PULSE-ECHO ULTRASONIC EVALUATION OF THE INTEGRITY OF SEAMS OF SINGLE-PLY ROOF MEMBRANES

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This paper summarizes results of a study to develop an ultrasonic pulse-echo method for evaluating the integrity of seams of single-ply roofing membranes. A prototype pulse-echo apparatus (the field scanner), which was designed to scan across seams of roofs while maintaining acoustic coupling to the seam surface, was developed. Results of initial laboratory investigations showed that voids in seams could be distinguished from well-bonded sections using the intensity of the echo from the adhesive layer. This echo was relatively weak for well-bonded seams, and relatively strong for voids. Seams of existing EPDM single-ply membranes were examined by roof-top scanning to evaluate the performance of the field scanner in practice.

The findings indicated that the field scanner was sensitive to detecting micro-cavities that could be created in the adhesive layer at the time of seam fabrication, thus resulting in "false positive" readings. The formation of the micro-cavities were attributed to the temperature-induced volatilization of residual solvent remaining in the adhesive layer after its application on the membrane material. It was concluded that, at least in its present form, the field scanner had limited applicability to resolving micro-cavities from voids and delaminations in solvent- adhesive seams. It may be applicable to seams formed using other techniques.

KEYWORDS

Adhesive-bonding, EPDM rubber, field inspection, membranes, nondestructive testing, pulse-echo method, roofing, seams, ultrasonics, voids.

INTRODUCTION

A critical parameter associated with the performance of single-ply roofing membranes is the waterproofing integrity of field seams. The seams must be properly prepared and remain watertight over the service life of the roof. Field experience based on NRCA's Project Pinpoint surveys[1] has shown unsatisfactory seam performance to be the major problem reported for single-ply membranes. Consequently, many studies have been conducted to investigate the factors affecting seam performance.[2-8] These studies have addressed both short-term bond strength tests and long-term creep rupture tests. However, few investigations have been performed regarding inspection methods for assessing the condition of in-place seams. In particular, nondestructive evaluation (NDE) inspection procedures are not presently available to determine that bond formation is complete in all sections of the seams.[9]

An inspection is normally carried out visually by an inspector who checks for large bubbles, fishmouths or lack of bond at the edge of the seam. The inspector may also probe along the seam edge by using a blunt instrument to judge subjectively the extent of the bond. Visual inspection does not generally detect voids or delaminated areas which can be hidden in the interior portions of seams and which may lead to seam failure. Voids are usually incorporated in seams at the time of fabrication because of the lack of complete adhesive application; delaminations usually occur through disbonding of the seam after fabrication.[10]

Destructive methods, primarily peel tests, have been proposed as an indicator of whether a field seam has been properly prepared.[6,11] A short-coming of such testing is that it evaluates only small portions of the entire seam. A complementary NDE method to evaluate the entire seam area is needed to assure the completeness of adhesive application and bond formation.[9] An additional benefit of an NDE method would be the availability of a tool to investigate seam delamination during service, in the event that such a problem were to occur.

Preliminary investigations have been conducted at the National Institute of Standards and Technology (NIST) on the feasibility of NDE methods to detect voids in adhesive-bonded seams of single-ply membranes.[9,10,12] It was shown that the pulse-echo ultrasonic method, using a wheel transducer that could scan continuously along the length of a seam specimen, was successful in detecting voids intentionally incorporated in the adhesive layer. Thus, it was recommended that future study be directed at further investigations of the pulse-echo ultrasonic method, and in particular, towards the development of equipment for field use.[9]

Ultrasonic NDE methods have been applied extensively to detect voids or delaminations between sheet materials.[13-17] Most of these applications have been on adhesive-joints of rigid materials such as metals. A few have dealt with synthetic flexible membrane materials including geomembranes.[18] A benefit of the extensive experience with ultrasonic NDE methods was the availability of equipment that could provide the basis for the development of an apparatus for the field scanning of seams of single-ply membranes.

A two-phase study was conducted to investigate further the use of the pulse-echo ultrasonic method for detecting, in the field, voids and delaminations in seams of single-ply roofing membranes. A prototype apparatus (herein called the field scanner) incorporating pulse-echo equipment was developed for inspecting seams in service. The prototype was investigated in the laboratory to evaluate variables such as: 1) sensitivity and practical limitations in detecting voids, 2) optimal operating conditions, and 3) influence of the properties of the seam (e.g., adhesive thickness) on void detection.

In addition, the prototype was evaluated in the field. Adhesive-bonded seams of existing single-ply membranes, as well as those of roofs under construction, were inspected. Based on the results of both the laboratory and field inspections, recommendations are made on the use of the pulse-echo ultrasonic method for evaluating the integrity of seams of single-ply membranes.

PRINCIPLE OF THE METHOD AND FIELD SCANNER APPARATUS

Principle of the Pulse-Echo Ultrasonic Method

The principle of the pulse-echo ultrasonic method for detecting voids in single-ply roof membranes has been previously described.[10] Sound waves with a frequency above the audible range are induced in a seam by a piezoelectric transducer and waves reflected off discontinuities (e.g., voids and delaminations) and from interfaces (e.g., between membrane and thermal insulation or air) are recorded. A completely bonded seam would produce an echo signal that would pass through two membrane layers. If the region of the seam being inspected was unbounded, the echo would only pass through one layer. Therefore, the echo signal would require twice the time to pass through a bonded region as through an unbonded region of a seam.

Coupling Method for the Field Scanner

Reliable acoustic coupling of the transducer with the top surface of the seam of a single-ply membrane was a critical requirement in the development of the NDE field scanner.[9,10] A main concern was that field conditions such as unevenness of the roof membrane surface could hamper effective coupling. Another consideration was the availability of sufficient amounts of water (a normal couplant) on the roof of a building under construction.

Figure 1 shows a schematic diagram of the technique selected to provide coupling of the transducer to the membrane surface using the prototype field scanner.[20] An immersible transducer was placed in a plastic cylindrical container (subsequently referred to here as the transducer holder) that was filled with water. The bottom of the holder is a flexible rubber (urethane) diaphragm, having a thickness of 0.03 in. (0.8 mm). A flexible bottom was selected to provide a conformable surface and, thus, help to keep continuous contact of the holder with irregular (i.e., uneven or non-planar) surfaces of the field seam specimens. When the holder was filled with water, the diaphragm bulged slightly, which also helped to maintain contact with the surfaces of test specimens. The rubber of the diaphragm was selected to have an acoustic impedance similar to that of water to minimize reflection of the ultrasonic echo at water-diaphragm interfaces,[20] and also to have minimum attenuation of the signal passing through the diaphragm.

In the laboratory, the transducer holder was set directly on a seam test specimen. Alternatively, for field applications, the transducer holder was mounted as part of the pulse-echo field scanner as described in the next section.

Description of the Field Scanner

Figure 2 shows a schematic of the field scanner. The basic components, typical of pulse-echo ultrasonic test equipment, are: the transducer (here incorporated in the holder), the pulser- receiver unit and a couplant reservoir. The components are mounted on a wheeled luggage carrier to allow continuous scanning and maneuverability along lengths of seams in the field.

The couplant, whose flow can be adjusted using a plastic valve on the reservoir, is supplied to the seams in front of the transducer holder through a paint-brush pad. This brush pad is intended to drag across the seam surface during scanning to increase wetting of the surface with the couplant and to wipe away coarse particles.

The pulser-receiver unit has an oscilloscope for detecting echoes and is compatible with transducers with nominal frequencies ranging from 1 to 10 MHz. The unit contains a rechargeable battery that can operate for eight hours. It can also be directly connected to normal AC house lines. The operating controls include coarse- and fine-gain switches for adjusting the intensity of received echoes displayed on the oscilloscope, and were used to measure echo intensities.

The laboratory evaluation provided guidance on the optimum conditions for use of the field scanner.[20] For example, 5-MHz focusing and non-focusing transducers were selected for use in the field. Although not without limitations, encouraging evidence was obtained in the laboratory that the field scanner may be used for inspections of single-ply seams in service.

Echo Measurement Procedure

Before testing a seam specimen, the pulser-receiver was turned on with the transducer holder suspended in air. Two echoes with comparable intensity were observed on the oscilloscope. The first echo was associated with the interior water-diaphragm interface, whereas the second was due to the exterior air- diaphragm interface. When the transducer holder was acoustically coupled to a seam specimen, these echoes were still visible, but the second echo markedly decreased in intensity. In this case, the second echo was due to the interface between the diaphragm and the top surface of the seam. The decrease in intensity of the second echo provided a qualitative measure of coupling. Whenever coupling was not achieved, the second echo was generally as intense as the first echo associated with the interior water-diaphragm interface.

Figure 3 shows a schematic illustration of the echoes typically observed for seam specimens. Figure 3a is a well-bonded seam which, for purposes of this paper, is one without voids or delaminations. Figure 3b depicts the echo pattern for a seam that contains a void (in this case due to a lack of adhesive) in the adhesive layer. In these figures, the X-axis represents relative time and the Y-axis represents the relative intensity of the echo. For both the well-bonded seam and the void-containing specimen, four echoes are usually observed, representing four interfaces from which the ultrasonic pulses are reflected. These four echoes have been designated as follows (Figures 1 and 3):

- ED1—Echo from the water-diaphragm interface.
- ED2—Echo from the diaphragm-specimen interface, (or in the absence of a specimen, the air-diaphragm interface).

- EM1—Echo from the adhesive layer* in the seam, (or for a single sheet of membrane material, the echo from the interface of the sheet and its substrate).
- EM2—Echo from the seam specimen-substrate interface.

In Figure 3, note the difference between the echo pattern for a well-bonded specimen and for a specimen with a void. In the former case, the intensity of EM1 is relatively low and comparable to that of EM2. When a void is present, the relative intensity of EM1 greatly increases and that of EM2 becomes extremely weaker. In this case, the ultrasonic pulse is mainly reflected at the void.

A quantitative measure of peak intensity was needed to analyze and compare the various echo patterns obtained as a function of the variables investigated during the study. To this end, the intensity of an individual echo was increased using the gain controls of the pulse-receiver unit until the peak height reached, but did not exceed, 100 percent of the relative intensity scale on the oscilloscope. The dB (absolute) value at which maximum peak height was attained was recorded as the intensity of the echo. This technique was used in lieu of equipment that could directly measure echo intensity. Using this technique, the echo intensities were recorded as negative dB values, indicating that the original signals were amplified to reach maximum intensity. The larger the dB value, the weaker was the intensity of the echo.

INVESTIGATIONS OF FIELD SEAM SAMPLES

The Initial Field Investigation

As the laboratory investigation of the field scanner was nearing completion, an opportunity arose to inspect seams of a low-sloped EPDM roof in the greater Washington, D.C. area. The roof was scheduled for replacement due to problems not associated with the seams. They had reportedly been performing satisfactorily without excessive maintenance.

As an initial investigation, the inspection was conducted qualitatively on the roof without recording echo intensities. However, unexpectedly (in view of the report that the seams were performing satisfactorily), the intensity of the EM1 echoes observed as the field scanner was pushed over section of seams was relatively strong. It was generally comparable to that obtained for the single sheet of the membrane (when measured away from the seam), and indicative of an echo associated with a void or delamination. This observation was not limited to isolated sections of the seam, but consistent across all areas that were inspected.

A small section of the seams was cut from the membrane, delaminated from the insulation below, reset on the roof, and re-inspected using the field scanner. The observed echoes were again indicative of voids or delaminations in the specimen. The seam specimen was manually delaminated to expose an adhesive layer that was bonded and without voids. The finding was called a "false positive." Another seam specimen was cut from the roof and returned to the laboratory for further analysis in an attempt to explain the observed false positive. As will be discussed, the findings of the initial field inspection were not an isolated event—other roofs gave false positives.

Results and Discussions of the Roof Investigations

The Roofs Investigated—The number of field and related investigations was limited to five roofs, including those of the initial investigations. A 5-MHz focusing transducer was used for these investigations. Table 1 summarizes the five roofs. In each case, the membrane used on the roof was a non-reinforced EPDM rubber with a nominal thickness of 0.06 in. (1.5 mm).

Their seams were all reportedly performing satisfactorily by the individuals inviting the NIST research staff to perform the NDE inspections. With the exception of roof No. 2, all were inspected on site using the field scanner. In this case, a seam specimen was sent to the NIST laboratories for analysis with the field scanner.

Roof No. 3 was under construction at the time of the NDE inspection. As a result, the seams inspected ranged from a few minutes to nine days in age. From observations of the seam fabrication process, the workmanship appeared to be satisfactory. It is noted that the seams constructed during the inspection of roof No. 3 were fabricated shortly after noon on a warm, sunny day when the black EPDM membrane material was noticeably warm to the touch. No information was available concerning the time of year or day when the other roofs were constructed.

For roof Nos. 1, 2 and 3, seams with a considerable amount of the single sheet of the membrane material intact were sampled. This made it possible to test the seams in the laboratory under controlled conditions, and to examine them after delamination to ascertain whether voids or delaminations were present. Also, the single sheet of the membrane could be examined, as a control, using the field scanner when the specimen was placed loose-laid on a substrate.

Results of Laboratory Tests of Seam Samples—Figure 4 presents the results of the laboratory inspections of the seam specimens cut from roof Nos. 1, 2 and 3. There was overlap of the intensities of the EM1 echo for the seam specimen with those of the single sheet. For each roof, a number of echoes for the seam specimens were less than those of the single sheets. On the other hand, no echoes due to the seam were greater than those of the single sheets.

At first glance, the results in Figure 4 imply that the seam specimens contained some voids or some delaminations. In some cases, not only were the EM1 echoes of the seam specimens the same as those of the single sheet, but the intensity values were comparable to those recorded for voids in the laboratory. For example, voids were shown to have EM1 echoes ranging from −46 to −62 dB.[20] However, delamination of the seam specimens from roof Nos. 1, 2 and 3 showed the adhesive layers to be free of voids or delamination, as was found for roof No. 1 at the time of the initial field investigation.

Results of Field Inspections Using the Scanner—Figure 5 gives the results of the measurements of roof Nos 3, 4 and 5 using the field scanner. In the case of roof No. 3, which was under construction, observations were made of seams of varying ages.

In the case of roof No. 4, a duplicate set of measurements was made with a 5 MHz, non-focusing transducer to determine whether a transducer effect was apparent. For all com-

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* *The upper and lower surface of the adhesive layer in the seam did not show two corresponding echoes. Instead, only one signal having one peak was observed on the oscilloscope. In the following discussion this reflected signal is named "EM1" and is treated as an echo from the adhesive layer.*

parative sets of measurements, there was considerable overlap of the intensities of the EM1 echoes of the seam specimens with those of the single sheet. As was the case of the laboratory tests, the measurements could be interpreted as indicating some voids and delaminations in the seams. The presence of such defects could not be confirmed or denied in the field because cuts were not made except for one seam of roof No. 3. However, there was no reason to believe that voids or delaminations were extensive throughout the seams. It was difficult to believe that the seams would have performed satisfactorily for up to four years (e.g., roof No. 4), if they consisted mainly of voids and delaminations. Consequently, the results in Figure 5 were also considered to be generally false positive.

Discussion of the Results—As part of the investigations to explain the false positives observed in the field inspections, peel tests were conducted on the specimen sampled from roof No. 3. It had been cured in the laboratory for four weeks at ambient conditions before testing. The average strength and standard deviation were 4.7 and 0.7 lbf/in. (0.82 and 0.1 kN/m), respectively.

The results were consistent with the visual observations that the seam was bonded without voids or delaminations in the adhesive. The average peel strength was comparable to those obtained from other field specimens,[6,21] but approximately one half of that normally obtained in the laboratory. The mode of failure during peel was cohesive, which was typical of seam specimens having butyl-based adhesive applied to cleaned rubber surfaces.[7] Sections of the delaminated seams were examined using scanning electron microscopy (SEM). A significant feature observed in the SEM analysis was the presence of micro-cavities** in the adhesive layer. A typical micrograph illustrating the micro-cavities is shown in Figure 6. The diameters of the micro-cavities ranged from about 0.001 to 0.002 in. (0.03 to 0.05 mm). They are flaws in the adhesive layer which would be anticipated to cause lower-than-expected peel strengths as compared to specimens without micro-cavities present.

Specimens from roof Nos. 1 and 2 were also subjected to SEM analysis. Micro-cavities were also visible in the micrographs.

The presence of micro-cavities in the adhesive layers could explain the false positives obtained during the NDE inspections using the field scanner. The acoustic impedance of the neoprene- or butyl-based adhesive materials is similar to that of the EPDM membrane material.[20,22] This similarity is the reason why ultrasonic pulses are not reflected greatly at the membrane-adhesive interfaces.[23] But the porosity of a material can make its acoustic impedance significantly lower than that of the bulk of the material.[23] This suggests that, when the adhesive layer in the seam is porous, ultrasonic pulses should be reflected at the adhesive-membrane interface because their impedances are no longer similar.

It was hypothesized that the presence of the micro-cavities was due to the temperature-induced volatilization of residual solvent remaining in the adhesive layer after its application on the roofing membrane material.[20] In past studies,[9,10,20] well-bonded seam specimens fabricated in the laboratory transmitted the ultrasonic pulse, while some of those inspected in the field did not. This was attributed to differences in the environmental conditions at the two locations. Notably, in the field, solar radiation on the black EPDM rubber often heats it to temperatures well above ambient. This would promote volatilization of residual solvent in the adhesive layer. In contrast, in the laboratory, where the membrane never experiences solar heating, residual solvent in the adhesive layer could slowly evaporate without producing voids.

INVESTIGATIONS ON MICRO-CAVITY FORMATION

Laboratory investigations were conducted to test the hypothesis presented in the previous section and to explain the formation of micro-cavities observed in the adhesive layers of the field seams. The hypothesis is that they were caused by solar-heating induced volatilization of the residual solvent remaining in the adhesive layer at the time of bond formation.

Laboratory Experiment on Elevated Cure Temperature

Temperatures of black EPDM membranes measured in the field have been as high as 160°F (71°C) or more, depending on the locale, and time of day and year.[10] In the present experiment, seam specimens were prepared at room temperature and split into two groups. The control group was cured at ambient room temperature conditions, whereas the second group (referred to as "heat-cured") was cured in a laboratory oven at 158°F (70°C). Measurements of ultrasonic echo intensities, and adhesive thickness and mass were recorded as a function of cure time.[25] In the case of the heat-cured specimens, they were removed from the oven while the measurements were made. The results for the heat-cured specimens were compared with those for the control specimens.

Specimen Preparation and Measurement—Table 2 describes the 10 seam specimens included in the experiment and the conditions under which they were cured. The size of the specimens was 5 X 6 in. (130 X 150 mm) with a 4-inch-wide (100 mm) seam centered parallel to the long dimension. The rubber used for specimen preparation was a commercially available, non-reinforced, EPDM roofing membrane sheet having a nominal thickness of 0.060 in. (1.5 mm). The adhesives used for bonding were commercially available, butyl-based and neoprene-based products. The procedures for surface cleaning of the rubber sheets, application of the adhesives, and formation of the seam have been previously described.[20] Open times typical of those normally used in the application of adhesives to EPDM roof membrane sheets were employed in these experiments.

Results for the Specimens with Neoprene-Based Adhesive—Figure 7 gives the results of the echo intensity, adhesive thickness and adhesive mass measurements made for the seam samples having neoprene-based adhesives. With the exception of cure condition No. 4, all measurements were first made after the specimens had cured for a minimum of 20 minutes at room temperature. For each of the three variables, the initial measurements showed little variation. In the case of the pulse-echo measurements, the initial EM1 intensity of approximately −80 dB was comparable to that obtained for a well-bonded specimen.[20]

Over time, the specimen that was cured at room temperature (condition No. 1) continued to display an EM1 echo

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***The word "micro-cavity" is used to express the size of very small voids in the adhesive layers in the seam specimens. Note that the intent of the present study is to detect the absence of adhesives in the seam specimens. Such defects are much larger in size than the micro-cavities.*

intensity typical of a well-bonded specimen, although a slight increase above the initial value was observed. In contrast, the specimens subjected to a heat cure within a day of seam formation (condition Nos. 2 and 3) showed a significant jump in the intensity of the EM1 echo. The resultant value of -55 to -60 dB was typical of an echo from a void or delamination.[20]

The specimens showed a loss of mass (due to solvent evaporation) for at least the first week of cure (Figure 7). The loss was accelerated by the heat cure. The specimens contained residual volatile solvents when they were subjected to heat-cure condition Nos. 2 and 3, which produced the marked increase in intensity of the EM1 echo. It is evident in Figure 7 that the mass loss from the specimens approached a plateau as the cure time neared four weeks. It was assumed that the majority of the residual solvent had evaporated from the specimens over this period of time. Thus, it was expected that a specimen subjected to a heat cure after most of the solvent evaporated would not show an increase in the intensity of the EM1 echo upon heating. Figure 7 includes the EM1 echo intensities for the specimen cured at condition No. 4. The EM1 echo was essentially unchanged by the heat-cure, which was not applied until after a three-week cure at room temperature.

Figure 7 also includes data on adhesive thickness for the cure conditions Nos. 1, 2 and 3. When cured at room temperature, the adhesive thickness decreased as the adhesive lost mass. However, during the times of heat curing, the adhesive layers increased in thickness. This was most noticeable for cure condition No. 2, where the increase in thickness was about 60 percent of the original thickness. This percent increase could not be explained on the basis of normal thermal expansion of the rubber materials upon heating. It was assumed that the expansion was due to formation of micro-cavities (cellular structures) in the adhesive layers.

At the end of the experiment, the specimens were delaminated manually and visually inspected. Microscopic analysis was not needed to see micro-cavities in the adhesive layers of the specimens that showed strong EM1 echoes after heat curing (cure conditions Nos. 2 and 3). In contrast, specimens that displayed weak EM1 echoes showed no micro-cavities. Scanning electron microscopy was conducted, but micro-cavities were only observed in those specimens cured under conditions Nos. 2 and 3. Figure 8 shows a SEM photomicrograph of some of the micro-cavities generated in the experiment.

In summary, the data obtained from the heat cure experiment on seam specimens with neoprene-based adhesives were considered to be consistent with the hypothesis set forth on the mechanism of micro-cavity formation in adhesive layers. It was also shown that, if an adhesive layer cures long enough at a relatively low temperature, it will not be affected by subsequent exposure to elevated temperatures.

Results for the Specimens with Butyl-Based Adhesive—Figure 9 gives the results of the echo intensity, adhesive thickness and adhesive mass measurements made for the seam samples having butyl-based adhesives. The results of the EM1 echo intensity measurements for cure conditions Nos. 1, 2, 3 and 4 may be compared with those for the neoprene-based adhesive specimens given in Figure 7. Unlike the results for neoprene-based adhesives, the heat-cure (conditions Nos. 2 and 3) of the butyl- based adhesives produced no significant changes in the measured properties of the specimens.

Most notably, it was found for the butyl-based adhesives that no sharp increases in the intensity of the EM1 echo were observed after the heat curing. The implication of these pulse-echo measurements was that micro-cavities were not generated in the adhesive layers. This was found even though the specimens contained residual solvent, as demonstrated by the adhesive mass loss observed over time (Figure 9). Also, consistent with the lack of micro-cavity formation was the observation that no significant increases in the adhesive layer thickness were observed when the specimens were heat-cured. At the end of the four-week cure period, delamination followed by visual and microscopic examination of the exposed adhesive layer showed no evidence of micro-cavities.

The results for cure condition Nos. 1, 2 and 3 were neither consistent with the observed false positives observed in the field NDE inspections of butyl-based seams nor the hypothesis for the formation of micro-cavities as the explanation of the false positives. In re-thinking the experiment conducted on the butyl-based adhesives, it was considered that a major difference existed between the laboratory procedure and an adhesive application process that may occur in the field. In the field, the EPDM rubber may sometimes be hot (due to solar radiation) when the adhesive is applied. In the laboratory experiments, the adhesive application was performed at room temperature and the specimens remained at that temperature for a minimum of 20 minutes before heating. Consequently, an additional experiment (cure condition Nos. 5 and 6) was conducted on specimens with butyl-based adhesives. The rubber was either at room temperature or preheated when the adhesive was applied. After seam formation, in both cases, the specimens were immediately placed in an oven for curing at the elevated temperature.

The results of the pulse-echo measurements for the cure condition Nos. 5 and 6 are given in Figure 9 along with the data previously discussed. It was found that, when the adhesive was applied to the rubber at room temperature, the immediate determination of the intensity of the EM1 echo was akin to that of a well-bonded specimen. However, after 20 minutes of heat-cure, the EM1 intensity displayed a sharp increase and was typical of a void in the adhesive layer. In the case of cure condition No. 6, the intensity of the EM1 echo measured immediately upon seam formation was typical of that produced by a void in the adhesive layer. The observations for cure condition Nos. 5 and 6 were consistent with the formation of micro-cavities due to expansion of residual solvent in the adhesive layer. SEM analyses of the delaminated specimens at the end of the cure period showed micro-cavities to be present.

In summary, the findings for the elevated temperature cure of the butyl-based experiments indicated that micro-cavities could be generated if the seam specimens were heat-cured immediately after formation. This was considered to be consistent with the original hypothesis on the mechanism of micro-cavity formation. The hypothesis did not address the possibility that maintaining the newly-formed specimens at a room temperature for a short period of time (e.g., 20 minutes) before application of the heat- cure would prevent micro-cavity formation. Reasons why cure condition Nos. 5 and 6 produced voids, whereas cure condition Nos. 2 and 3 did not, were not investigated. It may have been associated with parameters such as volatility of the solvent, its solubility in the adhesive, factors affecting nucleation, and the cure of the butyl-based adhesive which is known to increase in strength in time, particularly in the first few hours immediately after application.[6]

Field Experiment on Elevated Cure Temperature

As a test of the hypothesis on the formation of micro-cavities, seam specimens were fabricated outdoors on a warm, sunny day in June 1990 at the NIST campus in Gaithersburg, Md. The intensity of the EM1 echoes for the specimens were recorded as a function of time.

Specimen Preparation and Measurements—Seam specimens were prepared, as in the laboratory experiment, using EPDM rubber membrane sheets and either butyl-based or neoprene-based adhesives. The two specimens were approximately 4 by 2 feet (1.2 by 0.6 m) with a 4-inch-wide (100 mm) seam at the center parallel to the long dimension. The EPDM sheets were placed on a 2-inch-thick (50 mm) expanded polystyrene insulation board 30 minutes before the adhesive was applied to their surfaces. After an adequate open time (about 15-20 minutes), the sheets were joined to form the seams. The specimens were kept in place on the insulation board during subsequent measurements. The temperatures of ambient air and the surface of the EPDM sheets were measured using thermocouples. The intensity of the EM1 echoes were measured using the pulse echo equipment placed stationery on three marked locations on each seam specimen. For each point in time, the reported value of the EM1 intensity is the average of the three measurements. In all cases, the coefficient of variation was 10 percent of less.

Results of the Field Experiment—Seam specimens having the neoprene-based and the butyl-based adhesives were formed at 11:00 a.m. and 11:30 a.m., respectively. The results of the temperature measurements (Figures 10 and 11) indicated that both types of specimens cured under the same conditions. In both cases, the surface temperatures of the rubber sheets were above 140°F (60°C) at the time of seam formation and remained above that level for more than three hours. They reached a maximum of about 150°F (65°C) shortly after noon.

The average values of the EM1 echo intensities as a function of time are given in Figures 10 and 11 for the neoprene-based and butyl-based adhesive specimens, respectively. In the case of the neoprene-based specimens, the initial intensity of the EM1 echo was −80 dB. This value increased to −56 dB in two hours and then remained constant. The butyl-based adhesive specimen showed the same trend as the neoprene-based specimen, but to a lesser degree. In the former case, the intensity at the time of seam formation was −72 dB. It increased to about −57 dB in two hours, and displayed a slight increase over the remaining time of the experiment.

For each type of adhesive, the initial values of the EM1 echo intensity were indicative of a well-bonded seam, whereas the subsequent values were typical of echoes from voids or delaminations. Because the specimens were considered to be well made, the interpretation of the data was that micro-cavities had formed in the adhesive layer of the newly-formed seams as they were exposed to the warming solar radiation. This finding was consistent with the field inspection of roof No. 3 (Table 1). In the latter case, newly formed seams, considered to be well made and inspected using the field scanner, showed strong EM1 echoes typical of the presence of voids.

In the present field study, a small section of each of the seam specimens was cut for delamination and visual inspection. A close examination revealed that the exposed adhesive layer contained micro-cavities. They were similar to those found in the laboratory experiment using elevated cure temperatures. They could be seen by eye and, therefore, microscopic examination was not conducted.

COMMENTARY OF THE RESULTS

The field scanner is an application of the pulse-echo ultrasonic method. The field investigations were undertaken after laboratory tests indicated that the field scanner offered promise for detecting voids and delaminations in seams. Unfortunately, as was described, testing in the field revealed that the method commonly indicated the presence of voids or delaminations in adhesive-bonded seams of EPDM membranes (i.e., "false positive"), even though no such large defects were present. The "voids" that are of importance in the context of field performance of seams are areas where adhesive is not applied (i.e., skips in the adhesive layer).

The findings of false positives should not be interpreted as a failure of the field scanner. On the contrary, the field scanner was doing exactly what it was designed to accomplish—i.e., to detect discontinuities in the seam system. The unfortunate aspect of the finding is that the pulse-echo method, at least as carried out, could not distinguish between micro-cavities in the adhesive layer and voids (i.e., skips) where adhesive was not applied. It is believed that, because of the mechanism of formation of the micro-cavities, they may be prevalent in seams bonded with solvent-based adhesives. The cases where they may not be found would include application of adhesives to the rubber substrate under conditions that limit solar heating of the sheets (e.g., cool, cloudy weather).

Inspection of seams formed from solvent-based adhesives may produce a considerable number of readings indicative of voids and delaminations. However, it could not be judged whether such readings would be true without extensive cutting of the membrane. This would defeat the purpose of having the field scanner as an NDE method for seam inspection. Thus, at least in its present form, the field scanner has limited application for inspecting seams prepared from solvent-based adhesives.

The question then arises as to whether the field scanner is applicable to seams fabricated using other techniques for EPDM and other membrane systems. Included here might be methods involving solvent welding (if entrapped solvent does not produce micro-cavities), heat welding (thermal fusion), hot asphalt bonding and tape bonding. Investigations of the ability of the field scanner to detect voids in seams fabricated using any of these methods was beyond the scope of the present study. However, the experiences gained from the limited number of roof-top inspections using the field scanner leads to the belief that the equipment may have applicability in these cases.

SUMMARY AND CONCLUSIONS

This paper summarizes the results of a study to develop an ultrasonic NDE method for evaluating the integrity of seams of single-ply roofing membranes. A prototype pulse-echo apparatus (the field scanner), which was designed to scan across seams of roofs while maintaining acoustic coupling to the seam surface, was developed. Results of initial laboratory investigations showed that voids in seams could be distinguished from well-bonded sections using the intensity of the echo from the adhesive layer. This echo was relatively weak for well-bonded seams, and relatively strong for voids.

The focus of the investigations was roof-top scanning of seams to evaluate the performance of the field scanner in practice. Seams of roofs with existing EPDM single-ply membranes, as well as those of roofs with EPDM membranes under construction, were examined using the field scanner. Further laboratory tests were conducted, as necessary, to confirm and explain observations made during the field tests of the seams.

The following is a summary of the key findings:

Investigations of Field Seams—When in-service seams fabricated with either neoprene- or butyl-based adhesives were inspected with the field scanner, many of their echo patterns were indicative of the presence of voids or delaminations. However, the roofs under test were reportedly performing satisfactorily or had been observed to be well prepared. Delamination of sections of some of the in-service seams did not contain delaminations or voids in the adhesive layer. The NDE findings were considered as false positive identification of voids.

The false positives were attributed to the formation of micro-cavities in the adhesive layer. It was hypothesized that the micro-cavities were formed due to the temperature-induced volatilization of residual solvent remaining in the adhesive layer after its application on the roofing membrane material.

Investigations of Micro-Cavity Formation—In the field, seam specimens were prepared on an insulation board on a warm, sunny day. The temperature of the rubber surface exceeded 140°F (60 °C) during the experiment. For both neoprene- and butyl-based adhesive specimens, the intensity of the adhesive layer echo was initially indicative of well-bonded seams. The intensity increased to a value indicative of the presence of voids. Again, the specimens were shown to be well-bonded, and to contain micro-cavities in the adhesive layers.

The following conclusions are made based on results from both the laboratory and field investigations:

- At least in its present form, the field scanner technique has limited applicability to detecting large voids (skips) and delaminations in solvent-adhesive seams. The reason is that the field scanner is sufficiently sensitive to micro-cavities or small bubbles that can be created in the adhesive layer at the time of seam fabrication. Unfortunately, the field scanner cannot distinguish between the voids due to a lack of adhesive and the micro-cavities. It appears that the micro-cavities may be prevalent in adhesive-bonded seams of existing roofs as currently fabricated. Consequently, NDE scanning of seams prepared with solvent-based adhesives could produce an extensive number of "false positive" readings.
- For a limited number of field inspections conducted, the use of the field scanner to inspect seams on roofs was found to be practicable. This implied that the field scanner may have applicability to the inspection of seams formed using other techniques such as solvent welding, heat welding or tape bonding. An investigation of the use of the field scanner for such seams was beyond the scope of the present study.

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REFERENCES

[1] Cullen, W., "Project Pinpoint Analysis: Trends and Problems in Low-Slope Roofing 1983-1988," National Roofing Contractors Association, Rosemont, Ill., 1989.

[2] Strong, A.G., "Factors Affecting the Jointing of Vulcanized Membranes," Proceeding, Paper No.60, Meeting of the American Chemical Society Rubber Division, Philadelphia, Pa., May 1982.

[3] Dupuis, R.M., "Analysis and Design of Adhesive Lap Splices for Elastomeric Single-Ply Membranes," Proceedings, 7th Conference on Roofing Technology, National Roofing Contractors Association, Rosemont, Ill., pp. 16-21, 1983.

[4] Martin, J.W., Embree, E. and Bentz, D.P., "Effect of Temperature and Stress on the Time-to-Failure of EPDM T-Peel Joints," Proceedings, 8th Conference on Roofing Technology, National Roofing Contractors Association, Rosemont, Ill., pp. 69-74, April, 1987.

[5] Rossiter, W.J. Jr., "Further Investigation of the Effect of Application Parameters on Adhesive-Bonded Seams of Single-Ply Roof Membranes," Materials and Structures, Vol. 21, pp. 243-249, 1988.

[6] Rossiter, W.J. Jr., Seiler, J.F. and Stutzman, P.E., "Field Testing of Adhesive-Bonded Seams of Single-Ply Membranes," Proceedings, 9th Conference on Roofing Technology, National Roofing Contractors Association, Rosemont, Ill., pp. 78-87, May, 1989.

[7] Martin, J.W., Embree, E., Rossiter, W.J., Jr., "Effect of Contamination Level on Strength of Butyl-Adhered EPDM Joints in EPDM Single-Ply Roofing Membranes," ibid., pp. 64-72.

[8] Watanabe, H. and Rossiter, W.J. Jr., "Effects of Adhesive Thickness, Open Time, and Surface Cleanness on the Peel Strength of Adhesive-Bonded Seams of EPDM Rubber Roofing Membranes," Roofing Research and Standard Development: 2nd Volume, ASTM STP 1088, American Society for Testing and Materials, Philadelphia, Pa., 1990.

[9] Rossiter, W.J. Jr., "Investigation of the Use of Nondestructive Methods for Inspection of Seams of Single-Ply Roofing Membranes," NBSIR 86-3455, National Institute of Standards and

Technology, Gaithersburg, Md., September 1986.

[10] Rossiter, W.J. Jr. and Clifton, J.R., "The Use of Nondestructive Methods for Inspection of Single-Ply Roofing Membranes," Durability of Building Materials, pp. 343-352, 1986.

[11] Rossiter, W.J. Jr., "Field Evaluation of the Quality of Newly Formed Adhesive-Bonded Seams of Single-Ply Membranes," in Roofs and Roofing, May J.O., Ed., Ellis Horwood Publisher, Chichester, N.Y., pp. 273-289, 1988.

[12] Clifton, J.C., Pizzutti Dos Santos, J.L. and Rossiter, W.J. Jr., "Nondestructive Evaluation Methods for Assessing the Quality of Seams in Single-Ply Membranes," Proceedings 2nd International Symposium on Roofing Technology, National Roofing Contractors Association, Rosemont, Ill., pp. 433-437, September 1985.

[13] Lethiecq, M., Baboux, J.C. and Perdrix, M., "Nondestructive Characterization of Thin Adhesive Bonds," Nondestructive Characterization of Materials II, Prenum Press, N.Y., pp. 395-402, 1986.

[14] Couchman, J.C., Yee, B.G.W. and Chang, F.H., "Adhesive Bond Strength Classifier," Materials Evaluation, pp. 48-50, April 1979.

[15] Yew, C.H. and Weng X.W., "Using Ultrasonic SH Waves to Estimate the Quality of Adhesive Bonds in Plate Structures," Journal of Acoustic Society of America, 77(5), May 1985.

[16] dos Reis, H.L.M. and Krautz, H.E., "Nondestructive Evaluation of Adhesive Bond Strength Using the Stress Wave Factor Technique," Journal of Acoustic Emission, Vol. 5, No. 4, pp. 144- 147, 1986.

[17] Szilard, J., "Ultrasonic Testing", John Wiley & Sons, N.Y., 1982.

[18] Spanner, G., "Nondestructive Techniques for Assessing Field Seam Quality of Prefabricated Geomembranes," Proceedings of International Conference on Geomembranes, Industrial Fabrics Association International, St. Paul, Minn., pp. 369-373, June 1984.

[19] McMaster, R.C., "Nondestructive Testing Handbook, Volume II," The Ronald Press Company, N.Y., pp. 44.6-44.19, 1959.

[20] Watanabe, H. and Rossiter, W.J. Jr., "Nondestructive Evaluation of the Integrity of Seams of Single-Ply Roofing Membranes: Laboratory Evaluation of a Prototype Test Apparatus," NISTIR 4424, National Institute of Standards and Technology, Gaithersburg, Md., November 1990.

[21] Rossiter, W.J. Jr., Seiler, J.F. and Stutzman, P.E., "Report of Roof Inspection: Characterization of Newly-Fabricated Adhesive-Bonded Seams at an Army Facility," NISTIR 89-4155, National Institute of Standards and Technology, Gaithersburg, Md., October 1989.

[22] Hartmann, B. and Jarzynski, J., "International Apparatus for Ultrasonic Measurements in Polymers," Journal of Acoustic Society of America, Vol. 56, No. 5, November 1974.

[23] Krautkramer, J. and Krautkramer, H., "Ultrasonic Testing of Materials," Second Edition, Springer-Verlag, New York, N.Y., 1977.

[24] Rosenfield, M.J., "Field Test Results of Experimental EPDM and PUF Roofing," Technical Report M-357, US Army Corps of Engineers, September 1984.

[25] Watanabe, H. and Rossiter, W.J. Jr., "Nondestructive Evaluation of the Integrity of Seams of Single-Ply Roofing Membranes: Results of the Field Investigations and Recommendations," NISTIR 4425, National Institute of Standards and Technology, Gaithersburg, Md., November 1990.

Roof No.	Location	Adhesive Type	Attachment to the Substrate	Seam Age[a]
1	VA	Neoprene	Fully-adhered	Minimum of 5 years
2[b]	IA	Butyl	Fully-adhered	About 4 years
3	VA	Butyl	Fully-adhered	A few minutes to 9 days
4	MD	Neoprene	Mechanically -fastened	About 4 years
5	VA	Butyl	Fully-adhered	About 1 year

[a] All seams were reportedly performing well; no observations were made to the contrary when the NDE inspections were conducted.

[b] This specimen was tested only in the laboratory; no field scans were conducted.

Table 1 Summary of roofs investigated.

No.	Adhesive		Cure Conditions[a]
	B[b]	N[c]	
1	X	X	Room temperature for 4 weeks
2	X	X	Room temperature for 20 min. followed by a heat cure at 158°F (70°C) for a total cure time of 4 weeks
3	X	X	Room temperature for 1 day followed by a heat cure at 158°F (70°C) for a total cure time of 4 weeks
4	X	X	Room temperature for 3 weeks followed by a heat cure at 158°F (70°C) for 1 week
5	X	-	Room temperature for 2-3 min. followed by a heat cure at 158°F (70°C) for a total cure time of 1 day
6	X	-	Adhesive was applied on rubber at 120 ± 2°F (49 ± 1°C); then the specimen was heat cured at 158°F (70°C) for 1 day

[a] Unless otherwise indicated, the rubber was at room temperature when the adhesive was applied to its surface.
[b] B indicates that the adhesives was butyl-based.
[c] N indicates that the adhesive was neoprene-based.

Table 2 Seam Samples and Cure Conditions.

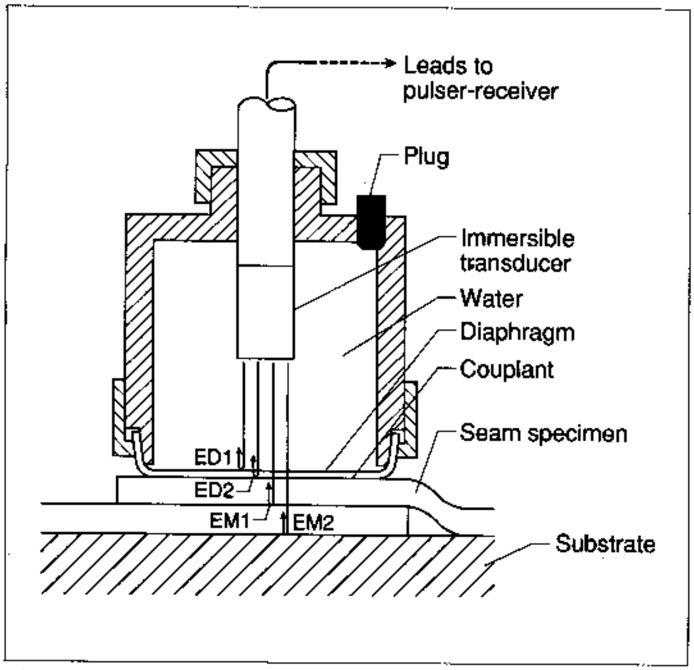


Figure 1 *Schematic illustration of the transducer holder acoustically coupled to a seam specimen. (Reflections of the ultrasonic pulse from the various interfaces are also illustrated, and are designated as ED1, ED2, EM1, and EM2.)*

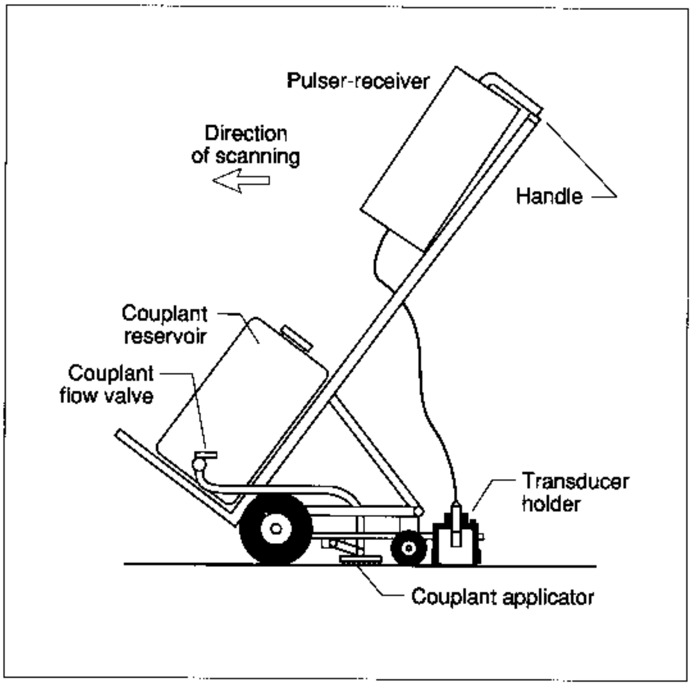


Figure 2 *Prototype field scanner used in the study. (The detail of the transducer holder is presented in Figure 1.)*

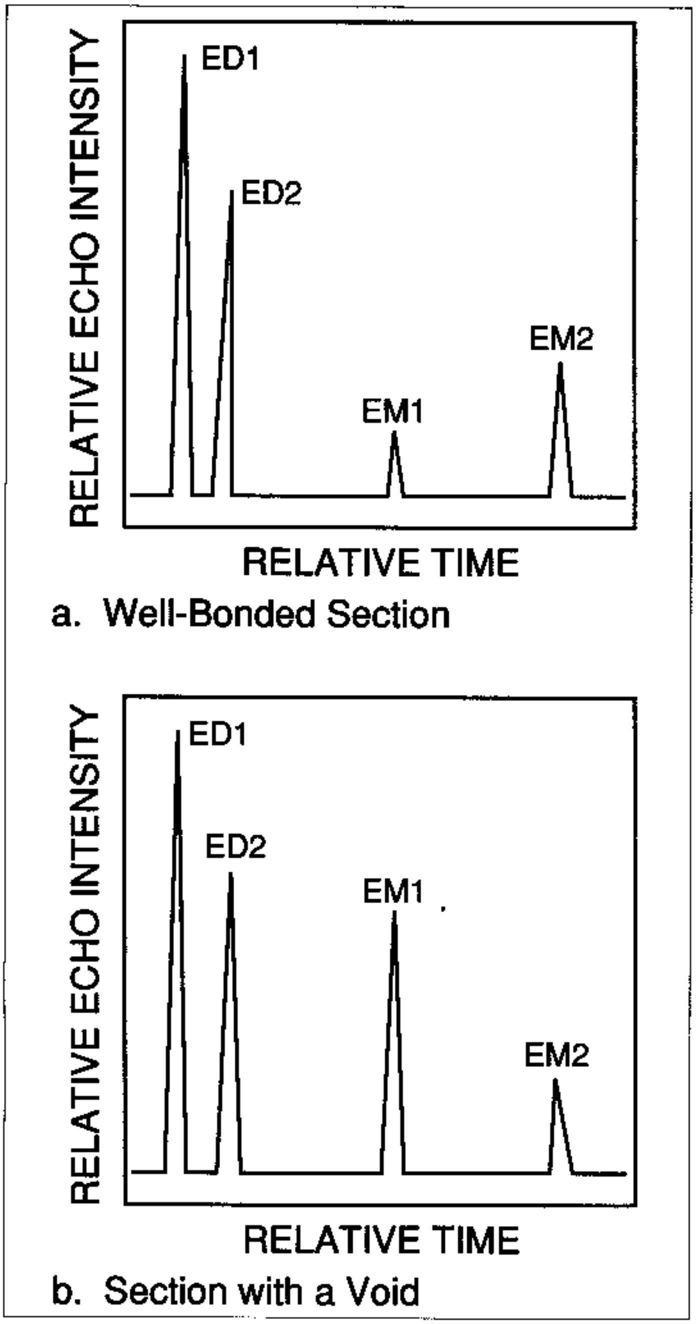


Figure 3 *Schematic illustration of echo patterns for a well-bonded section of a seam and a section with a void. (For the definition of the echoes, ED1, ED2, EM1, and EM2, see text and Figure 1.)*

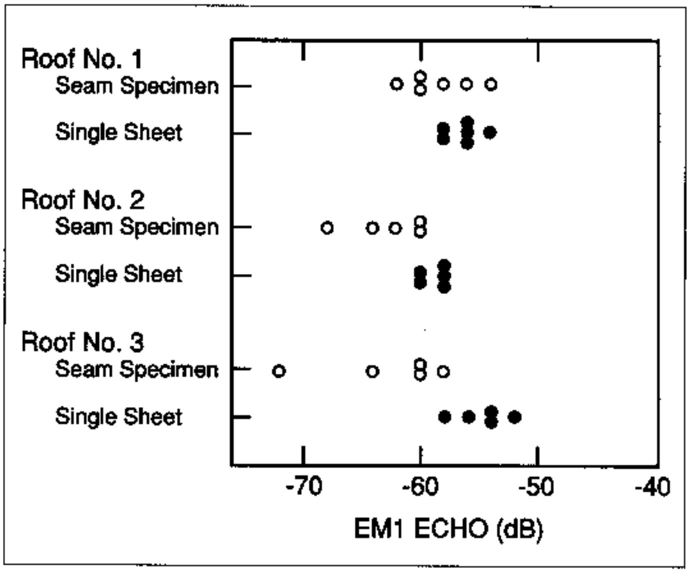
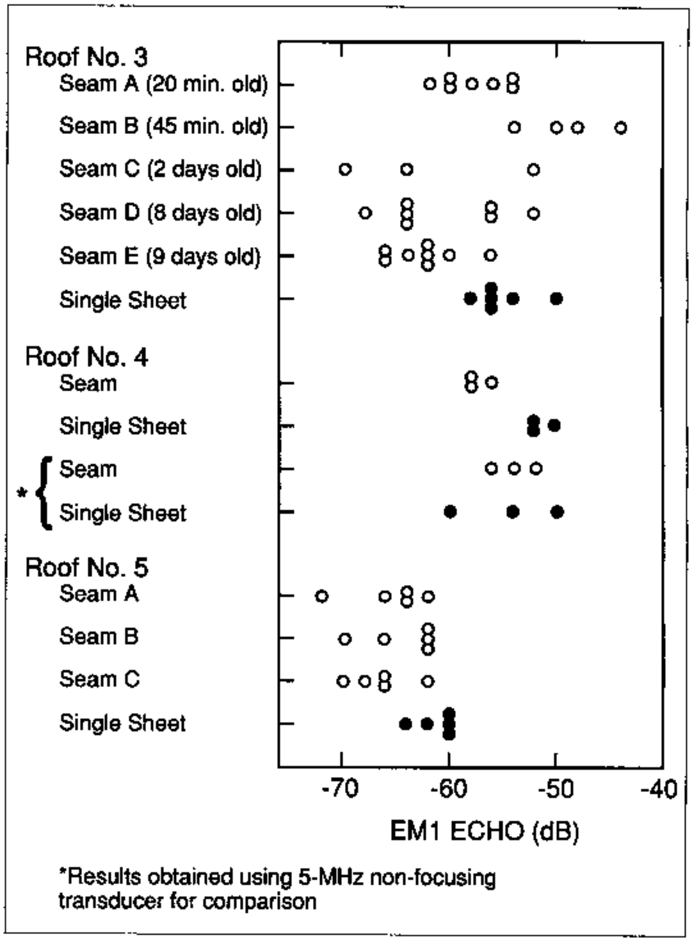


Figure 4 *Intensity of the EM1 echo measured for specimens cut from roofs Nos. 1, 2 and 3. (A 5-MHz focusing transducer was used in the measurement.)*



*Results obtained using 5-MHz non-focusing transducer for comparison

Figure 5 *Intensity of the EM1 echo measured in the field investigation for roof Nos. 3, 4 and 5. (Except where noted, a 5-MHz focusing transducer was used in the measurement.)*

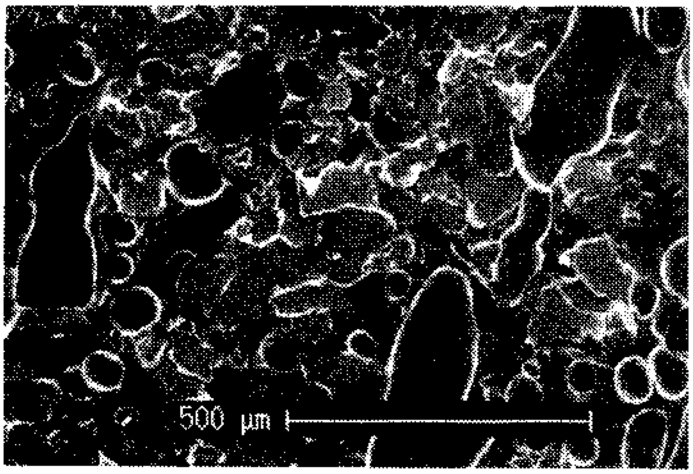


Figure 6 *SEM photomicrograph of the delaminated surface of the adhesive layer in seam A of roof No. 3.*

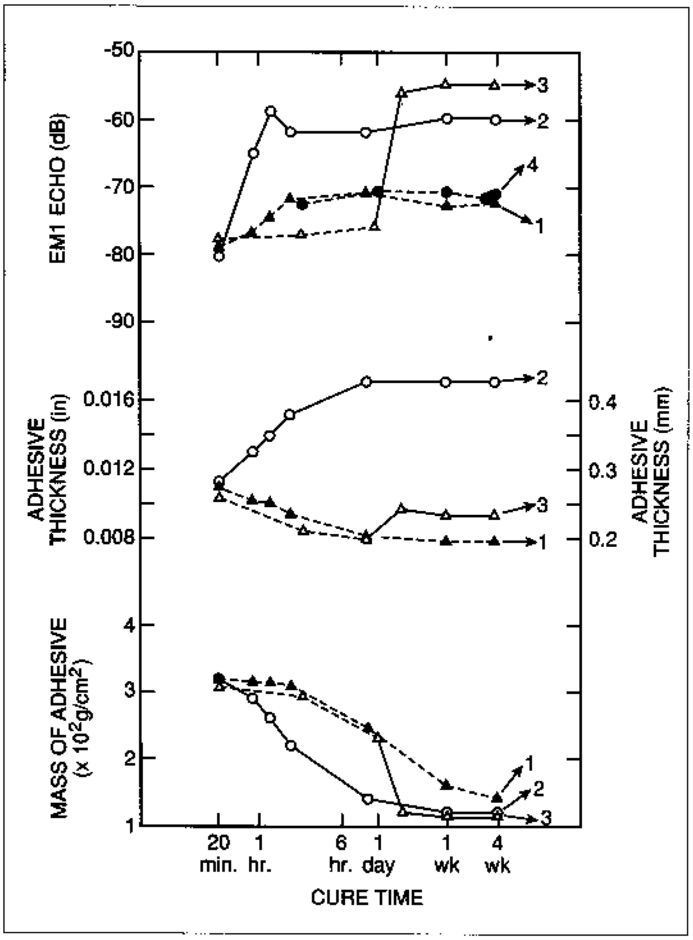


Figure 7 *Results of the laboratory experiment on elevated cure temperature for seam specimens using neoprene-based adhesives. (The numbers in the diagram indicate the cure condition given in Table 2.)*

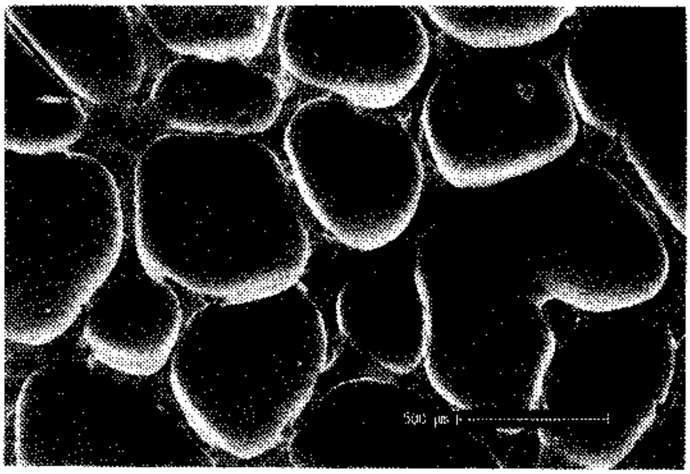


Figure 8 *SEM photomicrograph of the delaminated surface of the neoprene-based adhesive layer.*

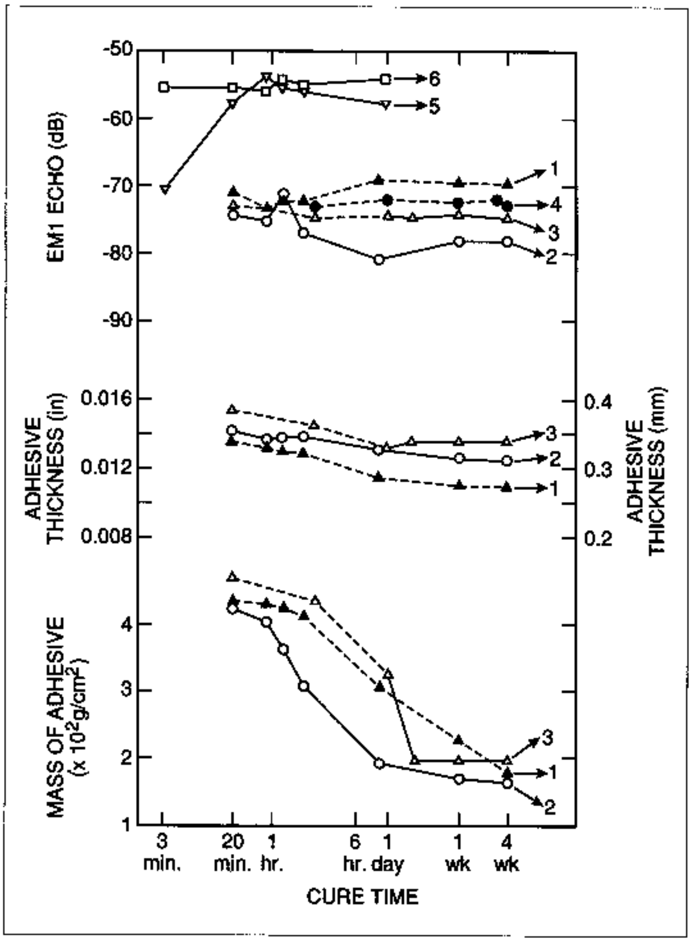


Figure 9 *Results of the laboratory experiment on elevated cure temperature for seam specimens using butyl-based adhesives. (The numbers in the diagram indicate the cure condition given in Table 2.)*

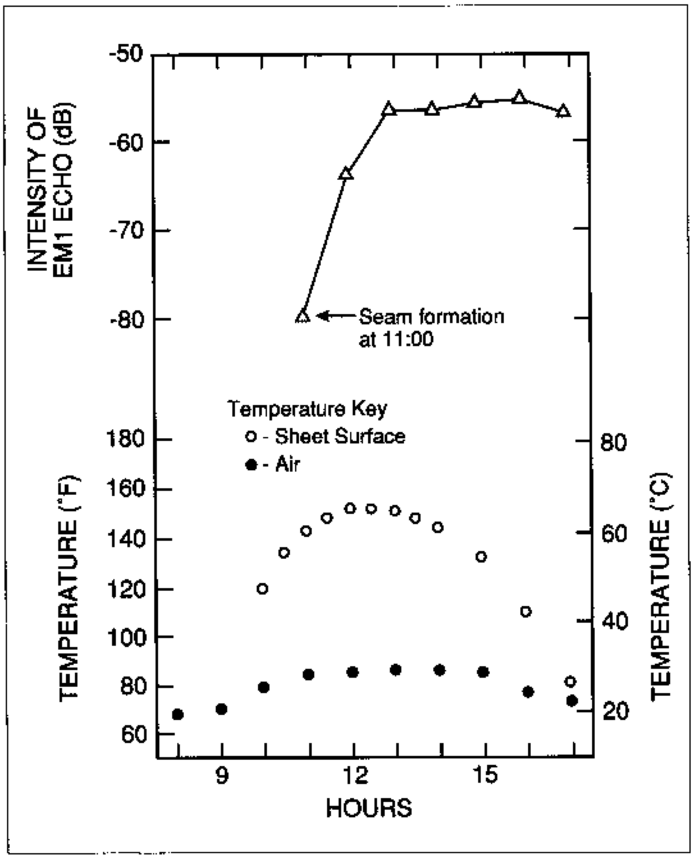


Figure 10 *Temperatures and EM1 echo intensity measured for the field-seam specimen using neoprene-based adhesive.*

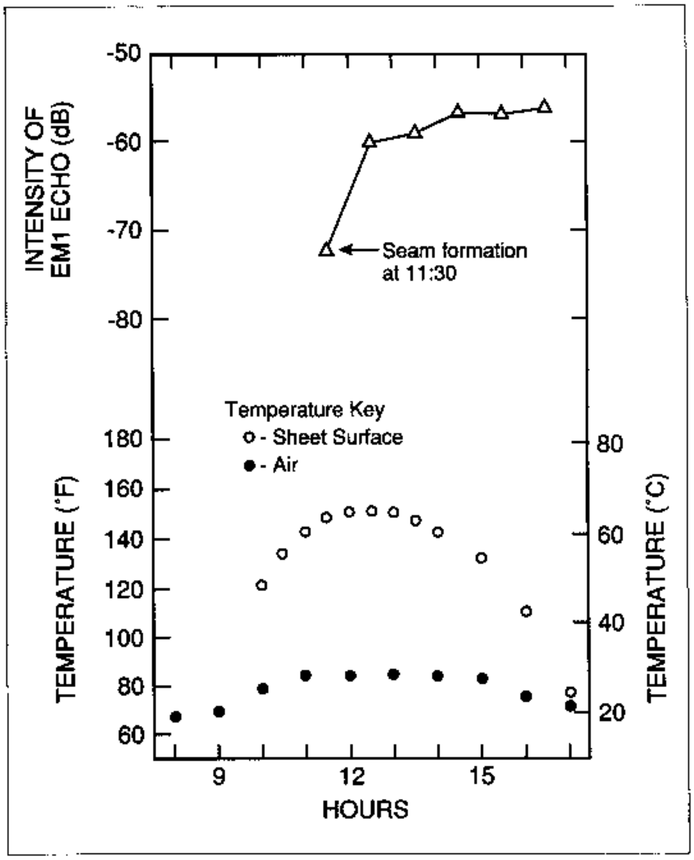


Figure 11 *Temperatures and EM1 echo intensity measured for the field-seam specimen using butyl-based adhesive.*