

FACTORS AFFECTING THE STRENGTH AND CREEP-RUPTURE PROPERTIES OF EPDM JOINTS

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The objectives of this research were:

- To determine from a small but important set of material and fabrication variables (adhesive thickness, cure time, mechanical load, adhesive type and surface cleanness), those variables which are primarily responsible for causing joint failures.
- To rank these experimental variables in the order in which they affect the strength and creep-rupture life of butyl-adhered EPDM joints.
- To determine the maximum peel stress an EPDM joint can sustain over its design life.

From our results, cure time and level of cleanness of the EPDM rubber surface have the greatest effect on joint strength; while the thickness of the adhesive and the magnitude of the mechanical load have the greatest effect on the time-to-failure of a joint. It was further observed that the maximum stress that an EPDM joint can sustain over time is only a small fraction (less than 5 percent) of its short-time peel strength. It was concluded, therefore, that efforts should be made to ensure that the field-formed joints are not subjected to large peel loads.

KEYWORDS

Butyl adhesive, creep-rupture life, EPDM, neoprene adhesive, peel strength, single-ply roofing, strain rate, time-to-failure.

INTRODUCTION

EPDM (ethylene-propylene-diene terpolymer) single-ply roofing membranes are widely used in the United States for low-sloped roof applications. The popularity of this system stems in part from the chemical inertness of EPDM, which provides the membrane with its excellent weathering properties. This inertness also makes it difficult to form a strong adhesive bond between two EPDM sheets which is needed to assure the quality of a field-formed seam joint. Evidence for this difficulty is readily apparent from Project Pinpoint's data,^{4,5,6,7} from case studies conducted by NIST staff,^{18,20} and from general survey papers on the performance of single-ply roofing systems.^{8,9,22} The objectives of this study were to determine the relative importance of several fabrication variables (adhesive thickness, cure time, level of surface contamination of the EPDM sheet, mechanical load and adhesive type) on the short-time strength and creep-rupture lives of butyl-adhered EPDM joints. The contaminants of interest are the talc-like particles (talc, clay, or mica) which are commonly applied onto EPDM membrane surfaces prior to

vulcanization. The experimental results for the butyl-adhered EPDM joints are compared against previously published results from neoprene-adhered EPDM joints. Only four factors were varied in the short-time strength and creep-rupture experiments; two of these variables (adhesive thickness and level of surface contamination) were common to both experiments. This redundancy was built into the experimental design to determine if the rankings of these factors remained the same at different strain rates.

The seams in this study were stressed in peel, as opposed to lap shear. Peel-type stresses are felt to be the primary cause of many field-observed seam defects; a conclusion which is supported by our laboratory results. Peel-type stresses may be either induced or built into a seam joint. Peel-type stresses may be induced into a peel joint whenever the EPDM membrane is heated to a temperature greater than the temperature at the time the roof was installed. Peel-type stresses result from the thermal expansion of EPDM rubber, which is greater than that of most of the other materials used in roof construction (e.g., concrete, steel and wood). The thermal expansion of the EPDM membrane can cause buckling at the seam joint which would be particularly deleterious since this would induce peel-type stresses into the joint seam. Peel-type stresses can also be built into a seam during roof installation. Common seam defects, having built-in peel stresses, include ripples, fishmouths and T-joints (see Figure 1).

Whereas peel-type stresses are felt to be a major cause of seam defects, lap shear stresses are believed to have a minor effect on seam failures. This belief is largely due to the high maximum working strain (normally ranging from 300 to 500 percent) inherent in elastomeric materials and the excellent shear performance of elastomeric joints.^{14,17} Seams are stressed in lap shear whenever the EPDM membrane shrinks or whenever it is subjected to hydrostatic tensile stresses. EPDM shrinks as the temperature decreases, or as a result of oil loss and crosslinking. These shrinkage strains are too small (normally less than 5 percent) to cause lap shear failures. EPDM membranes are subjected to hydrostatic tensile stresses whenever wind uplifting occurs. While the strains induced by wind uplifting are normally larger than those induced by membrane shrinkage, they are still small relative to the maximum working strains of elastomeric materials and, thus, are not believed to be able to cause many seam failures. This latter observation is supported by limited field observations in which seams subjected to high wind uplift loads have remained intact and by extensive laboratory observations. For example, in one experiment 30 lap shear specimens were continuously loaded to a load great-

er than 20 percent of their short-time ultimate strength (this is equivalent to a strain of approximately 100 percent) for over seven months. None of the specimens failed at the time that the experiment was terminated.

SHORT-TIME PEEL STRENGTH OF BUTYL-ADHERED EPDM JOINTS

Short-time strength tests are the most common method for assessing the quality of an adhesive joint (e.g., ASTM D 1876),¹ and are an easy and convenient way for obtaining results which may be useful in assuring seam quality. The objectives of the short-time strength experiments were:

- To assess the sensitivity of the strength of a peel joint prepared with one of two commercially available butyl adhesives at different cure times, adhesive thicknesses and levels of contamination.
- To determine the locus of failure for uncontaminated and contaminated peel joints.
- To determine the effect of adhesive thickness on peel strength at different cure times and contamination levels.
- To determine if the peel strength of an EPDM butyl-adhered joint is more sensitive to adhesive thickness or cleanness.

Each of these objectives is important in understanding failure mechanisms and providing the technical basis for guidelines for forming reliable joints. The experimental procedure for this phase of the research is discussed in detail in Martin, et al.¹⁷ The strength measurements and curing conditions for all of the joints in this experiment were made at 24°C and 45 percent relative humidity.

Results and Discussion

In a previous paper,¹⁸ it was concluded that the strengths of the joints adhered with the two commercially available adhesives were not significantly different from each other at cure times greater than six hours. Thus, the data sets for the two adhesives were combined and reanalyzed to determine if the thickness of the adhesive had an effect on peel strength.

Failure Mode

For joints comprised of a contaminated strip and cured for less than two hours, and for the clean joints regardless of cure time, failure was cohesive; that is, the locus of failure was within the adhesive layer (Location 1 in Figure 2). For joints containing a contaminated strip cured for longer than six hours, the locus of failure was most often between a talc-like particle and the EPDM substrate (Location 3 in Figure 2) or between two stacked talc-like particles (Location 4 in Figure 2). Less often, the locus of failure was between a talc-like particle and the adhesive (Location 2 in Figure 2).

Effect of Adhesive Thickness and Contamination of Peel Strength

Peel strength, S , changed linearly with adhesive thickness, T_{adh} ; that is,

$$S = a_{tk} + b_{tk} T_{adh} \quad (1)$$

where a_{tk} and b_{tk} depend on the cure time. At cure time

less than two hours, the slope of the regression line, b_{tk} , was negative (see Figure 3); at intermediate cure times (cure time between four and six hours), the slope of the regression line was close to zero, and at long cure times (cure times greater than six hours), peel strength increased with adhesive thickness. Thus, between four and six hours, the effect of increasing the thickness on joint strength made a transition from having a negative to a positive effect.

For contaminated joints, the trends in the parameters, a_{tk} and b_{tk} , with cure time were similar to those for clean joints. Contaminants appeared to block the formation of adhesive bonds between the adhesive and EPDM substrate. Hence, the presence of contaminants tended to reduce the effective load-carrying width of a joint.

Strength Versus Cure Time at Different Contamination Levels

Strength data at the five levels of contamination are plotted against cure time in Figure 4. Superimposed on each data set is the predicted Gompertz growth curve described in Martin, et al.¹⁷ The maximum predicted strength, S_0 , is plotted against greyscale level in Figure 5. Greyscale value is a measure of the amount of light reflected off an EPDM surface and detected by a video camera.¹⁷ If the EPDM surface was a perfect absorber, the greyscale value would be zero (for highly cleaned EPDM, the greyscale value was 33) while if it was a perfect reflector, the greyscale value would be 256. Thus, the greater the amount of light reflected from the EPDM surface, the greater the observed greyscale value. The relationship between greyscale value and the amount of talc deposited onto an EPDM surface is highly linear and highly correlated.¹⁷ Thus, the higher the greyscale value, the greater the amount of talc deposited onto the EPDM surface.

Superimposed on Figure 5 are the peel strengths for joints fabricated from an "as-received" strip (that is, from a strip cut from a commercial EPDM membrane sheet), which had greyscale values greater than 200 and which were cured for longer than 48 hours. Extrapolation of S_0 to these higher "as-received" greyscale levels resulted in a predicted strength which was greater than the observed strengths. This discrepancy may be due to the non-uniform distribution of contaminants on the surface of the "as-received" strips, or may be due to the activation of the EPDM surfaces during the laboratory cleaning process, resulting in an improvement in the adhesive properties of the laboratory prepared joints.

A comparison of the sensitivities of peel strength to contamination and adhesive thickness can be made from Figures 3 and 5, respectively. From Figure 3, the difference in the strength of joints at long cure times and over the range of adhesive thickness is 0.2 kN/m (approximately 1 lbf/in) while the maximum strength, S_0 , of the peel joints decreased by more than 50 percent over the range of contamination levels; that is, strength decreased by approximately 0.9 kN/m (5.1 lbf/in) over the experimental range of contamination levels. Thus, contaminants affect peel strength to a much greater extent than does a change in adhesive thickness.

Peel Strength Conclusions

The sensitivity of strength in peel of butyl-adhered EPDM joints to surface contaminants, adhesive thickness, cure time and adhesive type were experimentally determined. From the experimental results and for the range of conditions covered by them, it was concluded:

- The maximum strength, S_o , for butyl-adhered EPDM joints occurred when both strips are clean and the maximum strength decreased by more than one-half over the experimental contamination range.
- Peel strength increased linearly with adhesive thickness for joints cured for greater than six hours.
- Cleanness of the EPDM surface had a greater effect on peel strength than did thickness.

CREEP-RUPTURE LIVES OF BUTYL-ADHERED EPDM JOINTS

The major difference between short-time strength and creep-rupture experiments is one of strain rate. It is believed that the low strain rates inherent in creep-rupture experiments are more representative of the strain rates experienced by an in-service seam. For example, thermally induced strain rates have been estimated to be on the order of 10^{-6} in/in/min.¹² The strain rates for seam defects having built-in peel stresses depend on the loads to which the seam defects are subjected. To date, few creep-rupture experiments have been published.¹³ Instead, short-time strength experiments are preferred.^{9,19,23,24} Before the results of short-time peel strength experiments are accepted, however, it is important to ensure that the ranking of the experimental variables resulting from short-time strength experiments are the same as those resulting from creep-rupture experiments. Otherwise, performance requirements may be imposed which have little to do with the in-service performance of seams.

The objectives of the creep-rupture time-to-failure experiments were:

- To determine the effect of contamination level, adhesive thickness, cure time and mechanical load on the creep-rupture of butyl-adhered EPDM joints stressed in peel.
- To determine the failure mode of the contaminated and uncontaminated joints.
- To derive a mechanistically-based model for predicting the time-to-failure of a joint stressed in peel.
- To compare the rankings of the principal experimental variables as they affect short-time peel strengths and creep-rupture times-to-failure.

Results and Discussion

Failure Mode

From visual inspection of the failed peel strips and from mass measurements, it was concluded that all the creep-rupture joints failed cohesively (see location 1 in Figure 2), regardless of the level of contamination or the magnitude of the applied load. It was also observed that the rate of peel was constant for each joint, although the rate of peel was highly variable from joint-to-joint.

Effect of Adhesive Thickness and Contamination on Creep-Rupture Times-to-Failure

The dependence on the time-to-failure on applied stress has been studied for a wide-variety of materials including metals, polymers and polymeric fibers.^{2,13} One model, which is often fitted to the time-to-failure data, τ , as a function of

applied stress, σ_o , is a power law model of the form:

$$\tau = a\sigma_o^{-b} \quad \text{\{the "power law model"\}} \quad (2)$$

where a and b are parameters. For joints stressed in peel, the applied stress, σ_o , is given by

$$\sigma_o = (4PE_{adh}/WT_{adh})^x \quad (3)$$

where, for the present, it is assumed that Young's modulus of the adhesive, E_{adh} , and the width of a joint, W , are constants, while the creep-rupture load, P , and the thickness of the adhesive, T_{adh} , are measurable variables.

The power law model can be reformulated by substituting Equation (3) into Equation (2) yielding

$$\tau = a (4PE_{adh}/WT_{adh})^{-b/2} \quad (4)$$

Taking the logarithms of both sides and using the simplifying assumptions, Equation (4) becomes

$$\log_{10}\tau = a' - b/2 \log_{10} P/T_{adh} \quad (5)$$

where $a' = \log_{10}a - b/2 \log_{10} 4E_{adh}/W$.

In Figure 6, the power law model is fitted to all of the time-to-failure date (number of joints = 540). The parameter estimates of the power law model are: $a' = -5.0$ with an estimated standard error of the estimator of 0.11, and $b = -2.31$ with an estimated standard error of the estimator of 0.043. The standard error of the estimate, the residual standard deviation, is 0.19 and the squared correlation coefficient, r^2 , for the fit of Equation (4) to the time-to-failure data is 84.2 percent.

The power law model, Equation (4), predicts that the time-to-failure of a joint decreases with increasing mechanical load and/or a decreasing adhesive thickness. The dependence of time-to-failure on mechanical load is well-documented.^{2,13} The dependence of the time-to-failure on adhesive thickness is presented in Figure 7. From Figure 7, it is evident that the time-to-failure of a peel joint could be greatly increased by incrementally increasing the thickness of the adhesive. To verify this prediction, three joints, each comprised of two cleaned strips having a greyscale value of 33, were prepared and were loaded to 0.308 kN/m. The thicknesses of the adhesive at the time-of-failure of the joints were approximately 350, 410 and 430 μm , while the times-to-failure were 1030, 1469 and 788 hours, respectively. As can be seen from Figure 7, a doubling of the adhesive thickness from 175 μm to greater than 350 μm increased the times-to-failure by a factor of one hundred (from approximately 10 hours, for joints having a thickness of approximately 175 μm , to approximately 1000 hours, for joints having a thickness of approximately 350 μm).

Contaminants may also affect the time-to-failure of a joint. It was assumed that contaminants reduce the effective load carrying width of a joint in proportion to the level of contamination. Thus, the joint width term, W , in Equation (3) was modified as follows:

$$W_{\text{effective}} = c_1 W (G_{\text{max}} - G_{\text{act}})/G_{\text{max}} \quad (6)$$

where $0 \ll c_1 \ll 1$ and where

G_{act} = observed contamination level and
 G_{max} = maximum level of contamination for all the experimental joints (here, $G_{max} = 170$).

Substituting Equation (6) into Equation (4), and taking the logarithms of both sides, yields an equation of the form:

$$\log_{10} \tau = b_1 + b_2 \log_{10} [(G_{max} - G_{act})/G_{max}] + b_2/2 \log_{10} [P/T_{adh}] \quad (7)$$

where $b_1 = \log_{10} a - b/2 \log_{10} 4E + b/2 \log_{10} c_1 W$. The effect of contaminants on time-to-failure was statistically significant, but the effect of contaminants on the peel creep-rupture life of the butyl-adhered joints was minor relative to the thickness of the adhesive and the magnitude of the mechanical load.

Creep-Rupture Time-to-Failure Conclusions

The creep-rupture sensitivity of butyl-adhered EPDM joints to surface contaminants, adhesive thickness, cure time and mechanical load were experimentally determined, and the significance of each factor on time-to-failure evaluated and ranked. From the creep-rupture data, for the range of experimental conditions, it was concluded:

- The failure mode for all the creep-rupture joints was cohesive regardless of contamination level or applied mechanical load.
- A mechanistically based model, depending on mechanical load, contamination level and adhesive thickness, was derived and fitted to the creep-rupture data. The fit of the model to the data was quite good and from this analysis it was concluded that the creep-rupture life mainly depends on mechanical load and adhesive thickness, and to a lesser extent on contamination level. Thus, the ranking in importance of the two principal experimental variables (level of contamination and adhesive thickness) in the creep-rupture experiment was reversed from their ranking in the short-time strength experiment. This reversal is attributed to changes in the rheological properties of the adhesive when stressed at different peel rates.
- Performance requirements for seams should be based on creep-rupture results rather than short-time strength results.

COMPARISON OF THE TIMES-TO-FAILURE OF BUTYL- AND NEOPRENE-ADHERED EPDM JOINTS AS A FUNCTION OF LOAD RATIO

It is common in engineering practice to minimize the probability of failure of a structural element by assigning a working or design load, $P_{working}$, to the element which is only a fraction of its yield load, P_{yield} . Practically, this is achieved by reducing the yield load by a factor, FS; that is,

$$FS = P_{yield}/P_{working} \quad (8)$$

where in industrial applications, it is typical to apply factors of safety whose product is between one and three.⁹ (Applied loads are directly proportional to the applied stresses. Thus, the working load and yield load can just as easily be expressed as a working stress and yield stress.) The factors of safety account for unexpectedly high in-service loads and for reduction in the resistance properties of a structural ele-

ment. This results from statistical variations in the material properties and from variations introduced during fabrication.¹⁰ For visco-elastic structural materials (such as wood, geosynthetic materials and seams in EPDM roofing membranes), the greatest reduction factor (the reciprocal of the factor of safety) compensates for the creep-rupture effect; that is, the ability of visco-elastic materials to sustain only a fraction of their yield loads for prolonged periods of time.

The reduction factor for creep-rupture was found by plotting the mean time-to-failure of a roofing seam against the applied load ratio, $LR = P_{applied}/P_{yield}$, (or equivalently, the stress ratio) and selecting that load ratio (stress ratio) having a mean time-to-failure which is equal to or greater than the expected design life of the component. In practice, the applied load is normally constant, while the strength of an joint depends greatly on the strength of the adhesive. In particular, butyl adhesives tend to be approximately two to four times stronger than neoprene.^{18,21} Thus, if the applied load is one unit per unit width and the strength of a neoprene-adhered joint is four units per unit width and that of a butyl-adhered joint is 10 units per unit width, then their respective load ratios are 25 percent and 10 percent.

Stress ratio versus mean time-to-failure plots are shown in Figure 8 for neoprene- and butyl-adhered EPDM joints loaded in peel at temperatures far above the glass transition temperature of both the rubber and adhesives. For purposes of comparison, data for wood stressed in bending²⁹ and high density polyethylene (HDPE) stressed in lap shear¹¹ are also shown. From Figure 8, wood has been tested down to 65 percent and HDPE to 45 percent. Straight-line extrapolation of the wood and HDPE curves would indicate wood is capable of sustaining loads equal to approximately 60 percent of its short-time stress for 10 years, while HDPE can only sustain loads equal to 20 percent of its short-time strength for 10 years. Extrapolation of neoprene- and butyl-adhered EPDM peel joint time-to-failure curves indicate that these materials can only sustain 5 percent of their yield load for this period of time. That is, the design load for joints stressed in peel must be less than 1/20th of the short-time peel strength of both butyl- and neoprene-adhered peel joints. In other words, seams stressed in peel can sustain less than 5 percent of their short-time strengths for any period of time.

SUMMARY

The most frequently reported defect in EPDM single-ply roofing systems occurs in the field-formed seams. Seam defects occur most often within the first three years after installation of the roof, and may result from weaknesses in the materials, deficiencies in the fabrication technique, or as a result of environmental stresses. The objectives of this study were to determine the importance of several material and fabrication variables as they affect the short-time strength and creep-rupture life of butyl-adhered EPDM joints stressed in peel and, where possible, to derive mechanistically-based models. The joints were stressed in peel because it is believed that this stress mode is a primary factor causing seam delaminations.

The gain in strength of the joints as a function of cure time was modeled with the Gumpertz growth curve equation (see Figure 4). From the analysis of our strength data, peel strength was highly dependent on cure time and on the level of contamination, and to a much lesser extent on adhesive thickness.

From the creep-rupture experimental results, the joints always failed cohesively regardless of the level of contamination or the magnitude of the mechanical load. A mechanistically-based model, which was functionally dependent on mechanical load, adhesive thickness and contamination level, was fitted to the time-to-failure data. Times-to-failure were highly dependent on mechanical load and adhesive thickness, and to a much lesser extent on the level of contamination. One prediction of this model was that an increase in the thickness of the adhesive layer above the experimental thickness could result in a great increase in the service life of a joint. This prediction was experimentally confirmed.

Although the magnitude of the peel loads to which field-formed seams are subjected is unknown, it is possible to determine from the creep-rupture results the upper limit of peel stresses which can be sustained by a joint over time. Our experimental results indicate that butyl- and neoprene-adhered EPDM joints stressed in peel can only sustain less than 5 percent of their yield stresses over the design life of a roof. Thus, efforts should be made to ensure that field-formed seams are subjected to minimal peel loads. Where this is not possible, our results indicate that the service life of seams can be significantly improved by minimizing the magnitude of the peel loads, by increasing the thickness of the adhesive and, to a lesser extent, by ensuring the cleanliness of the seams.

The authors experimental results have identified several fabrication (surface cleanliness), material (adhesive thickness and cure time) and stress (peel load magnitude) variables as having a significant effect on the service life of EPDM roof seams. The variables examined in this experiment are only a subset of the material, fabrication, design and stress variables which may affect the service life of EPDM seams. In future experiments, these other variables can be interjected into the analysis to determine which have a significant effect on a roofing seam's service life.

ACKNOWLEDGMENTS

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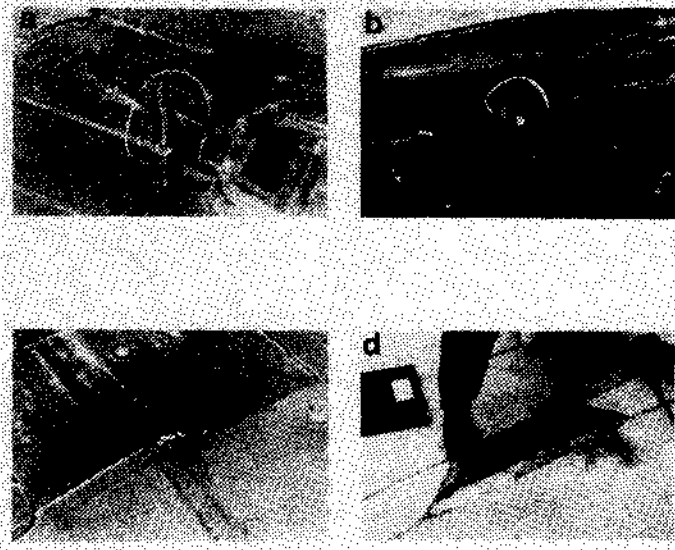


Figure 1 Seam defects in which a peel-type failure mode is implicated: (a,b) ripples in the EPDM membrane sheet and (c) T-joint. It is not clear what the failure mode is for the seam defect pictured in (d).

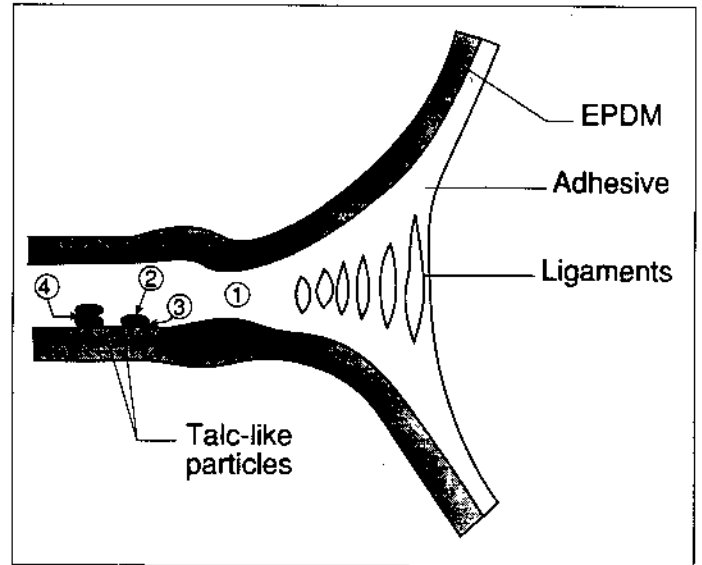


Figure 2 Schematic representation of failure locations: (1) failure within the adhesive layer which is typical in the case of cohesive failure, (2) failure at the adhesive/talc-like particle interface, (3) failure at a talc-like particle/EPDM interface and (4) failure at a talc/talc interface.

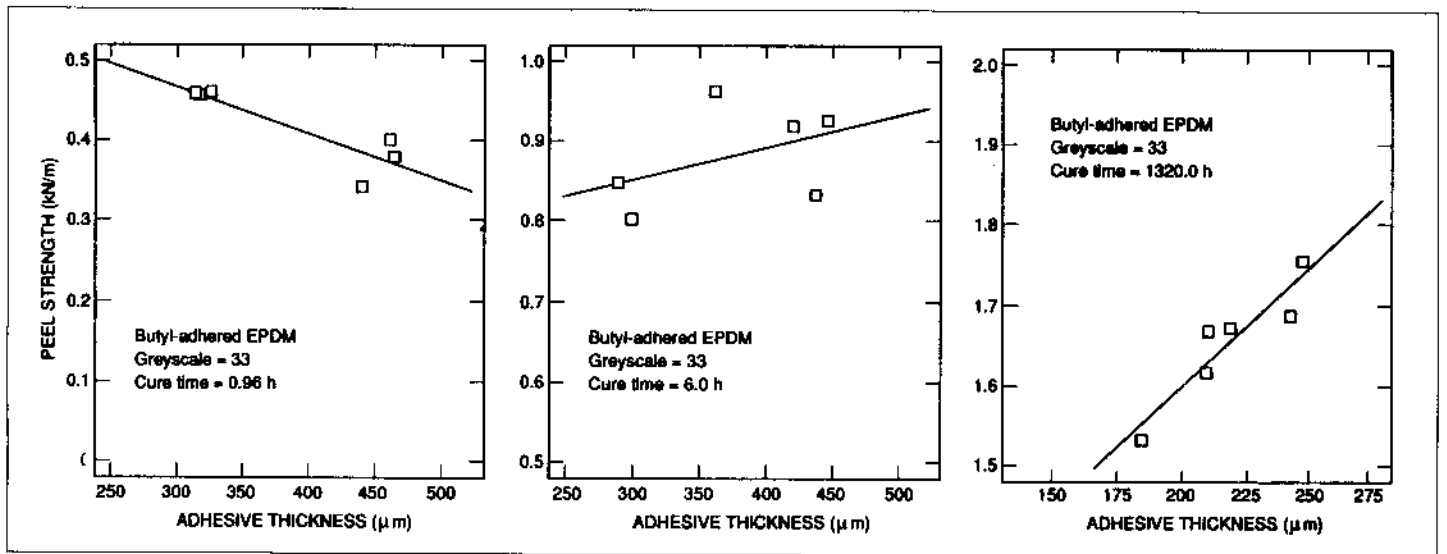


Figure 3 Change in peel strength versus adhesive thickness at three different cure times.

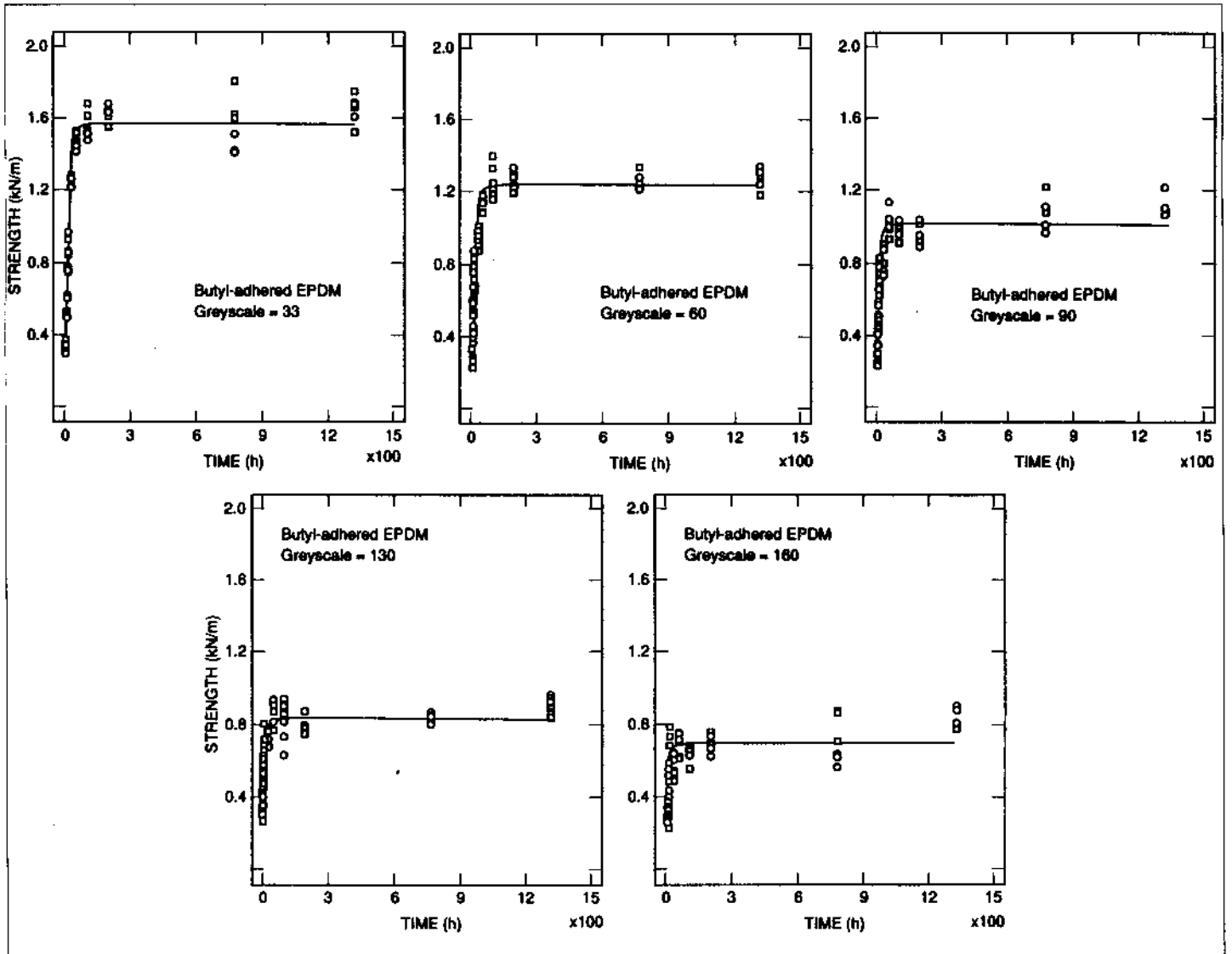


Figure 4 Peel strength versus cure time at five levels of contamination.

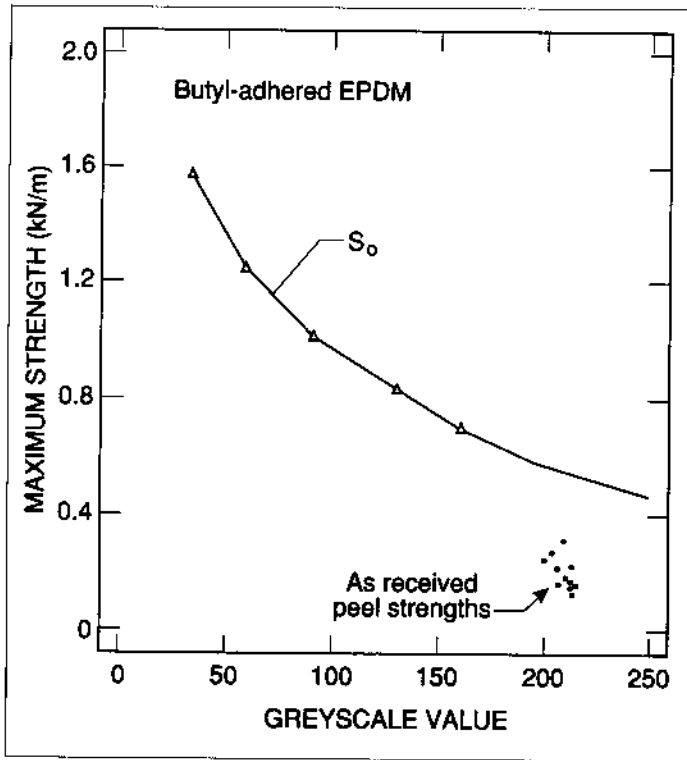


Figure 5 Maximum peel strength, S_0 , versus contamination level. Parameter S_0 is in units of kN/m.

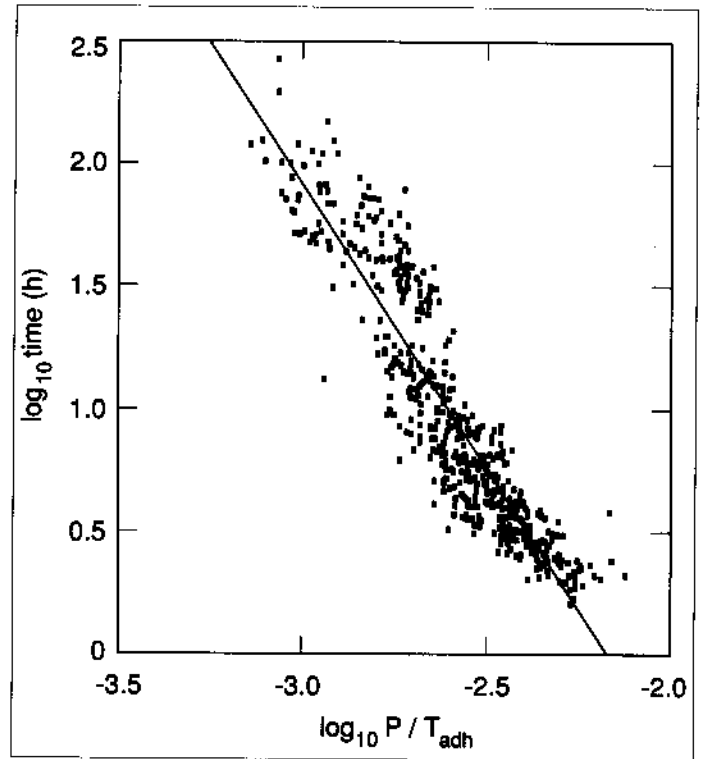


Figure 6 Power law model fit to the 540 times-to-failure from all of the treatments.

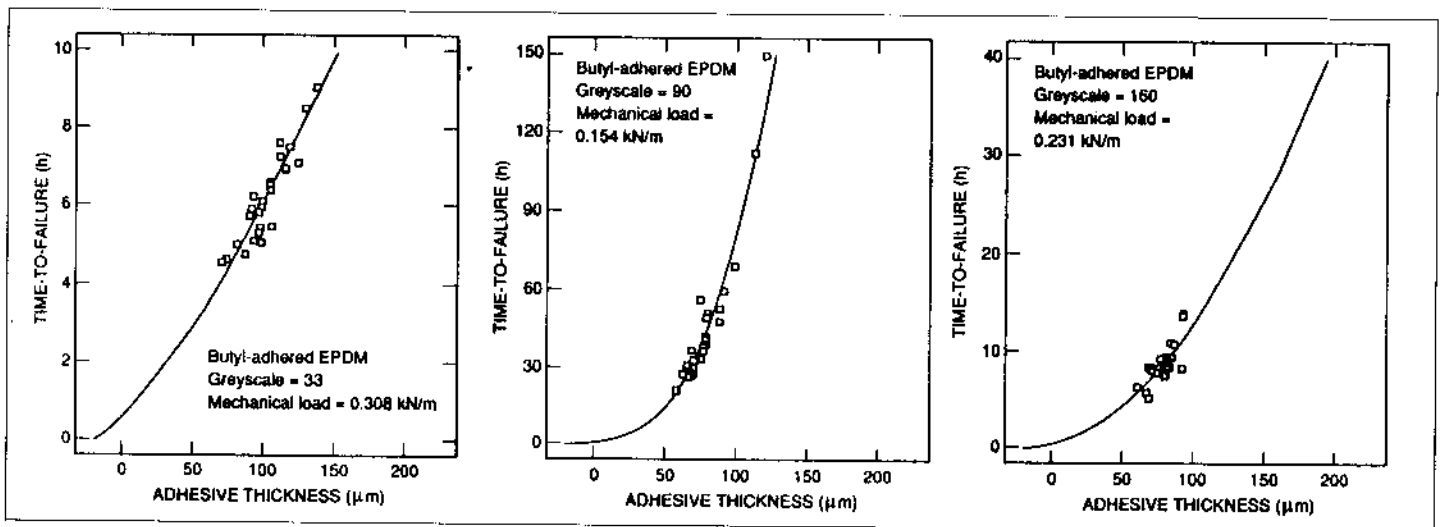


Figure 7 Time-to-failure versus total adhesive thickness for several representative combinations of mechanical load and contamination.