of adhesive thickness until a plateau strength value is approached at thicknesses of about 0.63 mm (0.025 in.). Thus, it was of interest to see whether a relation between thickness and strength existed for the field samples.

Figure 7 is a plot of peel strength versus thickness for the samples in the satisfactory and unsatisfactory performance groups. No significant correlation between strength and thickness was found for either group (if sample nos. 1, 5 and 6 are not included in the analysis). This was not surprising considering the diversity of the sample set. In addition to thickness, a variety of parameters such as surface cleanness of the rubber surface, voids in the adhesive layer, and pressure applied during application,[22] as well as exposure conditions,[23] affect peel strength. No control over any of these parameters was exercised during the sampling of the seams in this study.

A noteworthy feature of the thickness measurements was the relative thinness of the majority of the adhesive layers. About 85 percent of the measured thicknesses were 0.2 mm (0.008 in.) or less. This observation may be compared with the laboratory findings of Martin et al.[20] concerning increased resistance of seams to peel failure under creep conditions with an increase in adhesive thickness. The relatively thin adhesive layers found for the field samples implies that these seams may not be as resistant to peel failure in creep, as they possibly could be if they had thicker adhesive layers.

Failure Mode During Peel Testing

Table 3 includes the mode of failure of the samples during peel testing. Analyses were conducted to determine whether relationships between the type of failure during peel testing and the other parameters addressed during the study existed (data not shown).

Noteworthy observations from these analyses are:

- Neoprene-based adhesives peeled predominantly by adhesive failure. This was consistent with previous observations.[22]
- Most, but not all, of the butyl-based samples (which were all classified as having performed satisfactorily) delaminated cohesively. This was in general agreement with laboratory experience that seams prepared with butyl-based adhesive on cleaned EPDM surfaces fail cohesively in peel.[12,22] In the present study, butyl-based sample nos. 7 and 16 showed predominantly adhesive failure during peel testing, indicating the surface effects were playing a role. Note that sample nos. 7 and 16 had strengths of 0.51 and 0.39 kN/m (2.9 and 2.2 lbf/in.), respectively, which were the lowest values for the butyl-based seams.
- In general, when the strength of the seam was 0.44 kN/m (2.5 lbf/in.) or less, the failure mode was predominantly adhesive (with voids in some cases).
- Twenty-nine samples contained some void areas in the adhesive layer. For 14 of these samples (about one-third of the total sample set), the percentage of voids was estimated to be 20 percent or more of the delaminated area. This finding indicated that a significant percent of the area of the two bonded sheets that formed the seam speci-

mens was apparently not in contact. The effects of void areas on performance have not been ascertained, but they could be considered as flaws in the adhesive layers. However, it must be noted that only one sample with a high percent void area (no. 5) had been assigned an unsatisfactory performance rating.

Surface Contamination

After delamination, the exposed surfaces of the seam strips were visually examined for the presence of contaminants such as release agent, dirt, particles of insulation board or similar matter. The majority of the samples were visually seen to be free of such contaminants (Table 3). Nine of the samples showed some evidence of contamination, which generally did not cover the entire surface of the exposed strip. However, only one (no. 6) of the nine was rated as having performed unsatisfactorily.

Scanning Electron Microscopy—Scanning electron microscopy (SEM) was conducted on selected samples to characterize surfaces exposed during peel testing. Table 5 presents a summary of the SEM observations along with some seam characteristics such as type of adhesive, age, performance rating and the primary mode of failure observed during peel testing. The samples subjected to SEM analysis represented a cross-section of the seam set including new and old samples, the three types of adhesives, samples that had performed satisfactorily and unsatisfactorily, and those which showed adhesive and cohesive failure in peel.

Two major observations from the SEM analyses follow:

- Platelet particles typical of release agent were observed in a majority of the cases. Only in the case of sample nos. 1 and 2 where the peel failure was cohesive were no particles observed (the rubber-adhesive interfaces where release agent might be present were not visible). As an example of the effect of the release agent, note sample no. 8 which contained a butyl-based adhesive. This sample failed adhesively with relatively low strength (0.46 KN/m or 2.6 lbf/in.) for a butyl-based adhesive, which was consistent with laboratory data on the decrease in strength due to release-agent contamination of the rubber surface.[10,12]
- Release agent was present on the samples regardless of performance rating. In the case of sample nos. 3 and 4, both were neoprene-based, had about the same age, failed adhesively in peel, and displayed rubber surfaces covered with release agent. On the other hand, no. 3 was given a satisfactory rating; whereas no. 4 was described as unsatisfactory. Similarly, in the case of sample nos. 10 and 10-P, both showed rubber surfaces that were covered with release agent. No. 10 was an unsatisfactory sample, while no. 10-P was a satisfactory sample. Here, the patch sample had only half the age of the original seam.
- The finding that release agent was present on both the satisfactory and unsatisfactory samples implies that the presence of some contamination on the surface does not necessarily lead to failure (at least over the relatively short lives of the samples in question). However, this finding should not be taken to imply that surface cleanness of the rubber sheet is unimportant. Good adhesion practice dictates that surfaces to be bonded be well prepared.[24] Note again sample no. 8 with low strength, adhesive failure, and a contaminated surface.
- From a practical point of view, an implication of the finding of release agent on most of the sample surfaces may be very significant. The limited data here signify that the field practice of cleaning release agent from EPDM surfaces may be less effective, even if done according to prescribed methods, than previously considered. Although limited, the data provide strong evidence that a field method is needed to judge whether release agent and other contaminants are effectively removed from the rubber before adhesive application.
- Four samples (including both neoprene-based and butyl-based adhesives) displayed some micro-cavities (honeycomb structure) in the adhesive layer. The most notable example is given in Figure 8. As previously indicated, these micro-cavities represent defects in the adhesive, which could result in a lowering of the peel strength of the sample (as compared to that which might be observed if the micro-cavities were not present). For example, the peel strength of sample no. 31-3 was 0.44 kN/m (2.5 lbf/in.), which was among the lowest strengths observed for the butyl-based adhesive samples. This sample, which was too young to be assigned a performance rating, failed cohesively and had micro-cavities in the adhesive layer. The presence of micro-cavities in adhesives has not been previously reported, and their effect on long-term seam performance has not been investigated.

SUMMARY AND CONCLUSIONS

This study reported on the characterization of seam samples cut from EPDM roofs. Up to now, only limited data have been published on seam characteristics in spite of the fact that seams have been the main performance concern with many of the newer roofing systems. The data obtained in this study is expected to be beneficial in furthering the understanding of seam performance and in developing performance criteria for seams.

Forty-eight samples were cut from EPDM roofs whose seams were generally described as having provided either satisfactory or unsatisfactory performance. In the laboratory, the samples were subjected to testing which included identification of the adhesive, measurement of adhesive thickness and determination of peel strength and mode of failure during peel testing. In analyzing the data, comparisons to the performance rating were made.

A summary of the key findings follows:

- Methods for identifying the type of adhesive including a flame test, TG analysis, solubility and FTIR analysis were investigated. FTIR was the preferred method, providing positive identification if spectra of known products are available. Qualitatively, the flame test provided an indicator of a neoprene-based adhesive.
- The majority of the unsatisfactorily performing seams had neoprene-based adhesives and were 45 months or more in age. This finding reflected field experiences associated with EPDM roofing systems in that some seams fabricated with neoprene-based adhesives have encountered problems after some years of service. Three other samples with SEB-based adhesive also were described as having unsatisfactory performance. The limited number of seams with butyl-based adhesives were described as providing satisfactory performance.

- No relation between peel strength and performance was found. Relatively high strength was not a requisite for satisfactory performance. The butyl-based samples had peel strengths only slightly greater than those of the satisfactorily (and unsatisfactory) performing neoprene-based samples.
- No relation was found between adhesive thickness and performance for either the neoprene-based or butyl-based samples. The majority of the samples contained relatively thin adhesive layers which, based on previous laboratory tests, implied that they were not as resistant to peel failure under creep conditions as they could be, if the adhesive layers were thicker.
- The vast majority of the limited number of samples subjected to SEM analysis showed evidence of release agent on the rubber and adhesive surfaces analyzed regardless of type of adhesive or performance rating. This finding provided evidence that a field method to judge rubber surface cleanness before application of the adhesive is needed.
- Four samples subjected to SEM analysis showed micro-cavities in the adhesive layer. These observations raised a question regarding the extent to which micro-cavities affect measured strength of the adhesive. Effects of micro-cavities on long-term seam performance have not been investigated.

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Sample No.	Number of Specimens	Building Location	Membrane Manufact.	Type of Attachment	Membrane Age, mos
1	2	Iowa	3	Adhered	66
2	3	Iowa	2	Adhered	27
3	3	Iowa	1	Adhered	59
4	3	Iowa	unk[a]	Adhered	53
5	3	Iowa	3	Adhered	60
6	2	Iowa	3	Ballasted	60
7	2	Iowa	4	Ballasted	12
8	2	Iowa	4	Ballasted	1
9	3	Iowa	1	Ballasted	105
10	3	Iowa	2	Adhered	72
10-P	3	Iowa	2	Adhered	36
11	3	Iowa	2	Adhered	15
12	3	Iowa	1	Ballasted	96
13	3	Iowa	2	Adhered	72
13-P	3	Iowa	2	Adhered	24
14	3	Iowa	2	Adhered	15
15	1	Iowa	6	Adhered	50
16	2	Iowa	7	Adhered	30
17	3	Virginia	6	Ballasted	58
18	3	Virginia	2	Adhered	2
19	3	Virginia	6	Mech. Fast.	58
19-C	2	Virginia	6	Mech. Fast.	58
20	3	Virginia	2	Adhered	48
21	2	New Jersey	1	Mech. Fast.	88
21-P	2	New Jersey	1	Mech. Fast.	52
22	2	New Jersey	1	Mech. Fast.	100
23	2	New Jersey	1	Mech. Fast.	52
24	2	New Jersey	1	Mech. Fast.	64
25	2	New Jersey	1	Mech. Fast.	52
26	2	New Jersey	1	Mech. Fast.	52
27	2	New Jersey	1	Mech. Fast.	64
28	2	New Jersey	1	Mech. Fast.	76
29	1	New Jersey	1	Mech. Fast.	100
30	6	New Jersey	1	Adhered	88
30-P	6	New Jersey	1	Adhered	40
31-1	1	Virginia	2	Ballasted	2
31-2	1	Virginia	2	Ballasted	1
31-3	1	Virginia	2	Ballasted	1
32	2	Illinois	4	Ballasted	53
33	1	Texas	6	unknown	64
34	3	Pennsylvania	1	Ballasted	2
35	1	Missouri	unk	Adhered	48
36	2	Virginia	5	Ballasted	9
37	3	Virginia	1	Adhered	60
38	8	Maryland	4	Mech. Fast.	60
39	1	Virginia	1	Prot. Memb.	60
40	2	Virginia	unk	Adhered	unk
41	5	Virginia	unk	Adhered	48

[a]Unk indicates unknown.

Table 1 Seam data set.

No. of Samples:	48 including four patch samples and one cover strip sample.
No. of Locations:	8 states
No. of Manufacturers:	7
Types of Attachments:	4 (adhered, mechanically fastened, ballasted, and one protected membrane roof)
Range of Membrane Ages:	1 to 105 Months
Performance Judgment:	20 samples with satisfactory performance 19 samples with unsatisfactory performance 1 sample with mixed performance 3 samples too young to judge 2 samples not rated

Table 2 Summary of the seam-sample data base.

Sample No.	Adhesive Thickness		Peel Strength		Peel Mode Adh Coh Void percent			Surface[a]	Perform Judgment[b]
	mm	mil	kN/m	lbf/in	Adh	Coh	Void		
1	0.15	6	0.96	5.5	2	98	0	1	2
2	0.23	9	0.54	3.1	10	90	0	1,3	1
3	0.13	5	0.42	2.4	83	0	17	2	1
4	0.18	7	0.60	3.4	100	0	0	2	2
5	0.43	17	1.56	8.9	1	63	34	1	2
6	0.33	13	1.26	7.2	96	0	4	2,3	2
7	0.10	4	0.51	2.9	70	0	30	2,3	1
8	0.10	4	0.46	2.6	94	0	6	4	4
9	0.18	7	0.25	1.4	100	0	0	4	1
10	0.25	10	0.19	1.1	98	0	2	2	2
10-P	0.05	2	0.23	1.3	97	0	3	2,3	1
11	0.33	13	0.60	3.4	1	68	31	1	1
12	0.20	8	0.26	1.5	97	0	3	2,3	1
13	—	—	0.18	1.0	87	11	2	2	2
13-P	0.10	4	0.95	5.4	53	27	20	1,2,3	1
14	0.13	5	0.10	3.9	15	35	50	1,2	1
15	0.13	5	1.09	6.2	100	0	0	2	2
16	0.10	4	0.39	2.2	78	0	22	2	1
17	0.13	5	0.86	4.9	80	0	20	2	1
18	0.23	9	0.58	3.3	65	0	35	2	4
19	—	—	0.53	3.0	71	0	29	2	1
19-C	—	—	0.35	2.0	54	0	46	2	1
20	0.15	6	0.44	2.5	90	0	10	2,3	1
21	0.08	3	0.16	0.9	94	0	6	2	2
21-P	0.08	3	0.33	1.9	54	0	46	2	1
22	0.10	4	0.16	0.9	100	0	0	2	2
23	0.08	3	0.60	1.7	85	0	15	2	2
24	0.13	5	0.23	1.3	100	0	0	2	2
25	0.13	5	0.35	2.0	100	0	0	2	2
26	0.08	3	0.21	1.2	100	0	0	2	2
27	0.10	4	0.39	2.2	100	0	0	2	2
28	0.08	3	0.40	2.3	84	0	16	2	2
29	0.08	3	0.19	1.1	100	0	0	2	2
30	0.13	5	0.25	1.4	99	0	1	2	2
30-P	0.10	4	0.28	1.6	100	0	0	2,3	1
31-1	0.13	5	0.47	2.7	0	50	50	1	4
31-2	0.23	9	0.61	3.5	88	6	6	4	4
31-3	0.18	7	0.44	2.5	6	44	50	4	4
32	0.15	6	0.37	2.1	100	0	0	2	1
33	0.08	3	0.32	1.8	87	0	13	2,3	—
34	0.13	5	0.49	2.8	0	82	18	1	4
35	0.18	7	0.28	1.6	100	0	0	2	2
36	0.20	8	0.77	4.4	45	45	10	1,2	1
37	0.20	8	0.14	0.8	100	0	0	2	1
38	0.20	8	0.21	1.2	100	0	0	2	3
39	0.13	5	0.63	3.6	58	42	0	1,2	2
40	0.10	4	0.53	3.0	20	80	0	1,2	—
41	0.08	3	0.72	4.1	34	66	0	1,2	1

[a]Visual characterization of the surfaces after peel test: 1 = no obvious contamination on the adhesive; 2 = no obvious contamination on the rubber; 3 = some contamination noted; 4 = difficult to judge, maybe something present.
[b]Performance judgment: 1 = satisfactory; 2 = unsatisfactory; 3 = mixed; 4 = not applicable (relatively new seam).

Table 3 Seam characteristics.

Sample No.	Flame Color	TG Results Type	Solubility[a] Soluble	FTIR Results[a] Typical of	Adhesive Color
1	DTI[b]	Butyl-like	Yes	SEB control	Yellow-brown
2	DTI	Butyl-like	No	Butyl control	Black
3	Green	Neoprene-like	—	—	Black
4	Green	Neoprene-like	—	—	Yellow-brown
5	Orange	Butyl-like	Yes	SEB control	Yellow
6	Orange	Butyl-like	Yes	SEB control	Yellow-brown
7	Orange	Butyl-like	No	Butyl control	Black
8	DTI	Butyl-like	No	Butyl control	Black
9	Green	Neoprene-like	—	—	Yellow
10	Green	Neoprene-like	—	—	Yellow-brown
10-P	Green	Neoprene-like	—	—	Black
11	DTI	Butyl-like	No	Butyl control	Black
12	Green	neoprene-like	—	—	Black
13	Green	Neoprene-like	—	—	Yellow-brown
13-P	DTI	Butyl-like	No	Butyl control	Black
14	DTI	Butyl-like	No	Butyl control	Black
15	Green	Neoprene-like	—	—	Yellow
16	Orange	Butyl-like	No	Butyl control	Black
17	Green	Neoprene-like	—	—	Yellow
18	Orange	DTI	No	butyl control	Black
19	Green	Neoprene-like	—	—	Black
19-C	Green	Neoprene-like	—	—	Black
20	Green	Neoprene-like	—	—	Yellow
21	Green	Neoprene-like	—	—	Yellow
21-P	Green	Neoprene-like	—	—	Yellow
22	Green	Neoprene-like	—	—	Yellow-brown
23	Green	Neoprene-like	—	—	Yellow-brown
24	Green	Neoprene-like	—	—	Yellow-brown
25	Green	Neoprene-like	—	—	Yellow-brown
26	Green	Neoprene-like	—	—	Yellow-brown
27	Green	Neoprene-like	—	—	Yellow-brown
28	Green	Neoprene-like	—	—	Yellow-brown
29	Green	Neoprene-like	—	—	Yellow-brown
30	Green	Neoprene-like	—	—	Yellow-brown
30-p	Green	Neoprene-like	—	—	Yellow-brown
31-1	Orange	Butyl-like	No	Butyl control	Black
31-2	Orange	Butyl-like	No	Butyl control	Black
31-3	Orange	Butyl-like	No	Butyl control	Black
32	Green	Neoprene-like	—	—	Black
33	Green	Neoprene-like	—	—	Black
34	Orange	Butyl-like	—	—	Black
35	Green	Neoprene-like	—	—	Black
36	Orange	Butyl-like	No	Butyl control	Black
37	Green	Neoprene-like	—	—	Yellow
38	Green	Neoprene-like	—	—	Yellow-brown
39	Green	Neoprene-like	—	—	Black
40	Green	Neoprene-like	—	—	Yellow-brown
41	Green	Neoprene-like	—	—	Yellow-brown

[a]These tests were only conducted on adhesive samples that displayed TG curves typical of butyl-based product.
[b]DTI indicates "difficult to interpret."

***Table 4** Adhesive characteristics.*

Sample No.	Adhesive Type[a]	Age mos	Perform. Rating[b]	Peel Mode[c]	SEM Observations
1	S	66	2	Coh	Observed the adhesive surface: no release agent or other contamination present; some honeycomb structure was seen in the adhesive layer.
2	B	27	1	Coh	Observed the adhesive surface: no contamination present, adhesive surface was smooth with some particles mixed in the layer; some honeycomb structure was observed. Observed a small section of the rubber surface where failure was adhesive: platelet particles typical of release agent were present on the rubber surface.
3	N	59	1	Adh	Observed both the rubber and adhesive surfaces: both were covered with platelet particles typical of release agent.
4	N	53	2	Adh	Observed the rubber surface: it was covered with platelet particles typical of release agent.
8	B	1	4	Adh	Observed the rubber surface: it was covered with platelet particles typical of release agent.
10	N	72	2	Adh	Observed a section of the sample where rubber and adhesive surfaces could be observed together: they were both covered with platelet particles typical of release agent; some honeycomb structure was seen in the adhesive layer (Figure 8).
10-P	N	36	1	Adh	Observed the rubber surface: it was covered with platelet particles typical of release agent.
31-3	B	1	4	Coh	Observed the adhesive surface near the rubber sheet: platelet particles typical of release were present; some honeycomb structure was seen in the adhesive layer.

[a]This refers to the type of adhesive as identified in the present study: S = comparable to the SEB-based control; B = comparable to the butyl-based control; N = comparable to the neoprene-based control.
[b]This refers to the assigned performance rating: 1 = satisfactory; 2 = unsatisfactory; 4 = not applicable (relatively new seam).
[c]This refers to the primary failure mode observed during peel testing of the samples: Adh = adhesive failure; Coh = cohesive failure.

Table 5 *Summary of the SEM observations.*

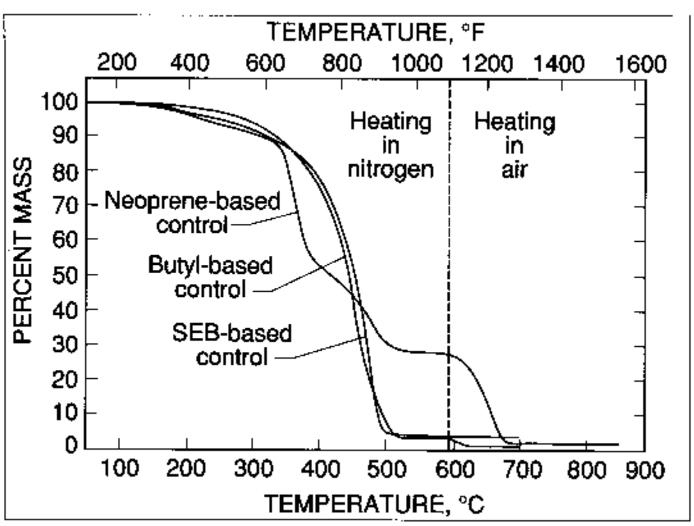

Figure 1 *Thermogravimetric analysis of the control adhesives.*

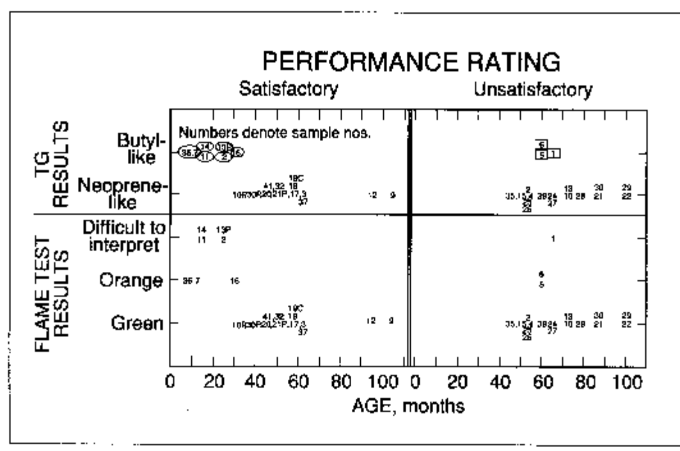

Figure 2 *Results of the flame tests and thermogravimetric analyses as related to age and performance rating. (The sample nos. in circles and boxes denote butyl-based and SEB-based adhesives, respectively. The remainder are neoprene-based.)*

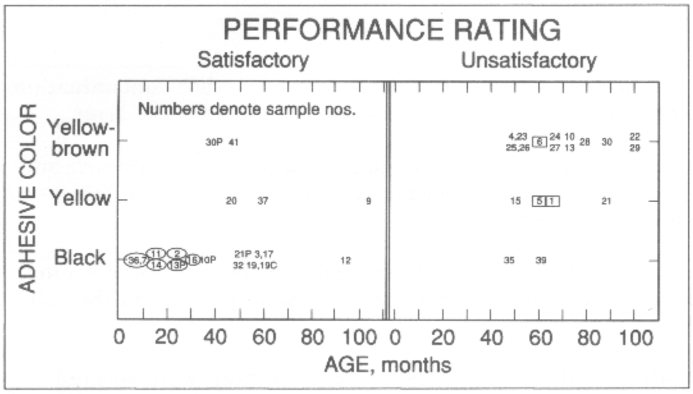

Figure 3 *Color of the adhesives versus age and performance rating. (The sample nos. in circles and boxes denote butyl-based and SEB-based adhesives, respectively. The remainder are neoprene-based.)*

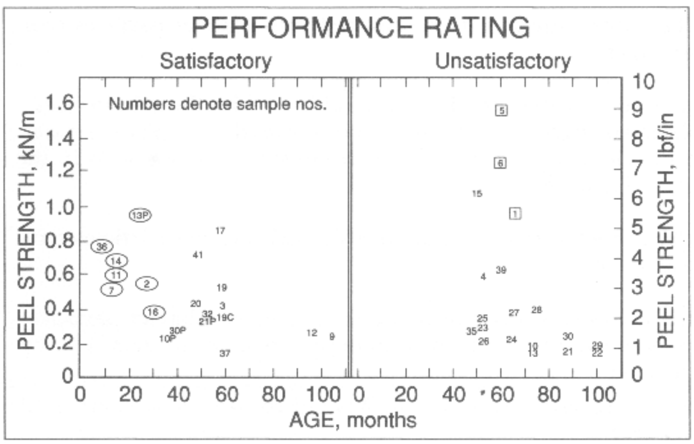

Figure 4 *Peel strength versus age and performance rating. (The sample nos. in circles and boxes denote butyl-based and SEB-based adhesives, respectively. The remainder are neoprene-based.)*

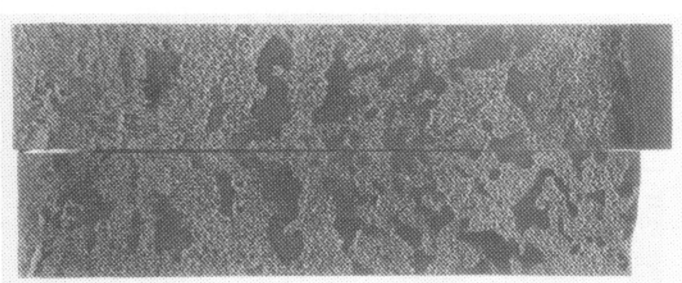

Figure 5 *Photo of the adhesive layer exposed on delamination of sample no. 5 showing its non-uniformity.*

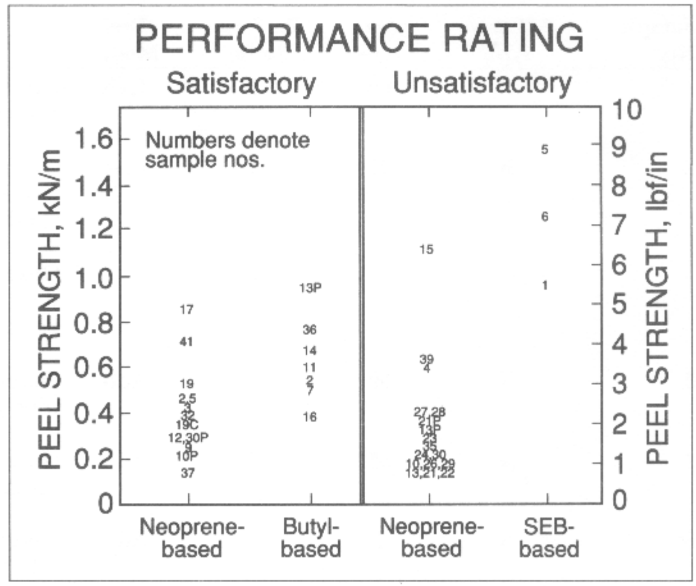

Figure 6 *Peel strength versus adhesive type and performance rating.*

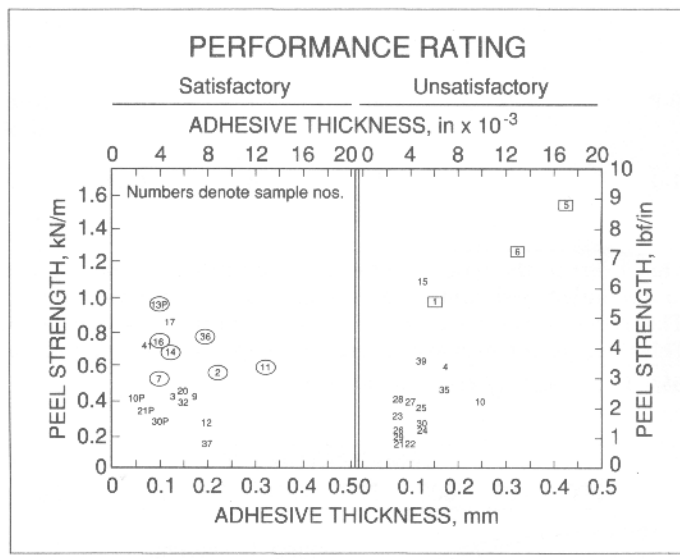

Figure 7 *Peel strength versus adhesive thickness and performance rating. (The sample nos. in circles and boxes denote butyl-based and SEB-based adhesives, respectively. The remainder are neoprene-based.)*

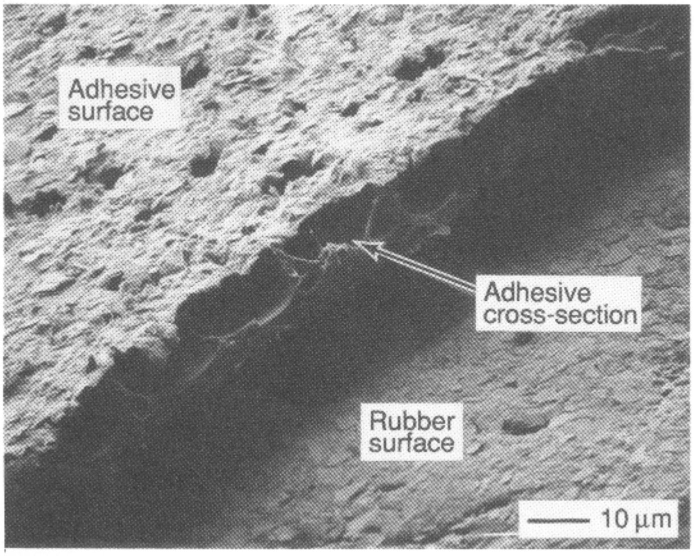

Figure 8 *SEM photomicrographs of a delaminated section of sample no. 10 showing micro-voids in the adhesive layer.*

APPENDIX

Experimental Procedures

A.1 T-Peel Tests—T-peel tests were conducted according to the procedure described in ASTM D 1876, "Standard Test Method for Peel Resistance of Adhesives (T-Peel Test),"[A1] except that the load was applied at a constant rate of 50 mm/min. (2 in./min.). The length of the bond delaminated was approximately 100 mm (4 in.). The testing machine was equipped with a microcomputer which was used to calculate the average peel force per unit specimen width.

A.2 Adhesive Thickness—The adhesive thickness was estimated for each sample as follows. Before peel testing, the thickness of the specimen was measured at two locations (about 25 mm or 1 in. from each end) using calipers sensitive to 0.0025 mm (0.0001 in.). The thickness of the rubber sheet comprising the seam was determined at four locations also using calipers. The adhesive thickness was the difference between the average thickness of the specimen and that of the rubber sheet, and estimated to be ± 0.05 mm (± 0.002 in.).

A.3 Beilstein Flame Test of the Adhesives—The "Beilstein Test" is a classical qualitative analysis procedure for the identification of halide-containing organic compounds.[A2] A small sample of the adhesive compound, scraped from the surface of the EPDM sheet after delamination of the seam, is burnt on a piece of copper using a laboratory gas flame. If a halide is present, a green flame results.

A.4 Thermogravimetry—Adhesive samples (5 to 15 mg) were heated from 50°C to about 700°C (122°F to 1292°F) at a rate of 20°C (36°F) per minute. Pyrolysis was conducted in nitrogen gas at a flow rate of 40 mL/min. until 600°C (1112°F) was reached. Air was then introduced at the same flow rate to combust the residual material. When consecutive tests were conducted, the instrument was allowed to cool to 50°C (122°F) and then purged with nitrogen for about 5 minutes prior to the next run.

A.5 Solubility in Toluene—Adhesive samples that displayed thermogravimetry curves typical of a butyl-based product were subjected to a qualitative but relatively rapid solubility test. The adhesive (6 to 10 mg) was placed in a test tube and toluene (2 mL) was added. The test tube was sealed with a cork and gently shaken by hand. It was allowed to sit overnight (16-18 hours) at ambient laboratory conditions. Then the test tube was examined by eye to see whether the adhesive, for the most part, dissolved.

A.6 Fourier Transform Infrared Spectroscopy (FTIR)—Adhesive samples which gave thermogravimetry curves typical of a butyl-based product were subjected to FTIR analysis. The adhesive (about 50 mg) was placed in a test tube to which toluene (2 mL) was added. The test tube was sealed with a cork and gently shaken by hand. It was placed in a water bath at about 65°C (149°F) for 3-4 hours over which time it was occasionally shaken by hand. Not all the adhesive always dissolved, but sufficient amounts went into solution to coat a film of the adhesive on NaCl crystals. FTIR transmission spectra were obtained using the coated NaCl crystals.

A.7 Scanning Electron Microcopy (SEM) Analysis—The specimens for SEM analysis were cut from delaminated seam samples into squares having about 8 mm to 10 mm (0.3 in. to 0.4 in.) sides. The cut pieces were adhered to SEM specimen mounting stubs with an epoxy adhesive. The mounted specimens were sputter coated with a nominal 20 nm (8 in. X 10^{-7} in.) gold conductive film to prevent surface electron charging during SEM analysis. The surfaces were examined in the SEM using an acceleration voltage of 30 kV at magnifications from X20 to X1000. Photographs were generally taken at X100 and X500 magnifications.

APPENDIX REFERENCES

A1 ASTM D 1876, "Standard Test Method for Peel Resistance of Adhesives (T-Peel Test), "Annual Book of ASTM Standards, Vol. 15.06, American Society for Testing and Materials, Philadelphia, Pa., 1983.

A2 Shriner, R.L., Fusion, R.C. and Curtain, D.Y., The Systematic Identification of Organic Compounds: A Laboratory Manual, 4th Edition, John Wiley and Sons, New York, N.Y., p. 60, 1956.