NISTIR 88-3893

Report of Roof Inspection: Partial Delamination of Adhesive-Bonded Seams at an Army Facility

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Gaithersburg, MD 20899

November 1988

Prepared for:
U.S. Army Engineer District, Baltimore
P O Box 1715
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ABSTRACT

This document was prepared at the request of U.S. Army Engineer District, Baltimore, to provide assistance in obtaining data on the delamination of seams of an EPDM roofing system at Fort Belvoir, Virginia. The investigation was beneficial to NIST, because it provided an opportunity to characterize adhesive-bonded seams in service and to obtain data relative to NIST laboratory research on the effect of surface contamination on seam performance.

Seam specimens were taken from the roof and analyzed for peel strength and surface condition of the rubber. In addition, seams were prepared in the laboratory using the same brand name rubber/adhesive system to obtain peel-strength values for comparison with those measured for the field specimens. The results of the study indicated that the field specimens had low T-peel bond strengths in comparison to the strengths achieved by the laboratory-prepared seams. Small voids in the adhesive layer of the seams of the field seams may have contributed, in part, to the low bond strength. SEM analysis of the field-formed seams indicated the presence of a talc-like contamination on the rubber surface which may have also contributed to the low strength. In addition, SEM analysis of some laboratory specimens cleaned with the proprietary wash solution showed a talc-like contamination which was not visible to the unaided eye. Other laboratory specimens cleaned using the wash solution did not show such contamination.

Key words: adhesive-bonding, bond strength, contamination, EPDM, field inspection, low-sloped roofing, membranes, roofs, seams, SEM analysis, surface condition

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1. INTRODUCTION

In September, 1987, the U.S. Army Corps of Engineers (COE), Baltimore District, requested the NIST Center for Building Technology to provide information on the performance of adhesive-bonded seams in EPDM (ethylene-propylene-diene terpolymer) roofing systems. The request centered on the roofing of the new State-of-the-Art Medium Terminal (SAMT) Building at Fort Belvoir, Virginia. The system had a fully-bonded EPDM membrane constructed in the late summer of 1986. During a COE inspection of the roof conducted shortly prior to the request, hairline cracks were found in the lap-sealant applied along the outside edge of the seams. Some delamination of small sections of seams was also observed. The COE asked that NIST research staff inspect the roof for purpose of assisting the COE in analyzing the problem occurring with the roofing, and in particular the lap-sealant.

Two NIST researchers visited the roof twice accompanied by a COE representative. The first visit was on 10 October 1987 to observe the roofing, and, if warranted, to suggest plans for further investigation. It was decided to conduct further investigation concerning the seam condition, particularly regarding contamination of the surface of the EPDM rubber at the seams. Thus, a second visit was made on 24 November 1987 to take samples of the seams for laboratory testing and analysis. Other observers from NIST, COE, and the Navy were present during the second visit.

This report presents a summary of the field inspections and laboratory testing. The investigation was beneficial to NIST, because it provided an opportunity to characterize adhesive-bonded seams in service and to obtain data relative to NIST laboratory research on the effect of surface contamination on seam performance.

2. OBSERVATIONS FROM THE FIRST FIELD INSPECTION

2.1 Roof Construction

At the time of this inspection, COE staff described the specified roof construction (Figure 1) as follows:

1. A monolithic steel deck constructed from steel plates welded at the seams. The thickness of the plates was not given. The steel could not be penetrated which precluded mechanical attachment of the insulation and/or membrane.
2. A single layer of polyisocyanurate cellular plastic board, 2 in. (50 mm) thick. The insulation was secured to the deck using hot asphalt.
3. A single ply of EPDM membrane, 0.060 in. (1.5 mm) thick, totally adhered to the insulation using a contact adhesive. Seams bonding adjacent sheets were about 10 ft (3 m) apart. A lap-sealant was applied to the outside edge of the field seams after installation of the roofing (Figure 1).
4. Field seams were prepared by cleaning (washing) the rubber sheets at the overlap areas with a proprietary solvent (wash) solution. This was a hydrocarbon-based solvent that was black in color. After cleaning, a proprietary contact adhesive was used to form the bonds.

4. The building was about 38,000 ft^2 (3500 m^2), with few penetrations. Figure 2 shows a plan-view outline of the roof without indicating the penetrations.

2.2 Observations and Notes

The following observations were noted during the inspection of 10 October 1987:

- The membrane was black rubber, typical of EPDM. The membrane was totally adhered and, in general, was flat on the substrate without wrinkles and buckles. One small area at the southeast corner showed some wrinkling for reasons which were not determined. Field seams between sheets were generally 6 in. (150 mm) wide. Lap sealant had been applied to all edges of the seams (Figure 1). In some areas, the lap-sealant was re-applied by the contractor because of hairline cracking that had occurred in the original lap-sealant (see discussion that follows).
- The slope was apparent and considered to be generally adequate. The specification reportedly required 1/4 in./ft (20 mm/m). One minor area of ponding of water was observed near a parapet wall at the northwest corner of the building.
- There was some "picture-framing[1]" of insulation boards. While walking across the roof, some of the boards could be felt to move when they were stepped on. This indicated that some boards were not well adhered to the deck. COE staff explained, when construction began, many insulation boards

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[1]Imaging of the outline or edges of the insulation board through the membrane.

did not lie flat on the steel deck. This was due to the raised welds between some deck panels. During roof construction, grooves were cut in some insulation boards to accommodate the welds in an attempt to allow the boards to set flat on the deck.

- In observing the in-place lap-sealant, many areas were seen where a hairline crack had formed parallel to the seam. The crack was along the edge of the upper sheet comprising the seam (Figure 1). (The hairline cracks were as described in the COE request for assistance, and were the reason for inspecting the roof.) In two small areas, a sample of sealant was removed from the seam using a knife. The removed sealant samples were judged to be pliable. Although the evidence from this observation was limited and subjective, it suggested that embrittlement of the lap-sealant had not occurred. Further testing of the lap-sealant was not performed.
- Many areas of the seam where hairline cracks were observed in the lap-sealant were probed using the tip of a jack-knife blade. The blade tip was inserted into the hairline crack and into the edge of the seam. It was found that, in many areas probed in this manner, the lap seam had delaminated along its edge. The extent of delamination varied from location to location on the roof. No consistent pattern was observed. The amount of delamination into the seam ranged from tenths of inches to a couple of inches or more (a few millimeters to 50 mm or more). No areas were found where the seam had completely delaminated across its entire width. However, not all the seams of the roof were probed. Dirt

was generally observed in the delaminated seam areas. The dirt was found on top of the adhesive, which was taken as an indication that it entered the delamination after adhesive application to the rubber sheet, and after seam formation.

- In considering reasons for the hairline cracks in the lap-sealant, the question was asked whether they were symptomatic of some delamination of the seam. This was raised because of the observation that some seam delamination at the edge of the lap was generally present where the cracks in the lap-sealant were found. Whether a cause and effect relationship existed between the cracks in the lap-sealant and the delamination of the seams was not known. Further, the question was not investigated in this brief investigation. Nevertheless, because some delamination was occurring in the seams, it was decided to conduct a laboratory investigation of the seam condition.
- During this first inspection, the COE representative had informed NIST staff that the roofing contractor had planned to re-apply the lap-sealant in the areas where cracking had occurred (see authors' note below). However, it was considered that such a repair might not eliminate the hairline cracks, if they were not due to poor lap-sealant performance, but were symptomatic of seam delamination. With that in mind, some areas of seams where a second application of lap-sealant had been applied over that originally installed were closely examined for the presence of cracks. In some locations, cracking of the second application of the lap-sealant had occurred.

- (Authors' note: Subsequent to the first inspection of the roof and prior to preparing the present report, COE had a Washington-based roof consultant perform an inspection of all areas of the seams. The consultant recommended that the seams should be repaired. The COE representative indicated that the seams were to be re-covered with strips of EPDM rubber, and that further application of lap-sealant to existing seams was not to be done.)

3. OBSERVATIONS & TESTS ASSOCIATED WITH THE SECOND FIELD INSPECTION

The second inspection was conducted to obtain seam samples for laboratory testing of strength and observation of the surface condition of the rubber sheets from the delaminated samples. The following tasks were associated with this part of the investigation:

- Taking seam samples
- Conducting T-peel tests of the field seam samples
- Noting condition of the rubber surface of the delaminated seam samples
- Preparing new laboratory specimens for T-peel testing
- Determining the type of adhesive used to prepare the seams
- Conducting scanning electron microscopy analysis of selected samples.

3.1 Test Samples and Observations Noted During Field Sampling

Test samples of the lap seam were taken from five general sections of the roof: each corner and in the center of the building (Figure 1). Before cutting the membrane, it was decided to take both "good" and "bad" samples from each of the 5 sections. Good samples were those having no hairline cracks visible in the lap-sealant; whereas bad samples had a crack in the lap-sealant and some delamination of the bond along the seam edge, though not across the entire width of the seam.

Four replicate good and bad specimens were cut at each sample location, providing a total of 40 specimens. At each location, the specimens in each set (whether good or bad) were sampled within a few inches of each other to allow repair of the cut with a single patch. Samples of the insulation board were not taken during the inspection.

During sampling, observations concerning the condition of the specimens and the roofing were noted. These observations are

summarized in Table 1. A major observation was the presence of small voids (1-2 mm) in the adhesive layer, which 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 is small air pockets were entrapped between the individual sheets mated to form the seam. Another suggestion is that the 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.

From Table 1, it can be seen that most of the sampled areas of the roof under the cut membrane appeared to be dry. Water was found under sample location No. 2 where the good samples were cut. Assuming that the roof was applied in a dry state, then the water apparently entered the system through a delamination that totally traversed the seam. No puncture or other defect was observed in the membrane in the location of the test sample cut.

Another important observation made during seam sampling was the poor adhesion of the facer sheet[2] to the insulation board observed at two locations (G2 & G4). Although the observation was limited to two locations, it suggested that, in some areas of the roof, the attachment of the membrane to the substrate was less than expected. The observation was taken as evidence of the importance of routine periodic inspection of this roof to assure,

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[2]Mat that was factory-bonded to the face of the insulation board.

among other factors, that the roof membrane remains adequately secured to the substrate.

3.2 T-peel Tests of the Field Seam Samples

The T-peel strengths of the lap seam specimens were determined using a portable testing machine[3]. The tests were based on ASTM Standard Method D 1876 and conducted at room temperature, 70 to 72 °F, (21 to 22 °C) at a rate of 2 in./min (50 mm/min). Samples G1 and B1 were tested in a room of the SAMT Terminal Building shortly after they were cut from the roof. This was to demonstrate the applicability of the portable test machine to obtaining data in the field, and its usefulness in addressing a practical problem. The remaining samples were tested in the NIST laboratories with the portable test machine.

The results of the T-peel tests are given in Table 2 for the individual sample sets and also the pooled sets of all specimens described as good and bad. The data for each set of good and bad samples, as well as the pooled data for all specimens, were compared using the statistical t-test technique. The comparisons were made at the 0.05 percent significance level. The results of the t-test comparisons are given in the right column of Table 2. As is evident, the average values of the peel strength were only significantly different for the good and bad specimens of Sample Set 1. The other sets (nos. 2-5) showed no significant difference.

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[3]Model SP-103A, Instrumentors, Inc. The brand name is given to specify adequately the equipment used. In no case does such identification imply recommendation or endorsement by NIST.

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 strength for all good and bad specimens were 2.4 and 1.9 lbf/in. (0.42 and 0.33 kN/m), respectively (Table 2). This difference was statistically significant at a significance level of 0.05%, even though four of the individual sets were not significantly different. The pooled data is 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. Nevertheless, the data indicate that, on the average, the samples cut from seam areas where the lap-sealant displayed hairline cracks had lower strengths than those areas where no cracking was present.

3.3 Observations on the Condition of the Field Seam Samples

After delamination of the T-peel specimens, each was examined visually in the laboratory for evidence of contamination or other factors that may affect bond strength. A summary of the comments recorded during the examinations is given in Table 3. Three major observations from the examinations may be noted.

First, the majority of the specimens (23 cases) showed little or no surface contamination as seen by eye. Some limited light microscopy was conducted on selected specimens at 25 magnification,

but the results gave no further evidence of the surface condition. Although extensive surface analysis of surface contamination was beyond the scope of this investigation, limited analyses using scanning electron microscopy were performed and are discussed in Section 3.6.

Second, six specimens (B1-1 through B1-4, B2-2, and B2-4) were found by eye to have noticeable surface contamination, which appeared as a brownish oil-like film on the rubber surface. The source of the film was not ascertained, and the oil-like material was not identified. Possible reasons for the presence of the film include random contamination of the rubber during seam formation, perhaps due to use of an unclean cloth in cleaning (wash solution) the rubber surface in the field, or maybe the migration of a foreign substance from the rubber to the sheet surface. If the latter were the cause, it is thought that the oil-like film would have been more wide spread. The finding of some obvious surface contamination raises questions as to whether adequate cleaning of the rubber surfaces was accomplished during fabrication of the field seams, and if other non-visible contaminants were present on the seam specimens.

The third significant observation was the presence of pockmarks in the adhesive layer. These pockmarks were numerous (see Table 3) and consistent with the observations made in the field that the specimens had voids (i.e., areas of apparently no bond) in the adhesive layer. It was considered that the pockmarks contributed, in part, to the low bond strength of the samples

(see Section 3.4), because they represented areas of little or no bond. In the areas of the pockmarks, the peel failure of the seam was generally seen to be "cohesive-like," in that, in these areas, the failure was by delamination through the void in the adhesive and not by peeling of the adhesive from the surface of the rubber.

3.4 Laboratory Specimens for T-peel Testing

COE staff provided NIST researchers with samples of the EPDM sheet, contact adhesive, and wash solution (Section 2.1), reported to be of the same generic type and brand names as those used to construct the Fort Belvoir membrane. The batches or lots of these materials were different from those of the roofing construction, because of the time that had elapsed between roof construction and the present investigation.

The surfaces of the EPDM sheet were only lightly dusted with a talc-like powder (release agent). Thus, the surface color was essentially black, unlike some new EPDM products which have a silvery-gray appearance due to a heavy dusting of release agent. The noted lack of excessive dusting raised a question as to the type of process used to coat the sheet with release agent during production. In particular: Was the process different than that used to dust sheets that are silvery-gray in color? And does it result in a dusting that is difficult to remove by cleaning with the wash solution?

The generic type of contact adhesive, whether butyl- or neoprene-based, was not known, and the container only bore the brand name identification of the product. COE construction records for the roof indicated that a "new" seam adhesive was to be used. This fact implied that the adhesive was butyl, because such adhesives were replacing neoprene adhesives during the time the Fort Belvoir roof was built.

3.4.1 T-peel Tests. Using the materials provided, seam specimens were prepared in the laboratory for testing in T-peel at 2 in./min (50 mm/min), using the same procedure applied to the field specimens. Six replicate specimens were tested for each application condition under which the seams were prepared. Table 4 gives the various conditions used in the preparation of the seams. The cleaned specimens were prepared using the proprietary solvent wash provided by COE personnel. The bond strengths of the laboratory specimens were compared with those of the field specimens. The laboratory tests measured values of bond strengths that might be expected for specimens cleaned using the solvent wash, as well as those of uncleaned specimens.

Table 5 presents the results of the T-peel tests for the laboratory specimens. The data are summarized in Figure 2 where a comparison with the field test results is also made. 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 lbf/in. (1.0 kN/m) provides an indication of the expected maximum strength that this rubber/adhesive system (including the wash solution) may reach

under close-to-optimum conditions of seam formation. (Longer cure times may produce stronger bonds, but this parameter was not further investigated in the present study.) The average maximum value of 5.7 lbf/in. (1.0 kN/m) was more than twice the average value of 2.8 lbf/in. (0.49 kN/m) found for all field specimens.

The value of 5.7 lbf/in. (1.0 kN/m) was suggestive that the adhesive was not neoprene-based, because neoprene adhesives for EPDM membranes generally achieve T-peel strengths of about 2 lbf/in. (0.4 kN/m.). On the other hand, some butyl-based adhesives have been found to reach bond strengths of 8 lbf/in. (1.4 kN/m), when applied to carefully cleaned EPDM sheet and cured for 7 days at room temperature.

The importance of the three application conditions (Table 4) on seam strength is evident from Figure 2. 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. The finding was not unexpected, since previous studies using T-peel tests had indicated loss in T-peel strength due to uncleaned surfaces. Moreover, in the present study, the average bond strength of any set of uncleaned specimens had lower strength than all cleaned specimens. In fact, the averages for specimens representing three of the four uncleaned conditions (Figure 2) were between 2 and 3 lbf/in. (0.4 and 0.5 kN/m), which was the approximate range for the average values of all field samples

(Table 2). Only the uncleaned specimens cured 14 days at 73 °F (23 °C) had strength values higher than those of the field samples.

It is also evident from Figure 2 that, as the cure time lengthened from 7 to 14 days, the bond strength generally increased. This result was also consistent with previous NIST findings regarding a butyl-based contact adhesives which cures over time. An exception was for the uncleaned specimens cured at 158 °F (70 °C). In this case, a slight decrease in strength was observed in time. No reason for the observation was apparent.

From Figure 2, the effect of cure temperature was quite apparent. For every comparable instance of cure time and surface condition, 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 was expected that higher bond strengths would be found with increased cure temperatures due to an acceleration of the curing reaction. Although the data were limited for this observation, they did suggest that the adhesive/rubber system used to prepare the laboratory specimens tended to give lower strengths when cured at high temperatures.

The data for the T-peel strengths of the laboratory specimens were compared using the statistical 3-way analysis of variance procedure. As just discussed, the effects of the three application conditions analyzed were: (1) lower bond strength due to lack of cleaning of the rubber, (2) greater bond strength due to longer

cure time, (3) lower bond strength due to higher cure temperature. The analysis showed that all three factors were statistically significant. In addition, it was found that an additive model for the effects of the factors was reasonable, indicating that the factors act independently.

Finally, in discussing the effect of the specimen preparation conditions, it is noted that the two lowest average bond strengths were found for specimens prepared using uncleaned rubber, and cured at 158 °F (70 °C). These strengths were about 2.1 to 2.3 lbf/in. (0.37 to 0.40 kN/m), which, perhaps coincidentally, were comparable to the average values found for the field specimens (Figure 2). 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 roof with a black EPDM sheet was installed in the summer in Washington, DC. which could have provided some periods of high temperature during curing. In addition, as discussed previously, a few of the field samples exhibited noticeable surface contamination. More importantly, as will be discussed in Section 3.6, SEM analysis indicated a talc-like substance on the surfaces of the rubber.

3.4.2 Appearance of the Adhesive of the Delaminated Specimens. Each of the laboratory specimens was examined after T-peel testing to observe the condition of the adhesive and to note the type of failure, whether adhesive or cohesive[4]. Another point of interest was to see if pockmarks were present in the adhesive layer, as was found for the field specimens. In the case of the laboratory specimens, pockmarks were not observed. A summary of the types of failures is given in Table 6. Note that the majority were adhesive failures even for the cleaned specimens, indicating that the weak link in the seam was at the interface of the adhesive and rubber sheet.

3.5 Nature of the Adhesive

Fourier Transform Infrared Analysis (FTIR) was conducted on the adhesive present in the field samples, and that used to prepare the laboratory samples. The intent was to determine whether the adhesives were the same, and if possible, to identify the generic type of adhesive, butyl-based or neoprene-based. This was done because the construction records did not indicate the type of adhesive.

FTIR spectra of selected field and laboratory samples were compared with those of a known neoprene adhesive and a known butyl adhesive. These known adhesives were commercially-used products available in the NIST laboratories. In this limited investigation, the FTIR evidence was not sufficient to provide

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[4]Adhesive indicates failure at the interface of the adhesive and rubber surface; whereas cohesive indicates failure within the bulk of the adhesive.

conclusive identification of the adhesive. The observations made were:

- The FTIR spectra for the known neoprene and butyl adhesives were distinctly different.
- The spectra of the field and laboratory samples were very similar. The comparison suggested that the adhesives might be the same. The finding was consistent with the fact that NIST staff were given a sample of adhesive reported to be the same generic type as that used in the field.
- No conclusions were made from the comparisons of the spectra of laboratory adhesive with that of the known neoprene or butyl adhesives, because the spectra of the materials in question were not clearly distinctive.

3.5.1 Beilstein Tests of the Adhesives. A classical qualitative analysis procedure for the identification of halide-containing organic compounds is the "Beilstein Test." In this test, a small sample of the compound is burnt on a piece of copper using a laboratory gas flame. If a halide is present, a green flame is produced.

The test was applied to the known adhesives and that from the roof in question. A difference in the flames of the known neoprene and butyl adhesives was apparent. The neoprene produced a strong, bright green flame. The butyl gave a strong yellow flame with a trace of green when initially ignited. The green color may have been generated by a trace of chlorinated polymer or solvent in the adhesive. The adhesive used to prepare the laboratory samples behaved almost identically to the known butyl adhesive. The adhesive from the field samples gave only a yellow flame. The conclusion was that the adhesives from the field and laboratory samples were not neoprene-based. This was consistent

with the T-peel data for some laboratory specimens that suggested, on the basis of strength values, that it was not neoprene.

3.6 Scanning Electron Microscopy

Scanning electron microscopy (SEM) analysis of selected rubber sheets including that received by COE personnel, those taken from delaminated field seams (Section 3.2), and those from laboratory-prepared seams (Section 3.4) was conducted to obtain information on the rubber surfaces. The SEM samples from delaminated seams were as follows: two field samples (B3-2 and B2-3), and two laboratory samples (one using rubber cleaned with the solvent wash, C4, and one using uncleaned rubber, UC4; both cured two weeks at 158 °F/70 °C). The samples of the rubber sheet received from COE personnel were as follows: one uncleaned sample (in the "as-received" condition), and one cleaned (brushed using laboratory detergent and water followed by a hexane wash and air drying). Photomicrographs were obtained for all analyzed samples at 100X and 500X magnification.

The SEM analysis, performed on the top surfaces of these samples, provided information concerning the condition of the rubber surfaces. The results were that, with the exception of the rubber sheet sample cleaned with soap and water and then hexane, the samples showed surfaces of rubber contaminated with a talc-like substance. Even the specimens cleaned using the proprietary wash solution showed the presence of talc-like particles across the surface. For all samples showing these particles, the coverage of the rubber surfaces was comparable. The particles

were clearly visible as platelets, having variable size ranging from less than 10 m to more than 100 m, that appeared to cover the rubber surface totally. Energy-dispersive X-ray analysis gave a strong indication of silicon in the substance. Silicon is a predominant element in talc, mica, or clay. In contrast to the specimens showing the talc-like particles, that cleaned with soap and water and then hexane was found to have little talc-like particle contamination. Apparently, for the initial laboratory samples under investigation, the cleaning technique using the wash solution provided by COE personnel did not totally remove the talc-like substance from the rubber surface.

Because the finding of the talc-like contamination on specimens cleaned using the proprietary wash solution was not expected, it was decided to clean (using the was solution) four other specimens of rubber for additional SEM analysis. Two of these specimens were cleaned one day and two were prepared another day in case the cleaning technique varied over time. Unlike the case of the initial specimens, the SEM photomicrographs of the additional four specimens showed the surfaces of the rubber to be generally free of the talc-like contamination. One of the four had some contamination, but it was not considered 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 talc-like particles were not determined. It was felt that normal caution was exercised in all laboratory cleaning operations, and

that the wash technique was expected to provide a surface relatively free of the talc-like particles. The finding that the talc-like substance was generally removed in the laboratory using the wash solution in one case and not in another provided evidence that a means for assuring the quality of the cleaning procedure is needed.

The SEM examination of the samples taken from the Fort Belvoir roof showed talc-like contamination. Although the observations of this investigation were limited, a question raised was whether the rubber sheets were improperly cleaned in the field (leaving some residue of the release agent), 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).

In the case of the contaminated surfaces, the talc-like substance was only observed by SEM analysis, which was in direct contrast to the visual observations made using the naked eye. By eye, the surfaces of all samples cleaned in the laboratory appeared to be free of talc-like substance. Also, the delaminated field samples were apparently talc-free. The finding that contamination was not seen by eye (nor by light microscopy at 25x), even though the SEM showed it to be present, provided strong evidence that the unaided eye is not adequate for characterizing surfaces. A question is whether the black proprietary wash solution may have darkened the rubber surface and prevented detection of talc-like contaminants by eye.

The findings of the SEM analysis were considered in relation to the results of the T-peel bond-strength tests for the cleaned (C4) and uncleaned (UC4) laboratory seam samples (Table 5). Although the rubber surfaces showed comparable SEM photomicrographs, indicating surface contamination in both cases, the cleaned and uncleaned seams displayed different average T-peel bond strengths. For room temperature cure, the average peel strength of the cleaned sample (C4) was 2.3 times that of the uncleaned sample (UC4). The presence of a layer of talc-like substance may explain the earlier-mentioned observation that the strength of the cleaned specimens (about 5 to 6 lbf/in. or 0.9 to 1 kN/m) were less than 7 to 8 lbf/in. (1.2 to 1.4 kN/m), as has been observed with other cleaned seams prepared by the authors with butyl adhesive. The wash solution apparently promoted increased adhesion in the case of cleaned specimens over that of the uncleaned specimens even though the talc-like substance was not totally removed. Perhaps partial removal of contamination during the cleaning was sufficient to provide the greater bond strength.

These results raise questions concerning the understanding of performance of the rubber, release agent, and adhesive system, and suggest that further investigation of their surface chemistry is needed. An important question is what effect any remaining talc-like layer on the rubber may have on the long-term seam performance. Another question is why the laboratory cleaning with the wash solution did not apparently, in at least one case, totally remove the talc-like release substance on the surface of the rubber sheet.

4. SUMMARY AND CONCLUSIONS

This investigation was a limited study of problems occurring with the seams of the EPDM membrane on the roof of the SAMT building, Fort Belvoir, Virginia. Based on the results of the study, the following summary of key observations is made:

- The seams of the membrane had experienced some delamination within about a year of formation. In the areas observed, the delaminations had not occurred across the entire width of the seams.

- Specimens of seams removed from the roof had low T-peel bond strengths in comparison to the strengths achieved by the given adhesive/rubber system when the seams were prepared under laboratory conditions using rubber washed with the proprietary wash solution.

- SEM analysis of the field-formed seams indicated the presence of a talc-like contamination on the rubber surface which may have contributed to the low strength. The contamination was not visible to the eye.

- Specimens of seams removed from the roof showed the presence of small voids in the adhesive layer. The small voids in the adhesive layer of the seams may also have contributed, in part, to the low bond strength, because they represented areas of little or no bond.

- Not cleaning the rubber surface and elevating the cure temperature had a negative effect on the bond strength of the laboratory-prepared seams. Seam strengths were reduced about 40 percent when the rubber surfaces were not cleaned. The strengths were also reduced by more than 20 percent when cured at 158 F° (70 °C) versus curing at 73 °F (23 °C).

- SEM analysis indicated that some laboratory specimens cleaned with the proprietary wash solution showed a talc-like contamination which was not visible to the unaided eye. Other laboratory specimens cleaned using the wash solution did not show such contamination. In both cases, a recommended cleaning technique was followed. These results provided evidence that a means for assuring the quality of cleaned rubber is needed.

From the study results, the following recommendation regarding the Fort Belvoir roof is made:

> The roof should receive thorough periodic inspection at 6-month intervals to assure that it performs as expected.

This recommendation may appear obvious, considering that acceptable roofing practice emphasizes routine periodic inspection (normally every 6 months) to maintain satisfactory performance. It is emphasized herein for the following reasons:

- The Corps of Engineers has indicated that repair of the existing seams is to be accomplished by adhering a strip of EPDM rubber over them. Experience has shown that some rubber patches made over weathered EPDM have not always provided satisfactory performance, but have prematurely delaminated. Thus, the periodic inspection should pay particular attention to the repair strips over the original seams to assure that they remain intact. On another point, the evidence obtained in the laboratory indicated that the talc-like substance on the surface of the rubber in question was not, in one case, totally removed by normal cleaning with the recommended wash solution. This suggests that the surface of the rubber may have been difficult to clean for the repairs.

- The investigation revealed some poor adhesion of the facer to the insulation board. Although not discussed in detail, this observation indicated that, in some areas of the roof, the attachment of the membrane to its substrate may have been less than adequate. Thus, the inspectors should check that the membrane is sufficiently adhered to the substrate, and not experiencing securement problems due to delamination of the facer from the insulation board.

In conclusion, this limited field investigation provided evidence concerning the surface condition of rubber sheets taken from seams experiencing delamination problems in service. The evidence raised questions why the wash solution used to clean the rubber before adhesive application did not totally remove the talc-like release agent from the rubber surface. The study suggested that continued research is needed to improve the understanding of the rubber-adhesive interface.

5. ACKNOWLEDGMENTS

The authors extend their thanks to Mr. Cy Miller, Corps of Engineers, Baltimore District, for his support in conducting the field inspections, and for obtaining the materials used for the laboratory tests. The authors also appreciate the assistance of their NIST colleagues: Paul Stutzman, Building Materials Division, for his enthusiastic efforts in providing SEM analysis; Geoffrey Frohnsdorff, Larry Masters, and Mary McKnight, Building Materials Division, for their many useful comments on a draft of this report; Gerald Sleater, Center for Analytical Chemistry, for his valuable review of a draft; and James Lechner, Center for Computing and Applied Mathematics, for his assistance with data reduction and analysis.

Table 1. Observations Noted During Sampling of the Seams

Sample[1] No.	Observations
G1, G3, G5, B2, B3, B4, & B5.	The seam specimens showed, as viewed along the edge, some small voids in the adhesive layer; no water was found in the cellular plastic insulation board which appeared dry to the touch.
B1.	The seam specimens showed, as viewed along the edge, some small voids in the adhesive layer; no water was found in the insulation and it appeared dry to the touch.
G2.	The seam specimens showed, as viewed along the edge, some small voids in the adhesive layer; water was found in the cellular plastic insulation board, as noted by pressing a finger on its surface; the adhesion between the membrane and insulation was poor; the facer on the insulation appeared to be readily delaminated; in one spot, an extended tape measure could be inserted 6-7 in. (150-175 mm) under the membrane.
G4.	The seam specimens showed, as viewed along the edge, some small voids in the adhesive layer; no water was found in the insulation and it appeared dry to the touch; the adhesion between the membrane and insulation was described as poor; the facer on the insulation appeared to be readily delaminated; in one spot, an extended tape measure could be inserted about 15 in. (375 mm) under the membrane.

1. G and B refer to "good" and "bad," respectively; see text (p. 7) for explanation.

Table 2. Results of T-Peel Tests of the Field Samples[1]

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 (p. 7) for explanation.
3. The column indicates whether a difference was found at the 0.05 percent significance level between pairs of good and bad specimens.
4. Average of three measurements.

Table 3. Observations on the Condition of the Field Seam Specimens[1]

Sample[2] No.	Specimen No.	Failure Mode[3]	Observations
G1	1	A - 95%	no visible signs of contamination; small pockets in the adhesive layer[4]; not shiny[5]
G1	2	A - 95%	as for G1 - 1
G1	3	A - 95%	as for G1 - 1
G1	4	A - 95%	as for G1 - 1
B1	1	A - 50%	significant contamination; brownish oil-like film on bottom of top sheet and on the top of adhesive of bottom sheet; small pockets in the adhesive layer[4]; shiny[5]
B1	2	A - 50%	as for B1 - 1
B1	3	A - 90%	as for B1 - 1
B1	4	A - 90%	as for B1 - 1
G2	1	A - 95%	no visible signs of contamination; small pockets in the adhesive layer[4]; shiny[5]
G2	2	A - 95%	as for G2 - 1
G2	3	A - 75%	as for G2 - 1
G2	4	A - 75%	as for G2 - 1
B2	1	A - 80%	as for G2 - 1
B2	2	A - 90%	as for B1 - 1
B2	3	A - 90%	as for G2 - 1
B2	4	A - 60%	as for B1 - 1

1. These observations were made by eye on the delaminated field specimens.
2. G and B refer to "good" and "bad," respectively; see text (p. 7) for explanation.
3. This column represents the percent area of the specimen estimated to fail adhesively (A); the remainder is cohesive failure.
4. This note refers to pockmark-like depressions in the adhesive in areas where the failure was "cohesive" in nature.
5. This note refers to the appearance of the surface of the adhesive in the pockmark areas.

Table 3. Observations on the Condition of the Field Seam Specimens[1]

Sample[2] No.	Specimen No.	Failure Mode[3]	Observations
G3	1	A - 90%	as for G2 - 1
G3	2		specimen not examined
G3	3	A - 90%	small spot (3-4 mm) of brownish oil-like film on bottom of top sheet; small pockets in the adhesive layer[4]; shiny[5]
G3	4	A - 95%	as for G3 - 3
B3	1	A - 95%	as for G2 - 1
B3	2	A - 95%	as for G2 - 1
B3	3	A - 90%	a couple of small white spots (3-4 mm) on top sheet; small pockets in the adhesive layer[4]; shiny[5]
B3	4	A - 95%	as for B3 - 3
G4	1	A - 80%	as for G2 - 1
G4	2	A - 80%	as for G2 - 1
G4	3		specimen not examined
G4	4	A - 75%	as for G2 - 1
B4	1	A - 75%	white streaks (talc-like), apparently at juncture of factory sheet welds; small pockets in the adhesive layer[4]; not shiny[5]
B4	2	A - 95%	small spot (5-6 mm) of brownish oil-like film on top of bottom sheet; small pockets in the adhesive layer[4]; not shiny[5]
B4	3	A - 90%	as for G2 - 1
B4	4	A - 90%	as for G2 - 1

1. These observations were made by eye on the delaminated field specimens.
2. G and B refer to "good" and "bad," respectively; see text (p. 7) for explanation.
3. This column represents the percent area of the specimen estimated to fail adhesively (A); the remainder is percent cohesive failure.
4. This note refers to pockmark-like depressions in the adhesive in areas where the failure was cohesive.
5. This note refers to the appearance of the surface of the adhesive in the pockmark areas.

Table 3. Observations on the Condition of the Field Seam Specimens[1]

Sample[2] No.	Specimen No.	Failure Mode[3]	Observations
G5	1	A - 90%	white streaks (talc-like), apparently at juncture of factory sheet welds; small pockets in the adhesive layer[4]; shiny[5]
G5	2	A - 75%	as for G5 - 1
G5	3	A - 60%	as for G2 - 1
G5	4	A - 60%	as for G2 - 1
B5	1	A - 60%	a few small areas (2-3 mm) of white deposit; small pockets in the adhesive layer[4]; shiny[5]
B5	2	A - 60%	as for G2 - 1
B5	3	A - 90%	as for G2 - 1
B5	4	A - 50%	as for G2 - 1

1. These observations were made by eye on the delaminated field specimens.
2. G and B refer to "good" and "bad," respectively; see text (p. 7) for explanation.
3. This column represents the percent area of the specimen estimated to fail adhesively (A); the remainder is percent cohesive failure.
4. This note refers to pockmark-like depressions in the adhesive in areas where the failure was cohesive.
5. This note refers to the appearance of the surface of the adhesive in the pockmark areas.

Table 4. Variables Studied for Their Effect on the Bond Strength of the Laboratory Specimens

Variable	Conditions
Cure Temperature	73 °F (23 °C) and 158 °F (70 °C); the high temperature cure included holding the specimens at room temperature for one day to minimize a risk of damaging the specimens due to rapid adhesive solvent release.
Cure Time	7 and 14 days
Surface Condition of the Rubber	cleaned and uncleaned; cleaning was done by washing with the proprietary solvent solution.

Table 5. Results of T-Peel Tests of the Laboratory Specimens[1]

Sample[2] No.	Cure Time days	Cure Temp °F (°C)	Strength, lbf/in. (kN/m) Range	Average	S.D.	COV %
C1	7	73 (23)	3.3 - 6.0 (0.58 - 1.1)	4.7 (0.82)	1.1 (0.19)	23
UC1	7	73 (23)	2.8 - 3.2 (0.49 - 0.56)	2.9 (0.51)	0.34 (0.06)	12
C2	14	73 (23)	4.2 - 6.6 (0.74 - 1.2)	5.7 (1.0)	0.95 (0.17)	17
UC2	14	73 (23)	2.5 - 4.8 (0.44 - 0.84)	3.6 (0.63)	0.94 (0.16)	27
C3	7	158 (70)	3.3 - 4.6 (0.58 - 0.81)	3.7 (0.65)	0.49 (0.09)	13
UC3	7	158 (70)	2.1 - 2.8 (0.37 - 0.49)	2.3 (0.40)	0.27 (0.05)	12
C4	14	158 (70)	3.9 - 5.8 (0.68 - 1.0)	4.4 (0.77)	0.72 (0.13)	17
UC4	14	158 (70)	1.9 - 2.1 (0.33 - 0.37)	2.1 (0.37)	0.085 (0.015)	4.2

1. Average of six measurements.
2. The C and UC refer to "cleaned" and "uncleaned" specimens, respectively.

Table 6. Modes of Failure of Laboratory Specimens During T-Peel

Specimen No.	Mode of Failure[1]	Specimen No.	Mode of Failure[1]
C1 - 1	Coh. - 100%	UC1 - 1	Coh. - 100%
C1 - 2	Coh. - 100%	UC1 - 2	Coh. - 100%
C1 - 3	Coh. - 100%	UC1 - 3	Coh. - 70% & adh. - 30%
C1 - 4	Adh. - 100%	UC1 - 4	Adh. - 100%
C1 - 5	Coh. - 20% & adh. - 20%	UC1 - 5	Adh. - 90% & coh. - 10%
C1 - 6	Adh. - 100%	UC1 - 6	Adh. - 60% & coh. - 40%
C2 - 1	Adh. - 100%	UC2 - 1	Adh. - 100%
C2 - 2	Adh. - 15% & coh. - 85%	UC2 - 2	Adh. - 100%
C2 - 3	Coh. - 100%	UC2 - 3	Adh. - 50% & coh. - 50%
C2 - 4	Adh. - 40% & coh. - 60%	UC2 - 4	Adh. - 50% & coh. - 50%
C2 - 5	Adh. - 90% & coh. - 10%	UC2 - 5	Adh. - 100%
C2 - 6	Adh. - 100%	UC2 - 6	Adh. - 100%
C3 - 1	Adh. - 100%	UC3 - 1	Adh. - 100%
C3 - 2	Adh. - 100%	UC3 - 2	Adh. - 100%
C3 - 3	Adh. - 100%	UC3 - 3	Adh. - 100%
C3 - 4	Adh. - 100%	UC3 - 4	Adh. - 100%
C3 - 5	Adh. - 100%	UC3 - 5	Adh. - 100%
C3 - 6	Adh. - 100%	UC3 - 6	Adh. - 100%
C4 - 1	Adh. - 100%	UC4 - 1	Adh. - 100%
C4 - 2	Adh. - 100%	UC4 - 2	Adh. - 100%
C4 - 3	Adh. - 100%	UC4 - 3	Adh. - 100%
C4 - 4	Adh. - 100%	UC4 - 4	Adh. - 100%
C4 - 5	Adh. - 100%	UC4 - 5	Adh. - 100%
C4 - 6	Adh. - 100%	UC4 - 6	Adh. - 100%

1. Adh. and coh. indicate adhesive and cohesive failure, respectively; the percent is the estimated area of the bond which failed in the stated mode.

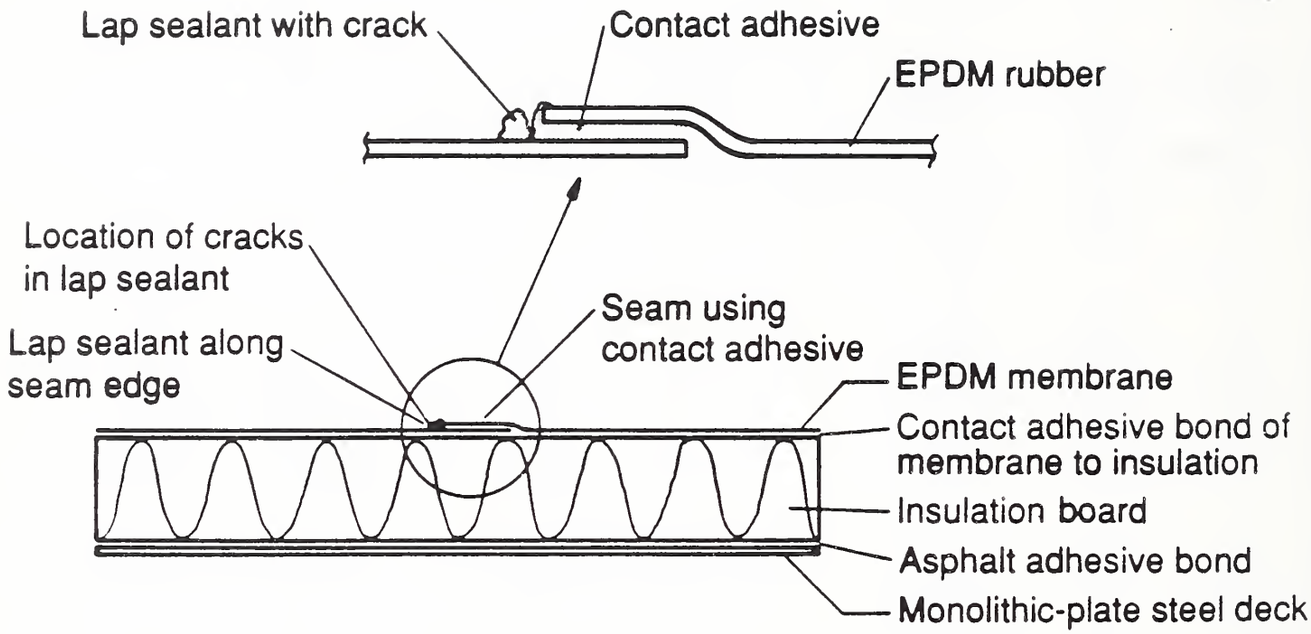

Figure 1. Cross Section of the Roof Construction Including a Seam and the Lap Sealant at the Seam Edge (Not to Scale).

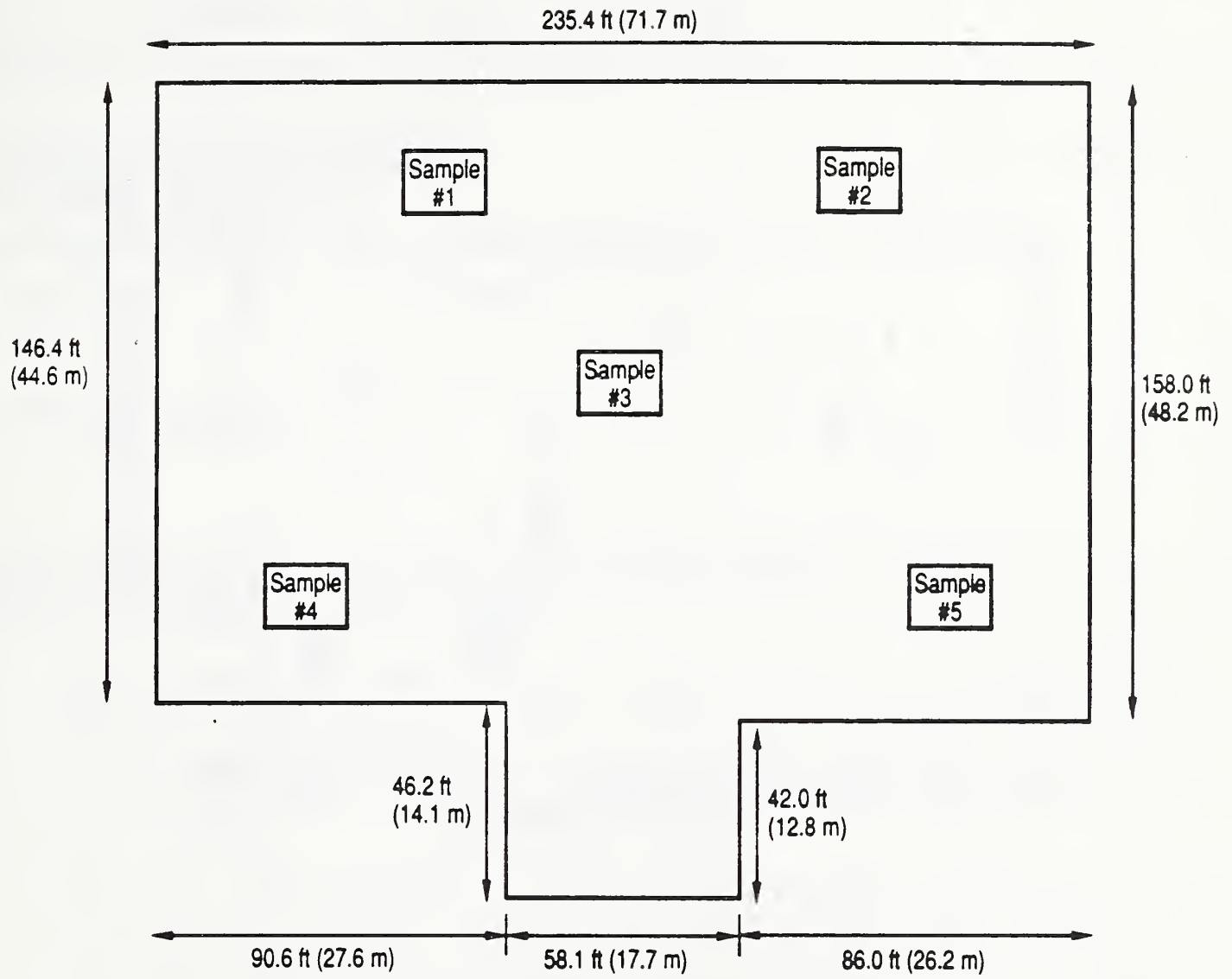

Figure 2. Plan Indicating Approximate Dimensions of the Roof and Locations of the Test Cuts.

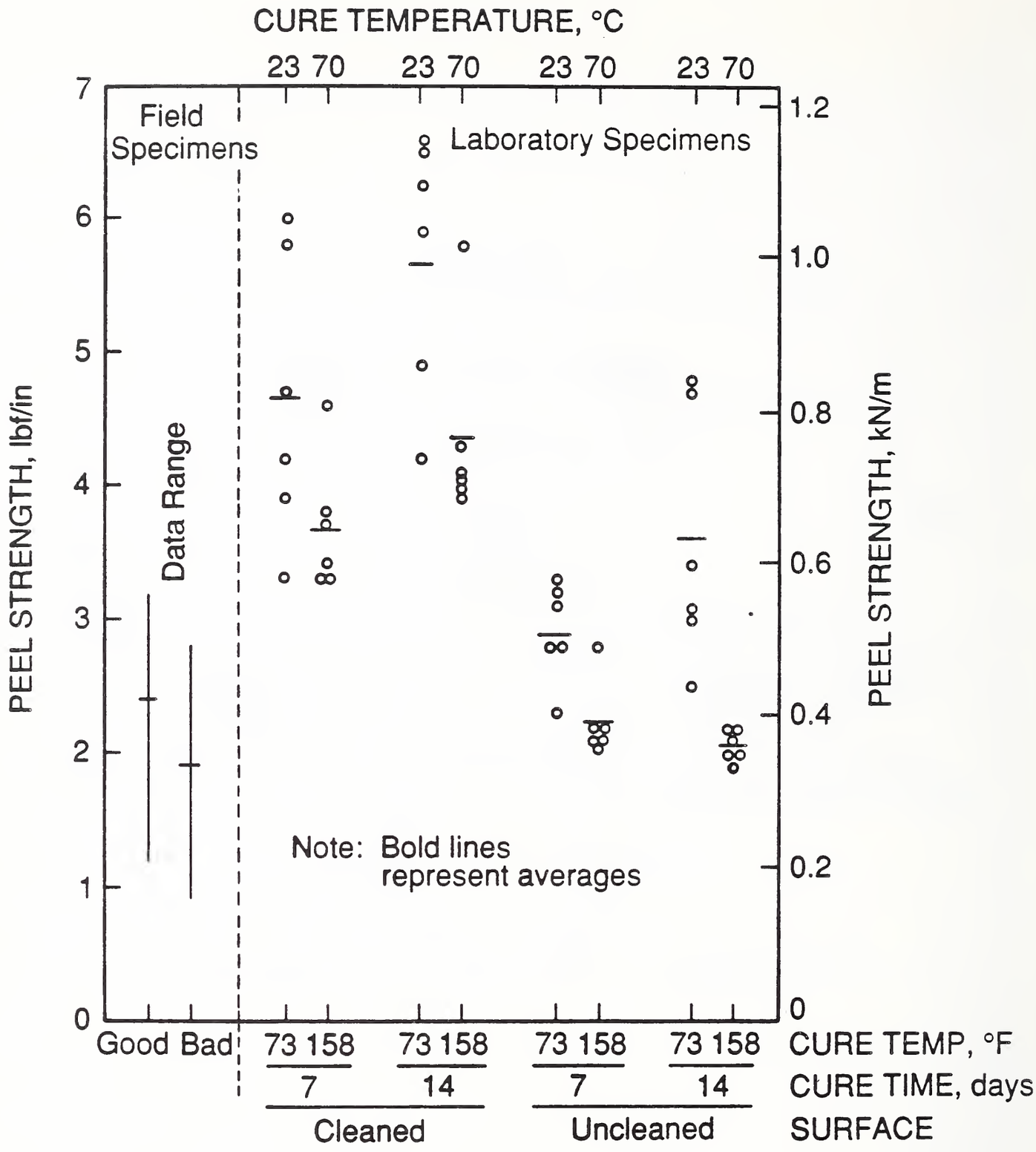

Figure 3. Results of the T-peel Tests for the Laboratory Samples in Comparison With Those of the Field Samples.

NBS-114A (REV. 2-8C)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET (See instructions)	1. PUBLICATION OR REPORT NO. NISTIR 88-3893	2. Performing Organ. Report No.	3. Publication Date NOVEMBER 1988

4. TITLE AND SUBTITLE

Report of Roof Inspection: Delamination of Adhesive - Bonded Seams at an Army Facility

5. AUTHOR(S)

Walter J. Rossiter Jr. and James F. Seiler Jr.

6. PERFORMING ORGANIZATION (If joint or other than NBS, see instructions)

NATIONAL BUREAU OF STANDARDS
U.S. DEPARTMENT OF COMMERCE
GAITHERSBURG, MD 20899

7. Contract/Grant No.

8. Type of Report & Period Covered

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (Street, City, State, ZIP)

US Army Engineer District, Baltimore
PO Box 1715
Baltimore, MD 21203-1715

10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

This document was prepared at the request of U.S. Army Engineer District, Baltimore, to provide assistance in obtaining data on the delamination of seams of an EPDM roofing system at Fort Belvoir, Virginia. The investigation was beneficial to NIST, because it provided an opportunity to characterize adhesive-bonded seams in service and to obtain data relative to NIST laboratory research on the effect of surface contamination on seam performance.

Seam specimens were taken from the roof and analyzed for peel strength and surface condition of the rubber. In addition, seams were prepared in the laboratory using the same brand name rubber/adhesive system to obtain peel-strength values for comparison with those measured for the field specimens. The results of the study indicated that the field specimens had low T-peel bond strengths in comparison to the strengths achieved by the laboratory-prepared seams. Small voids in the adhesive layer of the seams of the field seams may have contributed, in part, to the low bond strength. SEM analysis of the field-formed seams indicated the presence of a talc-like contamination on the rubber surface which may have also contributed to the low strength. In addition, SEM analysis of some laboratory specimens cleaned with the proprietary wash solution showed a talc-like contamination which was not visible to the unaided eye. Other laboratory specimens cleaned using the wash solution did not show such contamination.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

adhesive-bonding; bond strength; contamination; EPDM; field inspection; low-sloped roofing; membranes; roofs, seams, SEM analysis, surface condition

13. AVAILABILITY

- [X] Unlimited
- [ ] For Official Distribution. Do Not Release to NTIS
- [ ] Order From Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.
- [X] Order From National Technical Information Service (NTIS), Springfield, VA. 22161

14. NO. OF PRINTED PAGES

42

15. Price

$11.95

USCOMM-DC 6043-P80