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Cleaning of Aged EPDM Rubber Roofing Membrane Material for Patching: Analytical Techniques for Surface Characterization

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ABSTRACT

Proper seam formation is a critical parameter associated with the long-term performance of ethylene-propylene-diene terpolymer (EPDM) roofing systems. As time passes and the membrane weathers in service, patches and splices to an EPDM surface may be needed. A concern raised regarding the performance of EPDM roofing is whether weathering alters the rubber's surface characteristics such that successful bonding of the aged material becomes more difficult than with unaged rubber.

This report describes the results of the preliminary phase of a study to investigate surface analysis techniques for ascertaining whether the surface of aged EPDM rubber is properly cleaned before patches are bonded to it. The intent of the investigations was to develop experimental procedures applicable to EPDM rubber based on existing analytical methods. The surface analytical techniques investigated were: 1) scanning electron microscopy, 2) electron probe microanalysis, 3) Fourier transform infrared spectroscopy, and 4) contact angle (wettability) measurement.

Of the four methods, scanning electron microscopy, Fourier transform infrared spectroscopy, and contact angle measurement were found to be useful for general laboratory analysis of EPDM rubber sheets. Experimental procedures were developed for this purpose. In the case of electron probe microanalysis, it was found that the technique offered little information that could not be obtained by SEM analysis, unless it is desired to identify the elemental composition of the surface contaminants. Based on the results of the preliminary-phase investigations, it was recommended that research be continued on the application of the experimental procedures to analyze aged EPDM surfaces and to determine the effectiveness of different cleaning methods for removing surface contamination.

Key words: aged EPDM membrane; cleaning methods; contact angle; FTIR; low-sloped roofs; patching; roofing; seams; SEM; surface analysis; surface preparation methods; wettability

TABLE OF CONTENTS

	page
ABSTRACT	iii
LIST OF TABLES	vi
LIST OF FIGURES	vi
1. INTRODUCTION	1
1.1 Background	1
1.2 General Approach	1
1.3 Objective and Scope	2
2. PROGRESS OF THE STUDY	3
2.1 Task 1 -- Assessment of Repair Patches	3
2.2 Task 2 -- Methods for Characterizing EPDM Surfaces	3
3. INSPECTION OF THE FORT BENNING EPDM ROOF	9
4. SUMMARY	14
5. FUTURE WORK	15
6. ACKNOWLEDGMENTS	16
7. REFERENCES	16

LIST OF TABLES

page

Table 1. Patch and seam specimens sampled from Fort Benning 11

Table 2. Results of T-peel tests on the patch specimens . . . 13

LIST OF FIGURES

page

Figure 1. Contact angle of water and methylene iodide on EPDM rubber at 1 and 15 minutes as a function of surface contaminant coverage (expressed in terms of greylevel) 7

Figure 2. Polarity (polar force/total force) of EPDM rubber determined for 1- and 15-minute contact angles as a function of surface contaminant coverage (expressed in terms of greylevel) 8

1. INTRODUCTION

1.1 Background

The use of vulcanized ethylene-propylene-diene terpolymer (EPDM) rubber for low-sloped roofing membranes has become common in the U.S. Current estimates indicate that over 90 million square meters (one billion square feet) are being applied annually. EPDMs are non-polar, relatively inert rubbers. This makes the adhesive-bonding of sheets difficult. Proper seam formation is a critical parameter associated with long-term performance of EPDM roofing systems. A key concern expressed by the Construction Engineering Research Laboratory (CERL) is: as these membranes weather, will the rubber's surface characteristics be altered such that successful bonding of the aged material becomes more difficult than with unaged rubber. As time passes, patches and splices to an EPDM surface may be needed. For example, in the CERL study [1] of EPDM roofing at Fort Benning, it was found that some repair patches delaminated within months after formation. This may have been due to the use of improper repair materials and patching techniques. Nevertheless, it is illustrative of the need for developing a technical basis for standard methods used in preparing the surfaces of weathered EPDM.

1.2 General Approach

A two-phase study is being conducted. In the preliminary phase, investigations have been conducted on the use of surface analytical techniques for ascertaining whether the surface of aged EPDM rubber is properly cleaned before patches are bonded to it. In the main phase of the study, the surface analytical techniques, as well as short- and long-term peel tests, will be used to determine the effectiveness of different cleaning methods for removing surface contamination from aged EPDM samples. The first task performed in the preliminary phase of the study was an assessment of the performance of repair patches on aged EPDM roofing at Army facilities. This assessment focused on field techniques for cleaning aged rubber surfaces before patching, and ascertaining those which have been successful and those which have been unsatisfactory. Direct contact was made with users of EPDM roofing systems to learn of their experiences with the performance of patches in service. The major source of contacts was facility engineers at U.S. Army installations. This was complemented by contacts with roofing contractors who have made many installations of EPDM membrane roofing.

In the second task of the preliminary phase, laboratory and field tests have been performed to develop a methodology for preparing seams on aged EPDM rubber. The laboratory research is intended to provide the technical basis for determining whether the surface of aged rubber has been properly prepared before patches are applied.

The preliminary-phase laboratory study is investigating the use of surface analytical techniques such as Fourier transform infrared spectroscopy, scanning electron microscopy, and electron probe microanalysis as tools for assessing the effectiveness of procedures for preparing the surfaces of vulcanized rubbers for adhesive bonding. In addition, contact angle measurement (wettability) of rubber surfaces as a function of surface preparation is being investigated as a method for also assessing the effectiveness of the cleaning procedures.

1.3 Objective and Scope

The overall objective of the study is to develop a methodology for assuring the quality of bonded seams in weathered vulcanized-rubber membranes. A proposed series of tasks for conducting the study was suggested in the NIST report, "Suggested Research Topics for the Construction Engineering Research Laboratory (CERL) Program: Evaluation of Roofing Materials Degradation Processes" [2]. These suggested tasks were outlined as follows:

- Investigate methods for characterizing EPDM surfaces regarding properties relevant to bonding, such as scanning electron microscopy, Fourier transform infrared spectroscopy, surface energy, and other analytical surface chemistry techniques.

- Obtain samples of weathered EPDM membranes and characterize their surfaces; compare the characteristics of the weathered surfaces against those of unaged surfaces.

- Investigate techniques for preparing surfaces of weathered EPDM for bonding; characterize the surfaces of the materials before and after using the surface preparation techniques.

- Prepare seams from weathered EPDM sheets; measure the initial strength of the bonds as a function of the surface preparation procedures.

- Based on the results of the bond strength tests, conduct time-to-failure experiments on seams prepared from weathered material; compare the results to those obtained in previous NIST time-to-failure studies on unaged EPDM; develop a degradation model related to the rate of seam delamination.

- Prepare an interim progress report; publish a final report including recommendations for properly preparing the surface of weathered EPDM before bonding.

This report presents the results of the progress of the preliminary phase. The laboratory research has focused on the methods for characterizing the EPDM surfaces. In addition to the laboratory investigations, a field inspection of repair patches on an EPDM roof at Fort Benning was performed. Samples of patches were taken from the roof and T-peel tests were conducted in the laboratory. T-peel testing was selected because it has been shown to be sensitive to factors such as surface contamination that are detrimental to proper bond formation.

2. PROGRESS OF THE STUDY

2.1 Task 1 -- Assessment of Repair Patches

The results of the first task were described in the NIST Report, "Results of a Survey of the Performance of EPDM Roofing at Army Facilities" [3]. For details of the survey, the reader is referred to that report. In general, it was concluded that EPDM roofing has been performing satisfactorily on Army facilities. Few problems were reported for these facilities, but those mentioned involved seams. However, according to facility personnel, the seam problems were isolated. Because few problems with EPDM roofs had occurred, most Army facility personnel contacted had little experience with patches. Contacts with roofing contractors who have installed considerable quantities of EPDM roofing indicated that patches perform well, provided they are properly installed. The contractors generally emphasized that making patches is a difficult task, and that the rubber surface must be properly cleaned and cannot be wet. In addition, some stated that any wet materials below the area to be patched should be removed before patching.

2.2 Task 2 -- Methods for Characterizing EPDM Surfaces

This phase of the study conducted in 1989 comprised the major portion of the preliminary research activity. The intent was to provide experimental procedures in which the effectiveness of surface preparation and cleaning procedures for aged EPDM rubber may be ascertained. It is noted that the methods explored also have application to unaged rubber surfaces. As summarized in the sections that follow, the research conducted has indicated that the methods investigated are applicable to the analysis of EPDM surfaces. The surface analytical techniques discussed here are: scanning electron microscopy, electron probe microanalysis, Fourier transform infrared spectroscopy, and contact angle (wettability) measurements.

2.2.1 Scanning Electron Microscopy. Scanning electron microscopy (SEM) is a technique to characterize the morphology of surfaces, including the presence of foreign materials, from low to extremely high magnifications (e.g., x5000 to x10,000). An advantage of the SEM technique is that it provides good depth of field at high magnifications. In the present study, the "backscatter" method was used in specimen analysis. In this method, reflection of the electron beam from the specimen's surface provides an image of the surface. In general, the higher the atomic weight of the elements under the electron beam, the more the electrons are reflected. This phenomenon has utility in analyzing for the presence of surface contaminants such as talc, clays, and micas which have been applied to the surface of the EPDM rubber as release agents for processing. These materials have a high percentage of elements such as silicon which are heavier than the carbon and hydrogen that comprise a significant percentage of the EPDM sheet. Consequently, good contrast between the rubber and these surface contaminants is obtained in the surface image.

The SEM backscatter method was included in the investigation of techniques for characterizing EPDM surfaces. The objective of this work was to determine whether the SEM method could differentiate surfaces that contained varying amounts of release agent. Previous work at the NIST laboratories [4] had shown that SEM analysis had readily differentiated between rubber sheets that contained large quantities of release agent (applied in the factory) and sheets that were essentially free of release agent (due to cleaning in the laboratory). In the current investigation, cleaned surfaces of EPDM sheets were contaminated with varying amounts of release agent, according to the technique described by Martin et al. [5]. For the present investigation, the degree of contamination was described as slight, medium, and heavy. Heavy contamination was less than that present on surfaces of EPDM sheets as received from the plant.

The SEM analysis was able to differentiate between the amount of release agent on the surface for these three degrees of contamination. In particular, for the case of the sample that contained only a slight deposit of release agent, the micrograph showed areas of the rubber surface visible between particles of release agent. As the amount of release agent on the rubber surface increased, the areas of rubber surface that could be seen decreased. This differed with previous findings that showed that the surfaces of "as-received" sheets, coated with release agent, generally showed no areas of visible rubber surface.

The finding that the degree of contamination (with release agent) may be ascertained using the SEM indicated that the technique has applicability for characterizing surfaces after cleaning as a function of the cleaning method used. If some cleaning methods are more effective than others for removing surface contaminants, it is expected that the degree of removal of contaminants could be determined by SEM analysis, provided that the remaining contaminants are not so excessive that they totally cover the rubber. In these cases, particles of surface contaminants not removed by cleaning may be expected to be visible on the rubber surface. Consequently, in continuing future work, it is planned that the surfaces of weathered EPDM sheets will be characterized using SEM analysis before and after cleaning, and as a function of the method by which they are cleaned.

2.2.2 Computer-Aided Elemental Microanalysis and Mappings. Electron probe microanalysis (EPMA) is based on the physics of the interaction of energetic electrons with a solid. The interaction of the beam electrons can ionize an inner shell electron of an atom. Subsequent de-excitation can produce a characteristic X ray, whose energy identifies the elemental species present in the beam interaction volume. This technique is widely used for the characterization of elemental composition of materials on a micrometer scale. In recent years, there has been considerable development of the EPMA technique for quantitatively mapping the composition of a material's surface or immediate subsurface. This development has expanded the capabilities of EPMA in the studies of elemental distributions. Compositional maps are images in which the gray level or color scale is directly related to the concentrations of the elements in the specimen. The compositional maps not only provide the size and size distribution but also indicate the chemical specificity of a particular material. The technique does not replace the SEM technique for this purpose because the signal strength of the electron image is about 10,000 times stronger than that of the X-ray mapping, but rather supplements the SEM capability for compositional mapping.

The capability of today's EPMA technique, which can simultaneously image as many as 10 elements within five minutes, made this a potential technique for studying the presence and distribution of contaminants on the surfaces of EPDM sheets. The objective of this work was to determine the feasibility of using the EPMA technique to characterize surface contaminants. To address this question, cleaned and as-received (dusted with release agent) EPDM specimens and EPDM specimens having different levels of surface coverage of release agent were examined. Preliminary results indicated that the release agents mainly consisted of Al, K, and Si elements, with Fe and Mg elements present as minor constituents. The compositional maps of the elements clearly showed the distributions of the release agent particles and the boundaries between them and the rubber phase, particularly for the cleaned and lightly contaminated specimens.

As expected, the compositional maps of the elements overlapped each other because each element is chemically a part of the total composition of the release agent particle. Thus, for this type of contamination (i.e. one chemical type of contaminant and high coverage), the electron probe microanalysis and compositional mapping technique, although providing information on the chemical composition of the particles, did not add essential additional information to that obtained by the backscatter SEM method. Had there been two or more types of contaminants having different chemical compositions present on the roofing surface, the compositional mappings would have provided useful information about the distribution of each type of contaminant. This information cannot be obtained as readily with the SEM method. On the other hand, the SEM method is faster and has four times better resolution than the EPMA technique.

Thus, to examine for the presence of general surface contamination where it is desired to determine, for example, whether or not contaminants are removed by cleaning, the SEM method is more suitable. In cases where it is also desired to identify the composition of the contaminants, the EPMA technique may be used to obtain complementary information. Thus, in future work with EPDM sheets removed from roofs, the SEM method will be primarily employed and the EPMA technique will only be used on selected specimens where elemental composition of contaminants is of interest.

2.2.3 <u>Fourier Transform Infrared Spectroscopy</u>. The powerful combination of specific compound identification and functional group characterization have made infrared (IR) spectroscopy the most widely used analytical tool for investigating the molecular structure of chemical compounds. The development of Fourier transform infrared (FTIR) spectrometers, which increase sensitivity and reproducibility, has greatly enhanced the capability of IR as an analytical technique for surface studies. The application of FTIR for surface and interface studies has been well established. There are a number of techniques, such as diffuse reflection spectroscopy, photoacoustic spectroscopy, reflection/absorption spectroscopy, and attenuated total reflection (ATR) or multiple internal reflection (MIR) spectroscopy, which make the FTIR spectrometer particularly useful for studying the molecular structure of surfaces of solid sheets.

However, there is little information on the use of FTIR for characterizing surface chemical compositions of highly infrared absorbing carbon black-filled EPDM rubber. In this work, we are investigating the feasibility of using FTIR in combination with multiple internal reflection (FTIR-MIR or FTIR-ATR) spectroscopy for characterizing the chemical composition of the surface of EPDM sheet materials before and during service.

FTIR-MIR requires the use of an optically dense prism as an optical guide to obtain an infrared spectrum. The quality of the spectrum depends on a number of factors including the refractive indices of the prism and the sample, the number of reflections, and the closeness of contact between the sample and the prism. In this initial work, attempts were made to optimize various parameters in order to obtain spectra suitable for EPDM surface analysis. This was accomplished after considerable trial and effort. In these investigations, it was found that, because of the high infrared absorptivity of the EPDM materials, the length of the sample was critical to obtain a satisfactory FTIR-MIR spectrum; the longer the sample length, the poorer was the spectrum obtained. This finding was contrary to the conventional procedure used for less strongly absorbing materials where the longer samples provide stronger signals. The initial investigation also showed that having close contact between the EPDM specimen and the prism was important for obtaining a satisfactory FTIR-MIR spectrum.

With the development of a satisfactory experimental procedure, future work will proceed to examine the effects of ultraviolet (UV) exposure on the surface chemical compositions of aged EPDM materials, and also to investigate the effectiveness of various cleaning methods to remove surface contamination.

2.2.4 Contact Angle (Wettability). A drop of liquid on a horizontal solid surface usually exhibits a well defined contact angle. The magnitude of the contact angle is determined by several factors including the surface tension (force per unit length that opposes the increase in surface area) of the liquid, the surface free energy (energy required to form a unit area of surface) of the solid, the interactions between the liquid and solid, and the surface topography of the solid. Contact angle is a convenient and valuable measure of the wettability of a solid surface by a liquid. The wettability concept has been applied to many practical problems such as adhesion, lubrication, and surface cleanness. The objective of this work is to determine the feasibility of using contact angle measurements (wettability) to characterize the surface cleanness of EPDM materials. To address this question, as-received EPDM sheets were cleaned, and then varying amounts of release agent were applied to the cleaned surfaces. The degree of contamination was determined by Martin et al's image analysis technique [5] and expressed in terms of greyscale level. The higher the greyscale level, the greater was the quantity of release agent on the surface.

Contact angles of a polar liquid (water) and a nonpolar liquid (methylene iodide) on the EPDM sheets having different greylevels were measured using a contact angle goniometer. Figure 1 shows that the contact angles (obtained both at 1 and 15 minutes after a drop of liquid was deposited on the surface) decreased as the greyscale level increased. It may also be seen that water is more sensitive to the amount of release agent than methylene iodide. This is evident by the greater decrease of the water contact angle as the greyscale level of the surface increased.

The contact angle data were used to calculate the polarity of EPDM (Figure 2) using the harmonic-mean approach proposed by Wu [6]. As is evident in Figure 2, the cleaned specimens (greyscale level 30) had the lowest polarity which would be expected for a composite hydrocarbon material such as EPDM rubber.

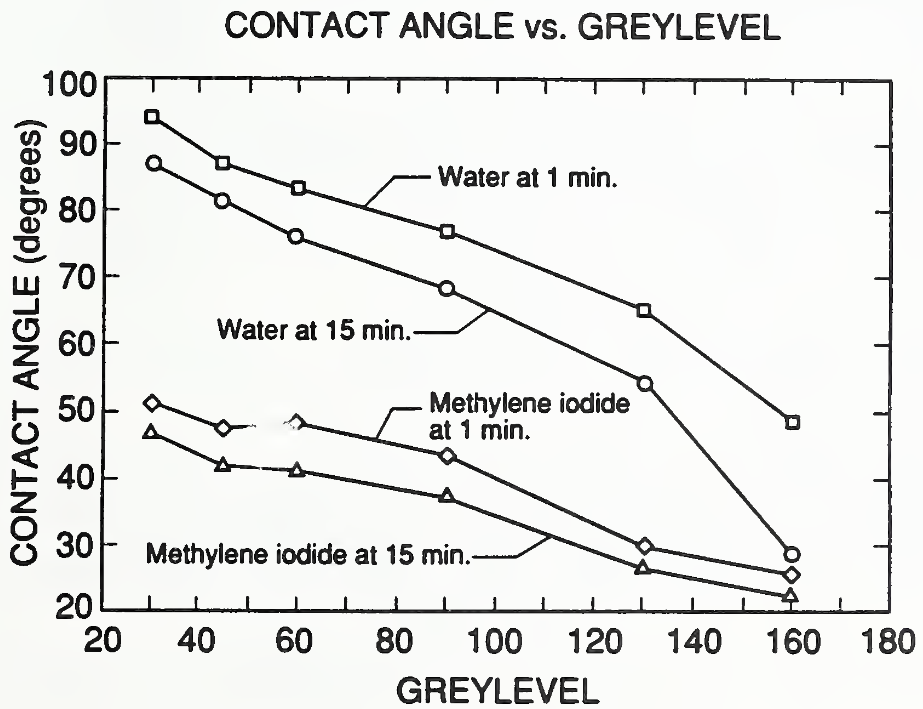

Figure 1. Contact angle of water and methylene iodide on EPDM rubber at 1 and 15 minutes as a function of surface contaminant coverage (expressed in terms of greylevel)

The preliminary results suggest that the water contact angle, the spreading rate of water on the EPDM rubber, and the polarity or the polar force component of the EPDM rubber may provide useful indicators of the extent of surface cleanness of the rubber. The implication is that the cleaner sheets will have greater contact angles and lower polarities, assuming that no hydrocarbon contaminants are present that would have the same surface characteristics as the rubber. In future work, the contact angle technique will be applied to study field samples and the effectiveness of different cleaning methods for removing contaminants and oxidation accumulated on the surface during service. Contact angles of cleaned sheets will be measured as a function of cleaning method, and related to bond strengths of "patches" made using the various cleaned sheets.

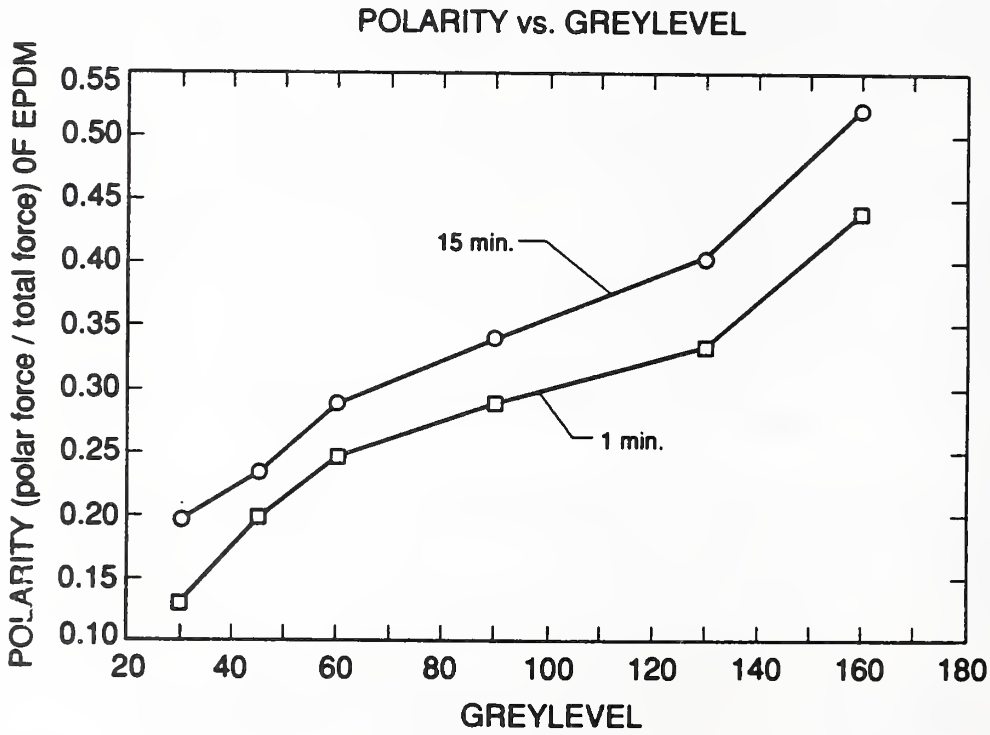

Figure 2. Polarity (polar force/total force) of EPDM rubber determined for 1- and 15-minute contact angles as a function of surface contaminant coverage (expressed in terms of greylevel)

3. INSPECTION OF THE FORT BENNING EPDM ROOF

On January 25, 1989, a visit was made to Fort Benning, GA to inspect the EPDM membrane roofing system constructed in 1980 as part of CERL's program to assess the performance of alternative membrane roofing systems. CERL staff and representatives of the membrane material manufacturer were present during the visit. The main purpose of the inspection was to observe the performance of patches made on the membrane and to obtain patch specimens for laboratory testing. A preliminary 1984 report [7] indicated that a patch made on a section of the EPDM membrane, from which a laboratory sample had been cut, was loose and needed repair. Subsequent inspections of the membrane system by CERL personnel showed that some patches were made using improper repair materials and methods [1].

During the inspection, a number of visual observations was made concerning the roofing and performance of the patches. The following is a summary of the observations made:

- The membrane was adhered to the insulation substrate and, consequently, exposed. As examined visually in place, it did not have a heavy build-up of dirt or other such contamination on the surface.

- A series of patches was visible over five locations on the roof: the four corners and the center of the building. This was consistent with the sampling pattern employed by CERL research staff.

- Patches were made with both adhesives and tapes. A manufacturer's representative indicated that both neoprene-based and butyl-based adhesives had been used for patching. The tape was described as a butyl-based product. At least one set of patches was made with what appeared to be a gray caulk or sealant.

- Some of the patches were loose and had allowed water penetration into the roof system. Where loose patches were removed, the insulation below was wet.

- It was questioned whether the neoprene adhesive was a product normally used for seam formation (i.e., a "lap adhesive"), or for adhering EPDM sheets to substrates (i.e., a "bonding adhesive"). Determination of the type was beyond the scope of the inspection.

- The gray caulk-like material used to form some patches was not a product recommended by manufacturers for fabricating seams or patches with EPDM roofing materials.

- The patches that were fabricated using the neoprene-based adhesives or caulk-like material were readily delaminated manually (even in areas where they appeared to be intact). As judged subjectively, little manual force was needed to peel these patches from the membrane. The patches made with the gray caulk-like material were so poor that specimens that were satisfactory for laboratory testing of bond strength could not be obtained.

- The patches made with the butyl-based tape appeared to be well bonded. It was difficult to delaminate them manually in the field. These patches generally appeared tight, although one patch showed a fishmouth.

- One patch sample was taken on the assumption that it was prepared using a butyl-based adhesive. (Subsequent examination of this sample in the laboratory indicated that it was a tape. Consequently, no patches made with butyl-based adhesive were tested in the laboratory.)

Five samples of some typical patches were taken along with two samples of the original seam. One of the patch samples was made on an original seam. The patches were returned to the NIST laboratories for measurement of their T-peel strengths. Table 1 describes the patches sampled. The ages of the patches were not known, and no distinguishing markings were present on the roof to indicate the times at which each of the patches had been made. Comments made by CERL staff and one manufacturer's representative indicated that the tape patches were made between 1984-1985; whereas the adhesive patches were fabricated prior to that time. Samples were taken from intact sections of the patches.

In addition to sampling the existing patches, the manufacturer's representatives made two new patch samples on the surface of the membrane (Table 1). The adhesive used was butyl-based. For one of the new patches, the original EPDM membrane surface was cleaned by scrubbing with detergent and water, and then by wiping with a solvent solution. In the second case, the new patch was made directly to the aged rubber sheet without any cleaning. Loose dirt was brushed aside using a dry clean cloth before application of the adhesive. Regardless of whether the original aged EPDM membrane surface was cleaned, the surfaces of the new EPDM rubber sheets used as patches were cleaned by wiping with the solvent solution.

In the laboratory, T-peel tests were conducted on patch specimens having dimensions 150 by 25 mm (6 by 1 in). One hundred millimeters (4 in.) of each specimen were delaminated using a universal testing machine at a rate of peel of 50 mm/min (2 in/min). Five replicate T-peel tests were performed for each of the patch samples. In case of the new patches made on the roof, the peel strengths were measured at three points in time after bond formation to determine whether changes occurred over time as the specimens remained in the laboratory at ambient temperature and humidity conditions.

Table 1. Patch and seam specimens sampled from Fort Benning

Specimen No.	Specimen Designation	Description
1.	Original Seam	The seams were prepared using a neoprene-based adhesive. Visual examination showed all original seams to be tight without any indication of deficiencies such as delaminations or fishmouths. No reports of leaks through the original seams were made by base facility staff.
2.	Original Seam	See commentary above for Specimen No. 1.
3.	Patch on Seam Made With Tape	A patch had been applied on an original seam using a butyl-based tape. In the area of the sample, the patch was tight on the seam; the seam appeared tight without deficiencies.
4.	Patch Made With Neoprene Adhesive	The patches in the area where this sample was taken appeared to be tight on the membrane surface. No signs of leaking through the patch specimen were apparent.
5.	Membrane Patch Made With Tape	A patch had been applied on the original membrane using a butyl-based tape. In the area of the sample, the patch was tight on the membrane; the patch appeared tight without deficiencies.
6.	Membrane Patch Made With Tape	See commentary above for Specimen No. 5.
7.	New Patch (cleaned)	The patch was made during the roof inspection on the surface of the original membrane using a butyl-based adhesive. The surface of the rubber was cleaned by scrubbing with an aqueous detergent solution and then with a proprietary solvent solution.
8.	New Patch (uncleaned)	The patch was made during the roof inspection on the surface of the original membrane using a butyl-based adhesive. The surface of the rubber was not cleaned before application of the adhesive.

The results of the T-peel tests are given in Table 2. Some preliminary observations are as follows:

- o The original seams (Specimen Nos. 1, 2 & 3-S[1]) had peel strengths slightly less than 0.18 kN/m (1 lbf/in). In all cases, the specimens failed adhesively during peel testing.

- o The neoprene adhesive patch (Specimen No. 4) gave a peel strength slightly less than 0.18 kN/m (1 lbf/in). This specimen failed primarily through a cohesive mode. Examination of the adhesive after specimen delamination showed it to be friable and to have mustard yellow-brown color, as if deterioration had occurred.

- o The patches (Specimen Nos. 3-P, 5 & 6) made with butyl tape had peel strengths of about 1.1 kN/m (6 lbf/in) or greater. The observed failure during peel testing was essentially adhesive.

- o The peel strength of the new patch (Specimen No. 7) prepared on the cleaned EPDM membrane surface was slightly greater than that of the new patch (Specimen No. 8) prepared on the uncleaned EPDM. However, no statistical difference was found between the values (t-test at 0.05 level). Both cleaned and uncleaned specimens failed, for the most part, adhesively and, in addition, contained numerous shiny spots indicative of areas of little or no bond.

- o Little change in the peel strengths of both the cleaned and uncleaned new patches (Specimen Nos. 7 & 8) occurred over the time period (about 12 weeks) of the tests.

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[1]For Specimen No. 3, the P and S refer to patch and seam, respectively. The sample taken from the roof consisted of a tape patch placed over an original seam in the membrane.

Table 2. Results of T-peel tests on the patch specimens

Spec. No.[d]	Peel Strength Range kN/m (lbf/in)	Av[a]	σ[b]	COV[c] %	Failure Mode[e]
1	0.18 - 0.17 (0.74 - 0.97)	0.15 (0.85)	0.02 (0.10)	12	100 adh
2	0.16 - 0.19 (0.89 - 1.1)	0.17 (0.97)	0.01 (0.074)	8	100 adh
3-P[f]	1.2 - 1.5 (6.7 - 8.7)	1.3 (7.6)	0.13 (0.72)	9	90 adh/10 coh; tape tore
3-S	0.15 - 0.23 (0.84 - 1.3)	0.17 (0.98)	0.03 (0.16)	16	100 adh
4	0.13 - 0.25 (0.76 - 1.4)	0.16 (0.93)	0.04 (.24)	26	30 adh/70 coh
5	1.0 - 1.5 (5.7 - 8.8)	1.2 (6.7)	0.21 (1.2)	18	90 adh/10 coh
6	0.88 - 1.2 (5.0 - 6.8)	1.0 (5.9)	0.13 (0.76)	13	90 adh/10 coh
7-8[g]	0.58 - 0.79 (3.3 - 4.5)	0.74 (4.2)	0.09 (0.51)	12	0 adh/70 coh; about 30% of the surface contained shiny areas indicative of little or no contact.
7-28	0.65 - 0.88 (3.7 - 5.0)	0.82 (4.7)	0.10 (0.58)	12	Same observation as 7-8
7-83	0.75 - 0.15 (4.3 - 4.8)	0.81 (4.6)	0.05 (0.26)	6	Same observation as 7-8
8-8	0.60 - 0.75 (3.4 - 4.3)	0.68 (3.9)	0.07 (0.41)	11	Same observation as 7-8
8-28	0.63 - 0.75 (3.6 - 4.3)	0.68 (3.9)	0.18 (0.29)	7	Same observation as 7-8
8-83	0.54 - 0.68 (3.1 - 3.9)	0.63 (3.6)	0.06 (0.33)	9	Same observation as 7-8

[a]Average of five measurements.
[b]Standard deviation.
[c]Coefficient of variation.
[d]The specimen corresponds to that described in Table 1.
[e]This refers to the percent of failure that was estimated through visual observation to be either adhesive (adh) or cohesive (coh).
[f]For Specimen No. 3, the P and S refer to patch and seam, respectively. The sample taken from the roof consisted of a tape patch placed over an original seam in the membrane.
[g]For Specimen Nos. 7 and 8, the number after the dash indicates the specimen age in days at which time the tests were conducted.

4. SUMMARY

The work conducted in the preliminary phase of the study focused on the investigation of surface analysis techniques for ascertaining whether the surface of aged EPDM rubber is properly cleaned before patches are bonded to it. The intent was to develop experimental procedures applicable to EPDM rubber based on existing analytical methods. The surface analytical techniques investigated were: 1) scanning electron microscopy, 2) electron probe microanalysis, 3) Fourier transform infrared spectroscopy, and 4) contact angle measurement.

Of the four methods, scanning electron microscopy, Fourier transform infrared spectroscopy, and contact angle measurement were found to be useful for general laboratory analysis of EPDM rubber sheets. Experimental procedures have been developed for this purpose. In the case of electron probe microanalysis, it was found that the technique offered little information that could not be obtained by another method. Specifically, unless it is desired to identify the elemental composition of surface contaminants, the results of electron probe microanalysis provide essentially the same information obtained by scanning electron microscopy. Moreover, the latter method is faster and gives more resolution of surface contaminants than the former technique.

Thus, based on the results of the investigations in this preliminary phase, it is recommended that the research be continued on the application of the experimental procedures to analyze aged EPDM surfaces and to determine the effectiveness of different cleaning methods for removing surface contamination. An outline of the proposed course of study is given in the next section of this report.

5. FUTURE WORK

The future work should proceed as previously proposed in the NISTIR 88-3870 [2]. In this regard, the following major tasks should be conducted:

- Obtain specimens of aged EPDM rubber membrane materials.

- Characterize the uncleaned surfaces of these materials using the experimental procedures developed during the current phase of the study. These procedures include scanning electron microscopy, Fourier transform infrared spectroscopy, and contact angle measurement.

- Clean the surfaces of the EPDM specimens using a variety of procedures. It is envisioned that cleaning methods such as detergent and water, detergent and water followed by solvent wash, solvent alone, and mechanical abrasion (either manual or powered) would be included in the study.

- Re-analyze the surfaces using the surface analysis procedures, thus characterizing the effectiveness of the cleaning procedure for removing surface contaminants.

- Prepare "patch specimens" in the laboratory using aged EPDM that has been cleaned using the cleaning methods under investigation; measure the bond strength and creep rupture resistance of "patches." Relate the results of these tests to the surface cleanness of the aged EPDM as determined by the surface analytical procedures.

- Prepare a final report which will include recommendations on procedures for cleaning the surface of aged EPDM before application of patches.

6. ACKNOWLEDGMENTS

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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.)

Proper seam formation is a critical parameter associated with long-term performance of ethylene-propylene-diene terpolymer (EPDM) roofing systems. As time passes and the membrane weathers in service, patches and splices to an EPDM surface may be needed. A concern raised regarding the performance of EPDM roofing is whether weathering alters the rubber's surface characteristics such that successful bonding of the aged material becomes more difficult than with unaged rubber.

This report describes the results of the preliminary phase of a study to investigate surface analysis techniques for ascertaining whether the surface of aged EPDM rubber is properly cleaned before patches are bonded to it. The intent of the investigations was to develop experimental procedures applicable to EPDM rubber based on existing analytical methods. The surface analytical techniques investigated were: 1) scanning electron microscopy, 2) electron probe microanalysis, 3) Fourier transform infrared spectroscopy, and 4) contact angle measurement.

Of the four methods, scanning electron microscopy, Fourier transform infrared spectroscopy, and contact angle measurement were found to be useful for general laboratory analysis of EPDM rubber sheets. Experimental procedures were developed for this purpose. In the case of electron probe microanalysis, it was found that the technique offered little information that could not be obtained by SEM analysis, unless it is desired to identify the elemental composition of the surface contaminants. Thus, based on the results of the investigations in the preliminary phase, it was recommended that the research be continued on the application of the experimental procedures to analyze aged EPDM surfaces and to determine the effectiveness of different cleaning methods for removing surface contamination.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)
aged EPDM membrane; cleaning methods; contact angle; FTIR; low-sloped roofs; patching; roofing; scanning electron microscopy; seams; surface analysis; surface preparation methods; wettability

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