FIELD TESTING OF ADHESIVE-BONDED SEAMS OF RUBBER ROOFING MEMBRANES

WALTER J. ROSSITER JR., JAMES F. SEILER JR. and PAUL E. STUTZMAN
National Institute of Standards and Technology
(formerly National Bureau of Standards)
Gaithersburg, Md.

ABSTRACT

Laboratory and field investigations were conducted to obtain data for supporting the development of a methodology to assure the quality of adhesive-bonded seams of vulcanized rubber membranes. The prime factors investigated were surface condition of the rubber, temperature of the rubber at the time of adhesive application and cure time of the adhesive. The laboratory data indicated that the T-peel test is sufficiently sensitive for use in a field methodology for assuring the quality of seams. Based on the laboratory results, it appeared possible to conduct the T-peel tests within a day (or perhaps less) of seam formation and to find significant differences in bond strength due to lack of proper surface cleaning of the rubber. Data from field applications are needed to demonstrate whether such differences are found on the job at one day's time or less. Seams sampled from two buildings were found to have low bond strengths, as compared to seams prepared using rubber carefully cleaned in the laboratory. These field specimens were also observed to be contaminated with talc-like particles on the rubber surface. It was considered that the talc-like particles on the surface contributed to the measured low strength of the seams.

Key words: adhesion, contamination, EPDM, field performance, microscopy, peel strength, quality assurance, quality control, roofing, seams, single-ply, vulcanized rubber.

INTRODUCTION

Recent estimates indicate that low-sloped roof systems having vulcanized EPDM membranes account for about one-third of the current market.[1,2] This corresponds to a billion square feet (90 million square meters) or more of EPDM membrane material installed annually. Although the performance of these systems has been generally satisfactory, their use has not been problem free.[3,4]

A critical factor affecting the performance of vulcanized EPDM rubber membranes is the integrity of adhesive-bonded seams.[5,8] EPDM is a non-polar, relatively inert rubber, which makes the formation of field seams difficult. Information from the National Roofing Contractors Association's (NRCA) Project Pinpoint shows that defective seams are the most often reported single-ply problem.[3] Moreover, it has been so since 1984 when the NRCA survey provided an early report on single-ply defects.[9] Recent articles in roofing trade publications have described specific instances of unsatisfactory seam performance and urged that steps be taken to provide the industry with improved means to assure the quality of the field-installed seams.[10-12] For example, Dregger and Ellingson stated that "an element as critical as field lap seams of EPDM membranes has been left with virtually no control or quality assurance. . . ."[10] Such statements support the Project Pinpoint results and provide evidence for the need for a quality assurance technique for assessing the quality of field fabricated seams.

To assist in developing such a technique, studies are underway at the National Institute of Standards and Technology (NIST) (formerly, the National Bureau of Standards, or NBS) to investigate both the long-term performance of seams[13,14] and their quality assurance during fabrication as influenced by various application parameters.[15-17] A methodology, requiring the meeting of two criteria, was proposed to evaluate the quality of newly formed seams.[17] The criteria are:

- First, the bonds prepared in the field should achieve a benchmark (minimum) value of strength, which has been recommended as being attainable under the given environmental conditions during application. (The basis suggested for the measurement was a T-peel test using a portable device and conducted relatively soon, perhaps a day or less, after seam formation.)
- Second, the seams prepared in the field should be totally adhered along their entire expanse, and not contain voids (to the extent practical) in the adhesive layer or delaminations between the adhered sheets. (The basis suggested for the determination was a pulse-echo scan of the newly formed seams.)

Although studies conducted to date have indicated that the proposed methodology appears feasible, further data are needed for supporting its development. For example, in the case of measuring bond strength shortly after seam formation (the first criterion), data must conclusively indicate that properly fabricated seams have significantly greater bond strengths (as well as longer service lives) than those improperly prepared. In particular, improper surface preparation of the rubber has been considered to be a major factor contributing to less-than-satisfactory seam performance.[6,7]

The present paper provides data from one laboratory study and two field investigations that should be considered in the development of the bond-strength criterion of the proposed methodology. The field investigations also included peel testing of laboratory-prepared seams as controls for comparison with the results of tests of seams sampled from the roofs. (General details of specimen preparation, testing and analysis are given in the Appendix.)

CASE I: THE EFFECT OF SURFACE CONDITION AND TIME ON STRENGTH

Ideally, as a field technique, measurements of initial bond strength should be made as soon as possible after seam formation. Many roofing practitioners consider it impractical to conduct quality assurance tests where the results are not available for several days.

In this case, a laboratory experiment was conducted to investigate the following effects on the T-peel strength of seams: (1) rubber surface condition (i.e., cleaned versus uncleaned), (2) rubber temperature during adhesive application and (3) cure time (i.e., time elapsed after bond formation and before testing). These three variables were considered important in that they could adversely affect seam formation and seam bond strength. It was, therefore, of interest to determine whether differences in bond strength due to these variables could be detected within a short time after seam formation.

Figure 1 presents the results of T-peel tests conducted over time on seams prepared using a typical commercial EPDM sheet (0.060 inch or 1.5 millimeter) and the butyl rubber-based contact adhesive available from the sheet manufacturer. Five seam specimens were tested for each data point in Figure 1. The coefficients of variation for strength ranged from 1 to 19 percent. The sheet was received with a heavy dusting of a release agent on its surface. In fabricating the seam specimens, the rubber was either cleaned (using the proprietary wash solvent available from the sheet manufacturer) or used uncleaned ("as-received"). The temperatures of the rubber during adhesive application were 32, 73 and 158 F (0, 23 and 70 C), which spanned a range over which adhesives may be applied in practice.[1] After formation, the seams were cured at room temperature and 50 ± 5 percent relative humidity.

The results in Figure 1 indicate that, depending on application temperature, two to six hours after seam formation the T-peel strengths of the cleaned rubber specimens were greater than those of the uncleaned specimens. At 24 hours and beyond, this difference was marked, with the cleaned specimens having strengths about two times as great as those of the uncleaned specimens. Using a two-way analysis of variance technique,[18] the differences in average bond strength between the sets of cleaned and uncleaned specimens were shown to be significant at the 1 percent level for cure times of 2, 4 and 6 hours (and beyond) at application temperatures of 32, 73 and 158 F (0, 23 and 70 C), respectively.

The finding that the uncleaned rubber surface produced seams having relatively low strength was consistent with the results of other NIST studies.[14,16] For example, Martin, et al.,[14] demonstrated a relationship between decreases in bond strength and the amount of release agent deposited on a cleaned rubber surface. Most importantly, the data in the present study indicate that the T-peel test is a sensitive technique appropriate for use in a field methodology for quality assurance of seams. Based on the laboratory results, it appears possible to conduct the T-peel tests within 24 hours (or perhaps less) of seam formation and to find significant differences in bond strength due to lack of proper surface cleaning of the rubber. Nevertheless, further data from field applications are needed to demonstrate whether such differences are found on the job at one day's time or less.

It is evident from Figure 1 that, when the surface condition was constant (i.e., cleaned or uncleaned), the temperature of the rubber at the time of adhesive application had little effect on bond strength. After adhesive application, the temperatures of all seam specimens while curing were about the same (i.e., room temperature). Thus, significant differences in bond strength due to application temperature may not have been expected. The lack of an effect due to the temperature of the rubber at the time of adhesive application may provide some simplification of a quality assurance methodology. That is, the application temperature may not need to be taken into consideration, provided the cure temperature is kept relatively constant.

The observation that no effect of cleaning the specimens was found over the first two to four hours was attributed to the nature of the butyl-based adhesive, which cured (i.e., chemically cross-linked) over time.[19] When first applied, this adhesive had relatively weak cohesive strength, which increased in time due to curing. Thus, when a seam specimen was tested shortly after formation (up to about four hours), specimen failure was generally cohesive, and consequently, the effect of rubber surface condition (cleanness) was not a factor. After the adhesive gained sufficient cohesive strength, the effect of surface cleanness became evident. For the cleaned specimens, the failure mode remained primarily cohesive (i.e., the bond to rubber was greater than the cohesive strength of the adhesive). In contrast, for the uncleaned specimens, the failure mode switched to predominantly adhesive (i.e., the cohesive strength of the adhesive was greater than the bond strength to the rubber).

The finding that, at early cure times, the cleaned and uncleaned specimens had comparable strengths was unfortunate with regard to the methodology development. It implied that some period of time, perhaps a minimum of six hours, had to elapse before the T-peel was sensitive enough to detect the effect of surface contamination. Perhaps heating of the specimens would accelerate the cure and shorten the time. Investigations of such effects were beyond the scope of the present study.

Scanning electron microscopy (SEM) analysis was conducted to assess the efficiency of the cleaning technique used to remove the release agent on the rubber surface. Figure 2 presents photomicrographs of the cleaned and uncleaned specimens. The micrographs are similar to those published by Westley.[6] The effect of the surface cleaning is clearly noticeable. The uncleaned specimen (Figure 2A) shows a surface covered with flakes or platelets, which are the particles of the release agent (in this case, talc-like) on the manufactured sheet. The cleaned specimen (Figure 2B) displays a rough surface marked with crater-like depressions, which are the surface irregularities of the EPDM sheet. Particles of the release agent are not present. This observation indicated that the cleaning technique removed most of the release agent.

CASE II: OBSERVATIONS FROM A PREMATURE SEAM DELAMINATION

Seam delaminations were occurring in a 14-month-old adhered EPDM roof system located in suburban Washington, D.C.[2] [20] Before repairs were made, NIST research staff obtained samples for measurements of bond strength and analysis of the condition of the rubber surface from which the seams were fabricated. The study involved two major activities:

1. cutting seam samples and noting observations of their condition during sampling, conducting T-peel tests of the field seam samples, and microscopy analysis of the rubber surfaces of some of the delaminated seam samples; and
2. preparing laboratory specimens for T-peel testing to provide points of reference for expected strengths of the field seams.

This EPDM sheet (0.060 inch or 1.5 millimeters thick), relative to that previously described (Case I), showed only a light dusting of a release agent on its surface. The generic type of adhesive, neoprene-based or butyl-based, was not described in the construction records. They only indicated that a "new" adhesive would be used, which implied that it was butyl. Subsequent testing of the adhesive (field and laboratory samples) using the Beilstein test[3][21] showed that it was not neoprene (chloroprene), and thus probably butyl.

Test samples of the seams were taken from five dispersed sections of the roof. Before cutting the membrane, it was decided to take both good and bad samples from each of the five sections.[4,5] Good samples were designated as those having no visible delamination of the lap; whereas bad samples were characterized as having some delamination along the seam edge. Delamination of the bad seams across their entire width had not occurred so that peel specimens could be obtained.

During sampling, observations concerning the condition of the seam specimens were noted. A major finding was the presence of small voids (estimated 1 to 2 millimeters) in the adhesive layer. The voids were readily seen when many of the cut seam specimens were viewed on edge. Reasons for the presence of the voids were not ascertained. One suggestion may be that small air pockets were entrapped between the individual sheets mated to form the seam. Another suggestion was that the adhesive solvent had not totally evaporated from the contact adhesive before formation of the seams. Then, after mating of the seam sheets, the residual solvent volatilized to create the voids. A third suggestion made by a reviewer of this paper was that the adhesive was applied too thin in the area of the voids with the result that contact was not made between the top and bottom layers of the adhesive during bond formation.

The T-peel strengths of the seam specimens, taken from the roof, were determined at room temperature, 70 to 72 F, (21 to 22 C). The results are given in Table 1. The data for each set of good and bad samples, as well as the pooled data for all specimens, were compared at the 0.05 significance level using the statistical t-test technique.[18] The average values of the peel strength were only statistically significant for the good and bad specimens of Sample Set 1 (Table 1). The other sets (numbers 2 to 5) showed no significant difference and no trends. In the case of Sample Sets 1, 2 and 4, the bad specimens had an average strength less than that of the good specimens. Sample Sets 3 and 5 were the opposite, with the bad specimens having greater average strengths.

The average peel strengths for all good and bad specimens were 2.4 and 1.9 foot pounds per inch (0.42 and 0.33 kN/m), respectively (Table 1). This difference was statistically significant at a significance level of 0.05, even though four of the individual sets were not significantly different. This is an example of pooled data being more sensitive to differences because of the larger number of points involved in the comparison. No practical significance is assigned to the difference in strength of the pooled good and bad samples at this time. The values in both cases were low in comparison to those expected for butyl adhesive applied to cleaned rubber surfaces.

After delamination of the field specimens, each was examined visually in the laboratory for evidence of release agent or other factors that may affect bond strength. Two key observations were noted. First, the majority of the specimens showed little or no release agent or contamination as seen by eye. (Some preliminary scanning electron microscopy was performed, and is discussed below.) Six specimens were seen to have some contamination, as evidenced by a brownish oil-like film on the surface of the rubber. The cause was not ascertained, and the oil-like material was not identified.

The second key observation was the presence of small void areas in the adhesive layer. These void areas were numerous and, in part, consistent with the observations made in the field that the specimens had voids in the adhesive layer. The void areas were characterized as having adhesive on both sheets of rubber comprising the seam. The surface of the voids was shiny, as if little or no contact of the adhesive had occurred in these areas. In the void areas, the peel failure of the seam during testing was seen to be "cohesive-like," proceeding by delamination through the voids in the adhesive and not by peeling of that section of the adhesive from the surface of the rubber. It was considered that the void areas contributed, in part, to the low bond strength of the samples, because they apparently represented areas of little or no bond. Figure 3 illustrates the voids, showing a strip of EPDM from the inside of a delaminated seam specimen. Most of the strip contains no adhesive on its surface, because the adhesive in the specimen peeled adhesively and remained on the second strip comprising the seam. Two spots show shiny areas of adhesive that failed in a "cohesive-like" manner through voids. One spot shows adhesive that peeled from the second strip.

Seam specimens were prepared in the laboratory for testing according to the conditions (cleanness, cure time and cure temperature) given in Table 2. The surfaces of the cleaned specimens were prepared using the wash solution commercially available for this EPDM/adhesive system.

Figure 4 includes the results of the T-peel tests for the laboratory specimens. Six replicate specimens were tested for each application condition under which the seams were prepared, and gave coefficients of variation ranging from 4 to 7 percent. Examination of the delaminated specimens showed an absence of void areas (adhesive with shiny surface) of the type found for the field specimens. The vast majority of the specimens, even those cleaned, failed adhesively, indicating that the weak link in the seam was at the interface of the adhesive and rubber sheet. This observation for the cleaned specimens was in marked contrast to those in Case I, where the cleaned specimens primarily failed (after 4 to 6 hours) cohesively.

The maximum average T-peel strength was achieved for a cleaned specimen, cured for 14 days at 73 F (23 C). This value of 5.7 foot pounds per inch (1.0 kN/m) provided an indication of the strength that might be expected of seams prepared from this rubber/adhesive system, which included a wash solution, under some acceptable conditions of formation. Longer cure times may have produced stronger bonds, but cure time was not further investigated in the present study. The average maximum value for the cleaned specimens after 14 days at 73 F (23 C) was more than twice the average value of 2.2 foot pounds per inch (0.39 kN/m) found for all field specimens. The value of 5.7 foot pounds per inch (1.0 kN/m) suggests that, consistent with the Beilstein test results, the adhesive was not neoprene based. Neoprene adhesives for EPDM membranes generally achieved T-peel strengths of about 2 foot pounds per inch (0.4 kN/m).[5]

The effect of the three application variables (Table 2) on seam strength of the laboratory specimens is evident from Figure 4. For every comparable instance of cure time and temperature, the uncleaned specimens displayed lower bond strengths than the cleaned specimens. The reduction in strength was of the order of 40 percent.

It was also evident that, for three of the four sets of cure temperature and surface condition, as the cure time increased from 7 to 14 days, the bond strength increased by about 20 percent. This result was in contrast with the findings of Martin, et al.,[4] who found that two butyl-based contact adhesives showed little increase in strength after 7 days. In the present study, the uncleaned specimens cured at 158 F (70 C) gave a slight decrease in average strength as the cure time increased from 7 to 14 days.

From Figure 4, the effect of cure temperature was quite apparent. For every comparable condition of cure time and surface cleanness, the average bond strength of specimens cured at 158 F (70 C) was less than that for specimens cured at 73 F (23 C). The finding was not expected. Rather, it had been considered that higher bond strengths might be found with increased cure temperatures due to an acceleration of the curing reaction. Although the data were limited for this observation, they suggested that the rubber/adhesive system used to prepare the laboratory specimens produced lower strengths when cured at higher temperatures.

The data for the T-peel strengths of the laboratory specimens were compared using the statistical three-way analysis of variance procedure.[18] The analysis showed that the effects of the three application conditions—(1) lower bond strength due to lack of cleaning of the rubber, (2) greater bond strength due to longer cure time and (3) lower bond strength due to higher cure temperature—were statistically significant at the 0.01 level or less. In addition, it was found that an additive model for the effects of the factors was reasonable, indicating that the factors act independently.

Note that the two lowest average bond strengths were found for laboratory specimens prepared using uncleaned rubber, and cured at 158 F (70 C). These strengths were about 2.1 to 2.3 foot pounds per inch (0.37 to 0.40 kN/m), which were comparable to the average values found for the field specimens (Figure 4). Although the evidence was limited, it raised questions as to what extent the cleaning procedure in the field and the cure temperatures contributed to the observed values of strengths of the field specimens. The roofing from which the seam specimens were taken was a black EPDM sheet installed in the summer in Washington, D.C. These conditions could have provided some periods of high temperature during curing. In addition, as discussed previously, a few of the field samples exhibited an oil-like film on the surface. More important, as will be discussed in the next section, SEM analysis of selected specimens indicated a talc-like substance on the samples' surfaces.

Scanning Electron Microscopy of Selected Case II Specimens

Scanning electron microscopy (SEM) analyses were conducted to characterize the surfaces of selected field and laboratory rubber samples in the Case II study. In an initial analysis, SEM specimens (Table 3) were prepared from delaminated field and laboratory seams, and also from rubber sheets that had not been fabricated into seams.

With the exception of the rubber sheet cleaned with detergent and water and then hexane (Table 3), the SEM specimens showed surfaces of rubber carrying a talc-like substance (Figure 5). Even the specimen cleaned using the proprietary wash solution showed the presence of talc-like particles across the surface. This was not expected, because cleaning of seams in other laboratory tests (see Case I) indicated that a wash solution had removed essentially all the release agent. For all specimens showing these particles, the micrograph images were comparable, with platelets (ranging in diameter from less than 10 to more than 100 μ) covering the rubber surface. Energy-dispersive X-ray analysis indicated the strong presence of silicon in the particles. Silicon is a predominant element in talc, mica and clays. In contrast to the samples showing the platelets, the one cleaned with detergent and water and then hexane (Table 3) was found to have few talc-like particles visible. Apparently, for the initial laboratory samples under investigation, the cleaning technique using the wash solution did not totally remove the release agent from the rubber surface.

Because the finding of the talc-like particles on specimens cleaned using the proprietary wash solution was not expected, four other specimens of rubber were cleaned with wash solution for additional SEM analysis. Two of these specimens were cleaned one day and two were prepared another day in the event the cleaning technique unknowingly varied over time. Unlike the case of the specimens described in Table 3, the SEM photomicrographs of the additional four specimens showed the surfaces of the rubber to be generally free of the release agent. One of the four had some contamination, but it was not as extensive as for the initial specimens investigated.

Reasons why the cleaning of the rubber using the wash solution in the laboratory did not, in the one instance, remove all the release agent were not determined. The finding was not without precedence, as Westley[6] had observed that cleaning did not always remove all of the release agent. For the present study, normal care was exercised in all laboratory cleaning operations. The finding that most of the release agent was removed in some cases, but not in one, provided evidence that a means for assuring the quality of the field cleaning procedure is needed. The two SEM specimens prepared from the seams taken from the roof also showed the presence of talc-like particles (Figure 5). Although the observations of the investigation were limited, a question raised was whether the rubber sheets were improperly cleaned in the field (i.e., prescribed techniques were not followed), or whether the proper cleaning technique was followed, but was not efficient and left a residue on the surface (as appeared to occur in the laboratory).

The observations from the SEM analysis were compared to the results of the T-peel bond-strength tests for the cleaned and uncleaned laboratory seam samples. Although the two rubber surfaces showed comparable SEM photomicrographs indicating the presence of talc-like particles, the cleaned seam had a peel strength about twice that of the uncleaned seam. Cleaning with the wash solution apparently promoted adhesion, although the release agent was not totally removed.

These data indicate that further investigation of the surface chemistry of the rubber, release agent and adhesive system is needed. An important question to be considered is what effect any remaining release agent on the rubber may have on the long-term seam performance. Another question is why the cleaning with the wash solution did not apparently, in at least one case, totally remove the release agent on the surface of the rubber sheet.

CASE III: EFFECT OF TIME ON THE STRENGTH OF FIELD SEAMS

In the spring of 1988, seam samples were obtained from an EPDM roofing system under construction in suburban Washington, D.C. Although the roof in question was relatively small (about 15,000 square feet or 1400 square meters), its construction had been in progress for more than four months, reportedly due to delays caused primarily by winter and spring weather conditions. Consequently, seams having varying ages were present on the roof. Four replicate specimens were cut from three different seams having ages, as reported by construction personnel, of eight days, about one month and about four months. In this case, the EPDM sheet (0.045 inch or 1.1 millimeter thick) was a different brand name product than those tested in the two previous cases. The adhesive was again a butyl rubber-based product that cured over time.

T-peel tests of bond strength were conducted at the job site using portable test equipment. The results are given in Figure 6, where it is evident that the average T-peel strengths increased with an increase in specimen age. For the eight day, one month and four month ages, the average strengths were 1.5, 2.0 and 3.0 foot pounds per inch (0.68, 0.91 and 1.4 kN/m), respectively, with coefficients of variation of 10, 36 and 15 percent. Statistical analysis indicated that the increase in strength with time was significant. It may have been due to continued cure of the adhesive over the four-month time period.

As measured in the field, the bond strengths of specimens in the three age groups were considered low in comparison to those of some seams prepared in the laboratory using cleaned rubber (see previous discussions). This observation raised questions such as whether these field seams were fabricated using EPDM that was not acceptably cleaned, whether the cure of the adhesive in the field was unexplainably retarded to yield the observed low bond strengths or perhaps both. The delamination of the eight-day old seams was primarily cohesive, indicating that the adhesive had low strength. Conversely, the delamination of the one-month and four-month specimens was primarily adhesive, indicating that surface effects could play a role.

To provide points of comparison of expected bond strengths of this adhesive/rubber system, seam specimens were prepared in the laboratory using cleaned and uncleaned EPDM sheet (0.060 inch or 1.5 millimeter thick[6]), having the same brand name as the product from the field. The cleaning was accomplished by washing with reagent-grade hexane.

These seam specimens were allowed to cure at room temperature for either eight or 32 days before T-peel tests were conducted. These periods were equal to the ages of the two younger specimens cut from the roof. Five replicate specimens were tested for each given condition of time and temperature. The coefficients of variation ranged from 6 to 9 percent.

The results of the laboratory T-peel tests are given in Figure 6 to allow comparison with the data from the field samples. The cleaned specimens achieved a bond strength of about 8 foot pounds per inch (3.6 kN/m). As is evident in Figure 6, the laboratory results showed a noticeable negative effect (i.e., lower strength) due to lack of surface cleaning. This effect was statistically significant at the 0.01 level. In this case, the average strengths of the uncleaned laboratory specimens were about one half those of the cleaned specimens. No effect due to cure time was observed, apparently because the tests were conducted at times when the cure is mainly complete. The strengths of the field specimens were markedly lower than those of the uncleaned laboratory specimens (Figure 6). For example, after eight days cure, the strength difference was about a factor of three. Although these data hinted that the surface of the rubber in the field seams may not have been properly cleaned, it was not definitive. For example, as previously indicted, the eight-day-old field specimens failed cohesively, indicating that the cure of the adhesive may have contributed to the low strength.

SEM analysis was conducted to assess the condition of the rubber surface of some delaminated field specimens. Three SEM specimens were prepared, one from a seam of each of the age groups. The microscopy results were comparable for the three. The photomicrographs indicated platy particles (indicative of a talc-like release agent) covering the surface of the rubber. Figure 7 presents an example and compares it to a rubber specimen cut from the roof and cleaned on its top surface (brushed with detergent and water and then wiped with hexane). The presence of particles on the delaminated seam specimen is quite evident (Figure 7A); whereas the cleaned specimen only shows the rubber surface (Figure 7B). Consistent with the previously obtained data on the negative effect of not removing the release agent on bond strength, it was considered that the particles remaining on these field specimens contributed, in part, to their observed low seam strengths (Figure 6). The investigation was not broad enough to determine whether other effects, if any, also played a role.

SUMMARY

This paper presents the results of laboratory and field investigations conducted to obtain data in support of development of a method to assure the quality of adhesive-bonded seams of single-ply membranes. The prime factors investigated were surface condition of the rubber, the temperature of the rubber at the time of adhesive application and cure time of the adhesive. A summary of the key findings are as follows:

- The laboratory data indicated that the T-peel test is sensitive for use in a field methodology for quality assurance of seams. Based on the laboratory results, it appeared possible to conduct the T-peel tests within a day (or perhaps less) of seam formation and to find significant differences in bond strength due to lack of proper surface cleaning of the rubber. Data from field applications are needed to demonstrate whether such differences are found on the job at one day's time or less.
- Seams sampled from two buildings in the Washington, D.C., area were found to have low bond strengths, as compared to seams prepared using cleaned rubber. These field specimens were also observed to have particles on the rubber surface indicative of release agent. It was considered that the particles contributed to the measured low strength.
- In a laboratory experiment, washing the EPDM with the prescribed wash solution did not totally remove the release agent. The peel strengths of these specimens were high in comparison to comparable specimens prepared using uncleaned rubber. Reasons why the wash did not completely remove the release agent were not determined.

- In one of the field studies, the seams from different sections of the roof had strengths that increased with specimen age. For all these specimens, the strengths were low in comparison to comparable specimens prepared in the laboratory using cleaned rubber. It was questioned whether unexpectedly slow (even under cool conditions) cure of the adhesive in service contributed to the low strength, in addition to the particles found on the rubber surface.
- Because of the unexpected findings of the study (e.g., the lack of complete removal of release agent due to washing), it was suggested that further investigation of the surface chemistry of the rubber, release agent and adhesive system is needed in the development of the proposed methodology.

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[20] Rossiter, W.J. Jr. and Seiler, J.F. Jr., "Report of Roof Inspection: Partial Delamination of Adhesive-Bonded Seams at an Army Facility," NISTIR 88-3893, National Institute of Standards and Technology, November 1988.

[21] 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, p. 60, 1956.

NOTES

[1] The adhesive was kept at room temperature for the 73 and 158 F (23 and 70 C) conditions; whereas it was cooled to 32 F (0 C) when the rubber was at this temperature. The reasoning was that, in practice, the adhesive may not reach the high temperature because it can be shielded from direct solar radiation, but it could cool to the low one as the air temperature drops.

[2] The roof was the responsibility of the U.S. Army Corps of Engineers, which arranged for the field sampling and provided samples of the rubber and adhesive having the brand name of those used for the roof construction.

[3] In this test, a small sample of the compound is burnt on a piece of copper using a laboratory gas flame. If a halide (e.g., chlorine) is present, a green flame is produced.

[4] This terminology is used throughout the report to describe these specimens.

[5] Four replicate good and bad specimens (10 sets) were cut at each sample location, providing a total of 40 specimens. The individual specimens comprising each set were sampled within a few inches of each other to allow repair of the cut with a single patch.

[6] A comparable sample of 0.045 inch (1.1 millimeter) was not available. For purposes of the comparison intended, the differences in sheet thickness were not considered important.

Sample[2] No.	Sample Set	Strength, lbf/in. (kN/m) Range	Average	S.D.	COV %	Sign[3] Diff.
G1	1	2.6 - 3.2 (0.46 - 0.56)	2.8 (0.49)	0.27 (0.047)	9.7	Yes
B1		1.2 - 2.0 (0.21 - 0.35)	1.6 (0.28)	0.42 (0.074)	26	
G2	2	2.5 - 3.2 (0.44 - 0.56)	2.7 (0.47)	0.34 (0.060)	12	No
B2		0.9 - 2.4 (0.16 - 0.42)	1.9 (0.33)	0.66 (0.12)	35	
G3[4]	3	1.2 - 2.1 (0.21 - 0.37)	1.6 (0.28)	0.44 (0.077)	27	No
B3		1.7 - 2.1 (0.30 - 0.37)	2.0 (0.35)	0.18 (0.032)	9.4	
G4[4]	4	2.1 - 2.7 (0.37 - 0.47)	2.5 (0.44)	0.31 (0.054)	12	No
B4		1.2 - 2.2 (0.21 - 0.39)	1.8 (0.32)	0.46 (0.081)	25	
G5	5	1.9 - 2.8 (0.33 - 0.49)	2.3 (0.40)	0.38 (0.067)	17	No
B5		2.3 - 2.8 (0.40 - 0.49)	2.5 (0.44)	0.26 (0.046)	11	
All Good		1.2 - 3.2 (0.21 - 0.56)	2.4 (0.42)	0.51 (0.089)	21	Yes
All Bad		0.9 - 2.8 (0.16 - 0.49)	1.9 (0.33)	0.48 (0.084)	25	
All Specimens		0.9 - 3.2 (0.16 - 0.56)	2.2 (0.39)	0.54 (0.095)	25	

1. Average of four measurements, unless otherwise indicated.
2. G and B refer to "good" and "bad," respectively; see text for explanation.
3. The column indicates whether a significant difference was found at the 0.05 percent significance level between pairs of good and bad specimens.
4. Average of three measurements.

Table 1 *Results of T-peel tests of the field samples* [1]

Variable	Conditions
Cure Temperature	73 or 158 F (23 or 70 C); the high temperature cure included the first day at room temperature in order to minimize a risk of damaging the specimens due to rapid adhesive solvent release
Cure Time	7 or 14 days
Surface Condition of the Rubber	cleaned or uncleaned; cleaning was done according to the prescribed directions using the proprietary solvent for the particular EPDM/adhesive system

Table 2 *Conditions used in the preparation of the laboratory samples included in the Case II study*

Number Analyzed	Description of Specimen and Conditions of Preparation
2	Delaminated field seam; cut from roof
1	Delaminated laboratory seam; rubber was *cleaned* by wiping with a cloth saturated with the proprietary wash solution, and seam was cured two weeks at 158 F (70 C)
1	Delaminated laboratory seam; rubber was *uncleaned* and seam was cured two weeks at 158 F (70 C)
1	Rubber sheet that had not been fabricated into a seam; it was cleaned by scrubbing with detergent and water followed by washing with hexane[1]
1	Rubber sheet that had not been fabricated into a seam; it was *uncleaned* or as received

Table 3 *Specimens subjected to SEM analysis in the Case II field study.*
– The scrubbing with detergent and water has been found by the authors to be an efficient means for removing the release agent from the surface of the rubber sheet

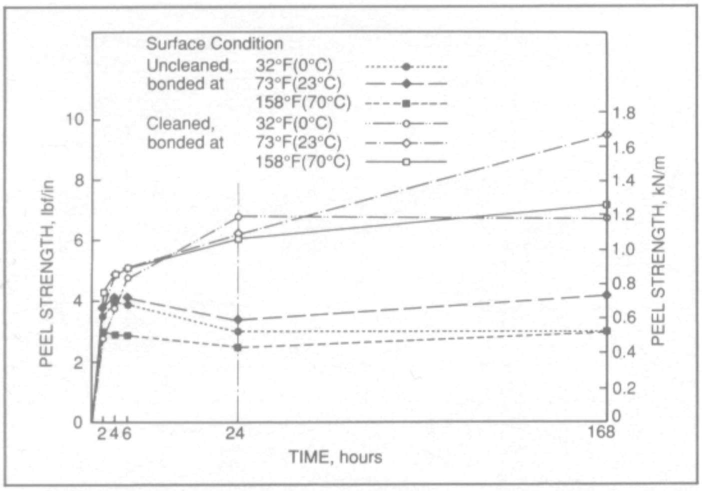

Figure 1 Effect of surface condition (cleaned versus uncleaned), temperature during application and cure time on the bond strength of EPDM seams (Case I)

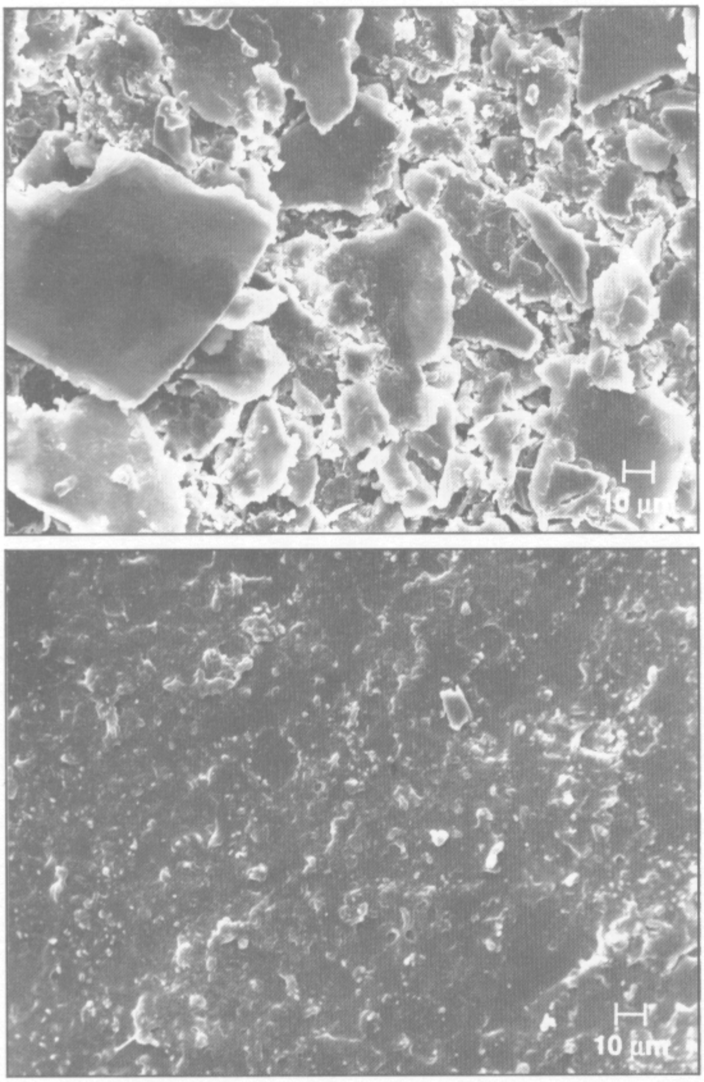

Figure 2 SEM photomicrographs at x500 magnification of the EPDM sheet used in the laboratory study (Case I): (top) the specimen was uncleaned, and (bottom) the specimen was cleaned

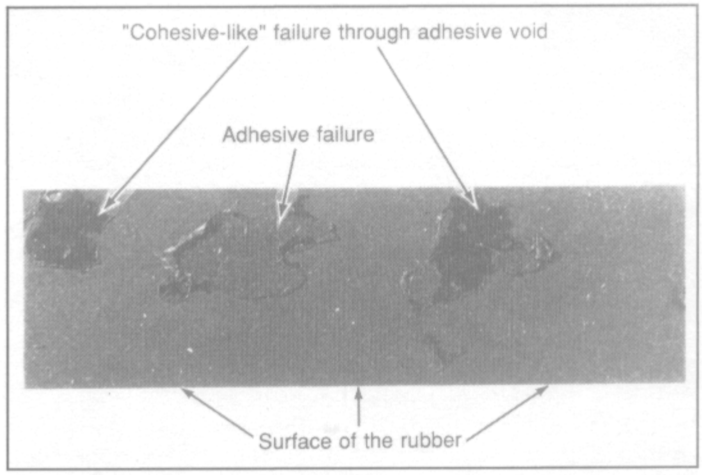

Figure 3 A strip of rubber from a delaminated seam showing residual adhesive that failed adhesively and cohesively through two voids in the adhesive layer. Note that the majority of the surface area has no adhesive on it

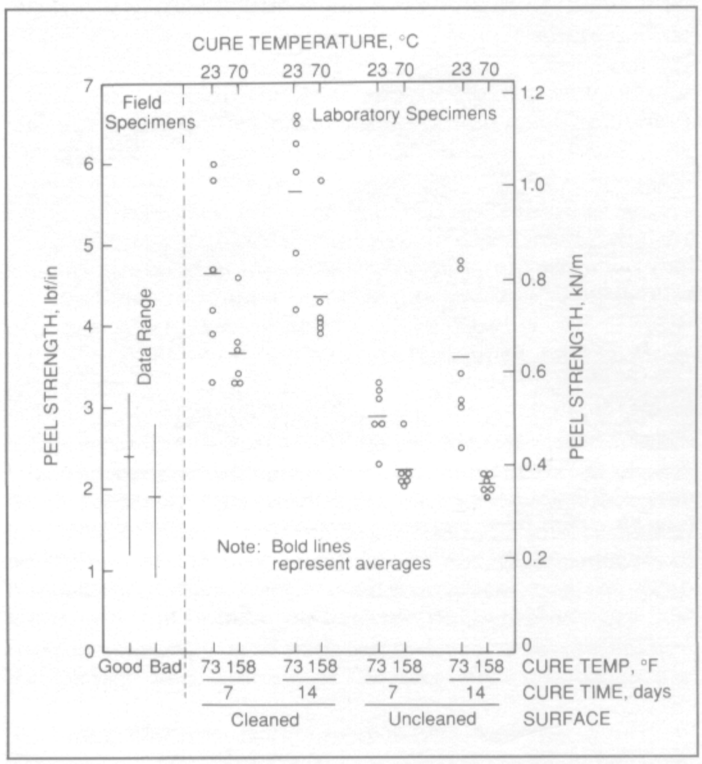

Figure 4 Peel strength of EPDM field seams as determined for Case II. The results are compared to those determined for the laboratory specimens prepared and cured under varying conditions

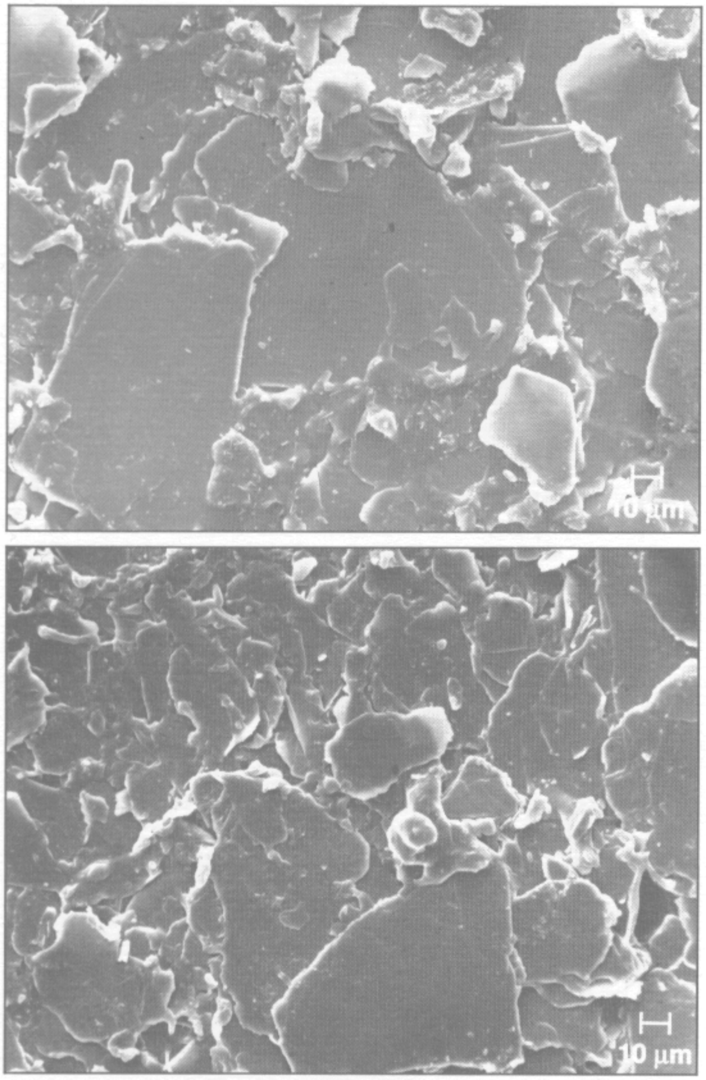

Figure 5 SEM photomicrographs at x500 magnification of selected EPDM sheets used in the Case II study: (top) an as-received specimen, and (bottom) a specimen taken from a delaminated seam cut from the roof

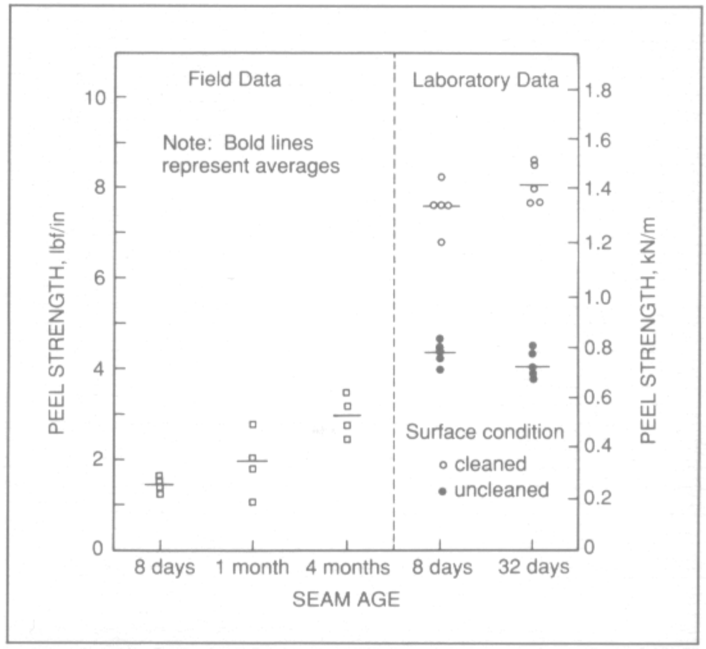

Figure 6 Peel strength of EPDM field seams as determined for Case III. The results are compared to those determined for the laboratory specimens prepared and cured under varying conditions

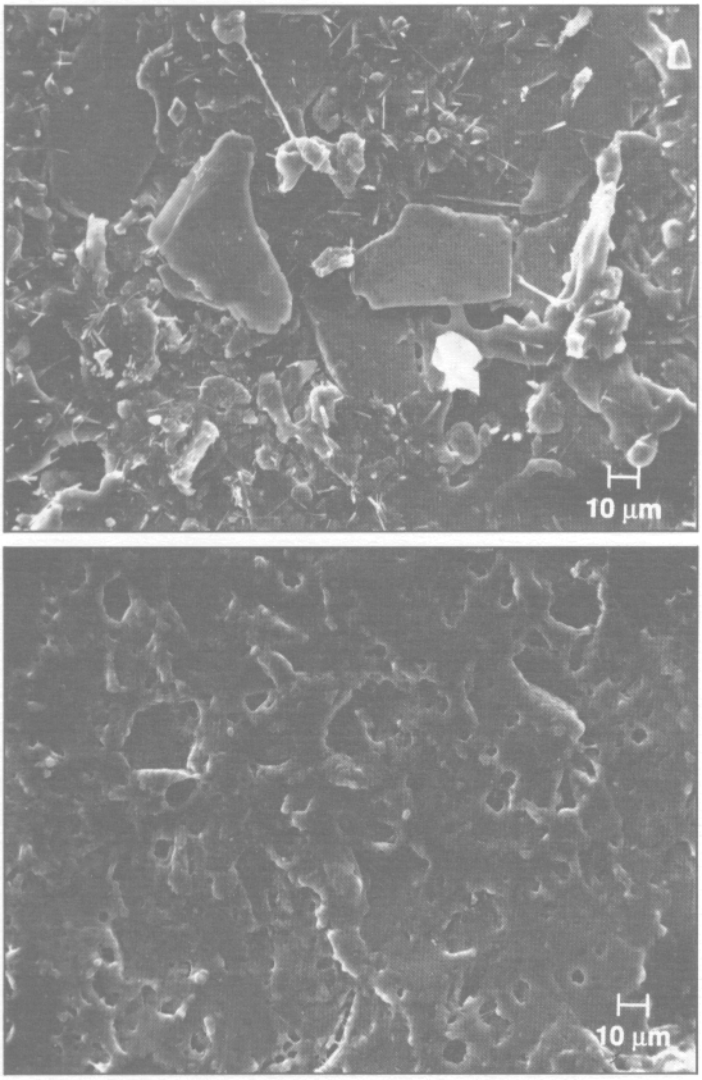

Figure 7 *SEM photomicrographs at x500 magnification of selected EPDM sheets used in the Case III study: (top) a contaminated specimen from the roof, and (bottom) a specimen from the roof that was cleaned in the laboratory*

APPENDIX: GENERAL EXPERIMENTAL PROCEDURES

Seam Preparation

In all cases, commercially available, non-reinforced EPDM sheet was used in the study. For each of the three cases (Cases I, II and III), a different brand name EPDM product was involved. All sheet products were dusted by the manufacturer with a talc-like release agent. For all tests, the EPDM sheets were cleaned according to prescribed procedures, unless an uncleaned surface condition was being investigated. For Cases I and II, proprietary solvent solutions, available specifically from the sheet manufacturers for their products, were used to clean the rubber surfaces; for Case III, hexane solvent was used.

Proprietary butyl-based contact adhesives, in each case specific to the individual EPDM sheet, were used to form the seam specimens. The adhesive was applied to the sheet within one hour after the cleaning. Within a minute after formation of the seam, the specimen was placed in a laboratory press for four to five seconds at 100 foot pounds per square inch (0.7 MPa) pressure. The seams were then allowed to cure in the laboratory at temperatures and times described in the main body of the report.

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 2 inches per minute (50 millimeters per minute). Most laboratory tests were conducted using a common universal testing machine. In these cases, the length of the specimen bond delaminated in the T-peel test was approximately 7 inches (175 millimeters). The testing machine was equipped with a microcomputer, which was used to calculate the average peel force per unit specimen width. The average peel strength was calculated for each specimen over the length of the displacement (after passing the initial peak), and not over only 5 inches (125 millimeters) as given in ASTM D1876.

Some T-peel tests were also conducted at the same rate using a portable T-peel testing machine. In this case, the specimen length was 6 inches (150 millimeters), and specimen delamination was carried out for 3 minutes. Because some elongation of the rubber occurred during testing, the specimens did not totally delaminate during tests. The test machine was equipped with microcircuitry that calculated the average peel strength of the specimens during testing.

Scanning Electron Microscopy (SEM) Analysis

The rubber specimens for SEM analysis were cut, either from the sheet material or from delaminated seam specimens, into squares having about 0.3 to 0.4 inches (8 to 10 millimeters) sides. The cut rubber pieces were adhered to SEM specimen mounting stubs with an epoxy adhesive. The mounted rubber specimens were sputter coated with a nominal 8 times 10^{-7} inches (20 millimeters) 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 and examined at magnifications from x20 to x1000. Photographs were generally taken at x100 and x500 magnifications.

Appendix Reference

A1. "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, 1983.