

EFFECT OF CONTAMINATION LEVEL ON STRENGTH OF BUTYL-ADHERED EPDM JOINTS IN EPDM SINGLE-PLY ROOFING MEMBRANES

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ABSTRACT

The most frequently cited failure mode for EPDM (ethylene propylene diene terpolymer) single-ply roofing systems is the failure of the field-formed seams. The cause of these failures has not, as yet, been isolated. The presence of talc-like particles, introduced during the manufacturing process, may be a primary factor, since the presence of these particles may act to block adhesive bond formation. To test this hypothesis, the strength of butyl-adhered EPDM T-peel joints was determined as a function of cure time and contamination level. It was concluded that the strength of an EPDM T-peel joint exponentially decreases with an increase in contamination. These results reinforce the need for strict adherence to proper seam cleaning techniques prior to the application of the butyl adhesive and the need for quality assurance techniques for assuring satisfactory seam formation.

Key Words: adhesive failure, butyl adhesive, cohesive failure, EPDM, Gompertz growth curve, seams, single-ply roofing membranes, surface contamination, T-peel joints.

INTRODUCTION

Single-ply roofing systems have achieved widespread acceptance in the United States due to their satisfactory performance and low installation cost relative to built-up roofs.¹ These systems are not without their problems, however.² In the case of EPDM (ethylene propylene diene terpolymer) single-ply roofing systems, the membrane material has demonstrated excellent weathering resistance properties, but the field-formed seams appear to be vulnerable to premature failure.³⁻⁷ The factors causing these premature seam failures have not, as yet, been isolated, but may include poor surface preparation, chemical and physical aging of the membrane and adhesive and mechanical loads.

Over the last few years, the National Institute of Standards and Technology (NIST) (formerly the National Bureau of Standards) has been conducting research to isolate the factors affecting the in-service performance of EPDM field-formed seams. In 1987, two of the authors⁸ reported on the creep-rupture sensitivity of neoprene-adhered, highly cleaned, EPDM T-peel joints to changes in temperature and mechanical stress. It was concluded from this study that (1) the joints were sensitive to increases in mechanical stress, but not to increases in temperature (at least in the temperature range investigated, 30 C to 75 C) and; (2) mechanical stress was not considered of prime importance in causing

EPDM seam failures, inasmuch as the experimental stresses were much higher than those experienced in the field. Moreover, at low stresses, which were more representative of those occurring in the field, fibers (often called "legs") formed in the adhesive between the separating strips and greatly increased both the strength and creep-rupture life of the neoprene adhered EPDM T-peel joints.

Since the publication of those results, butyl adhesives have largely been replaced by neoprene adhesives as the preferred adhesive for making field-formed seams. During the period over which this change has occurred, the frequency of reported seam problems for single-ply systems has not diminished, as evidenced by comparing the data in Cullen⁹ 1984 to Cullen⁴ 1988.* It seems reasonable to conclude that either the adhesive is not the primary cause of premature seam failure or that the same constituents that caused the neoprene adhesive to fail are present in the butyl adhesive. Since this latter possibility did not seem likely, we focused our attention on the presence of surface contaminants on the EPDM membranes as the prime candidate for causing premature seam failures.

The contaminants most likely responsible for premature seam failure are the talc-like particles (talc, clay or mica) commonly applied over the surface of most commercial EPDM membranes during the manufacturing process to prevent interply fusion of the membrane during vulcanization. The talc-like particles also act to deter interply sticking during the storage and shipment of the cured rubber^{7,9} and to facilitate the movement of the rubber sheets during the roof construction process. These beneficial effects are counterbalanced, however, by the negative impact they have on adhesion.^{5,7,9,10} Thus, there is no question that these talc-like particles need to be removed at the construction site prior to applying the adhesive. What is in question is how clean the surface of an EPDM membrane has to be to form a strong and durable adhesive joint or, to rephrase this question into a more experimentally tractable form, how sensitive the strength of a butyl-adhered EPDM T-peel joint is to surface contamination.

In the process of answering this question, several technical problems had to be overcome, including developing procedures for uniformly contaminating cleaned EPDM

* Cullen's data applies to all single-ply systems. Since EPDM single-ply systems command approximately 60 percent of the single-ply market, the authors interpreted the data to infer that the seam failure statistics for single-ply systems (approximately 25 percent of the reported problems) applies to EPDM systems.

strips and for quantitatively measuring the amount of contaminants deposited on a strip. These procedures are described below.

EXPERIMENT

Materials

A commercial 1.5-millimeter-thick sheet of EPDM rubber and two commercial butyl splicing cements were used in this experiment. The cements (hereafter termed adhesive 1 and 2) were complex blends of several rubbers, the exact compositions of which were unknown, but, according to the manufacturers, were close to the example formulations described in Chmiel and Cotsakis.¹¹ Butyl splicing adhesives are known to cure in the presence of moisture; therefore, to minimize this effect, the adhesives were removed from their shipping containers and poured into a number of smaller containers, each of which was immediately sealed. One of these small containers was opened at the start of each of the 10 experimental treatments (two adhesives times five levels of contamination).

An EPDM sheet was cut into approximately 800 strips, 25 by 160 millimeters. Each strip was cleaned by vigorously brushing both its sides with soap and water and thoroughly rinsing it with water. The cleanness of a strip was quantified using the computer image processing system described below and shown in Figure 1. If the cleanness of a strip did not meet predefined cleanness criteria (see section titled Contamination Level Quantification), it was either re-cleaned or replaced. Once all the strips were cleaned, they were matched into pairs for each T-peel joint; assigned an identification number; and randomly assigned to one of 10 treatment levels (two adhesives times five times contamination levels), each treatment containing 36 pairs of strips (12 cure times times three replicates).

Specimen Contamination

Within each pair, one strip (side A) always remained clean, while the other strip (side B) was contaminated. Contamination was achieved by moving a cleaned side B strip at constant velocity under an atomizer and uniformly spraying it with a well-mixed, low concentration of commercial grade talc-like particles suspended in heptane. Heptane was selected because it is a hydrocarbon solvent, akin to the splice wash solvents commonly used in preparing field seams for adhesion, and because it has other desirable properties, including high volatility and the ability to wet the surfaces of both the talc-like particles and the EPDM strips. The strips were contaminated to one of five levels of contamination corresponding to approximately zero, two, four, six, and eight passes under the atomizer.

Contamination Level Quantification

The level of contamination of each strip was quantified using a computer image processing system consisting of an eight-bit minicomputer-based computer image processor, a computer-controlled X-Y positioning table, a camera and a voltage-regulated fiber-optic tungsten light source (Figure 1). The principle underlying the measurement of cleanness (level of contamination) is that the reflectance of the tungsten light from the surface of a strip increases with an increasing level of contamination. Reflectance was quantified by the computer image processor in terms of greyscale level, where

an absolute black surface has a greyscale value of zero and an absolute white surface has a greyscale value of 256. To standardize the measurement procedure, the intensity of radiation emitted by the fiber optic light source was maintained at 120 ± 0.30 volts by the voltage regulator, and the positions of the camera and optical fiber cables remained stationary throughout the study (the fiber optic cables remained fixed at approximately 15 degrees from the vertical axis of the camera).

The greyscale values of the computer image processing system were calibrated against the quasi-standard Munsell neutral value scale chips,* a sequence of 37 neutral gray painted samples having color values between absolute black (0 percent reflectance) and absolute white (100 percent reflectance) and having a color value difference of 0.25 between chips.¹² A typical calibration curve of greyscale levels versus Munsell color values is exhibited in Figure 2, where the calibration curve is superimposed over calibration data collected on four different days. In the region of experimental interest (that is, greyscale values between 10 and 220) the calibration curve is nearly linear. Above a greyscale value of 220, the camera detector became saturated with light, greatly reducing its ability to differentiate between the white Munsell chip color values.

Greyscale values were also calibrated against talc-like particle deposition. The amount of talc-like particles deposited onto a strip was determined by placing a 22-by-22-millimeter glass cover plate over a portion of a cleaned membrane strip and passing the assemblage under the atomizer. After each pass, the greyscale level of the talc-like particle contaminated area of the strip was measured and the mass of the talc-like particles per unit area was computed by subtracting the mass of the contaminated glass plate from its original mass. These differences were measured to an accuracy of 0.1 milligrams and are recorded on the right axis of Figure 2. At greyscale values below 50, the mass of the talc-like particles was less than 0.1 milligram, so that the actual mass of the deposited talc-like particles was unknown but was between 0 and 0.015 milligrams per square centimeter.

To assure accuracy of the greyscale measurements, the greyscale output from the computer image processing system was checked after every 15 EPDM strips by reinserting one of the Munsell chips under the camera, measuring its greyscale level, and comparing this value against its expected value on the calibration curve. If the greyscale value of this chip fell within ± 1 greyscale level (the most common occurrence), the computer image processing system was considered to be in calibration; otherwise, the intensity of the tungsten fiber optic light source was adjusted until the greyscale level of the chip corresponded with its expected value on the calibration curve.

Greyscale values for each strip were obtained at 20 locations, each having dimensions of 5 by 5 millimeters, within the adhesion area of a T-peel joint as shown in Figure 3. From these 20 sampling areas, the average and standard deviation of greyscale values were computed for each strip. If the standard deviation for a strip exceeded eight greyscale levels, it was replaced with one that was more uniformly sprayed or cleaned.

* Identification is made solely to define experimental materials and not to endorse a particular product.

For the 360 side A strips (the clean side of a T-peel joint), the average and standard deviation of the greyscale values were 33.0 and 2.8, respectively (the maximum observed greyscale value for the side A strips was 36.6 and the minimum observed greyscale value was 27.2). For the 360 side B strips (the contaminated side of a T-peel joint), the average greyscale values were 33, 60, 90, 130 and 160. (Typical greyscale values for uncleaned factory strips ranged from 170 to 220.) To facilitate comparisons between the greyscale values of the side B strips assigned to adhesives 1 and 2, the data are displayed in the form of a multiple box-and-whisker plot¹³ in Figure 4. The central box of each box-and-whisker diagram covers the middle 50 percent of the data values between the lower and upper quartiles. The central, horizontal line within the box is the median greyscale value at each level of contamination while the vertical lines, extending out from the top and bottom sides of the box, extend to the extremes (minimum and maximum values) of the data. The mean greyscale values at each level of contamination for adhesives 1 and 2 were also compared using analysis of variance techniques. From this analysis, we concluded that there were no significant differences in the mean greyscale values for the strips assigned to adhesive 1 and 2 at each of the five contamination levels.

Specimen Preparation

Prior to applying the adhesive, the mass of each strip in a pair was determined. The adhesive was then applied with a brush onto the clean strip (side A), then the contaminated strip (side B), after which the brush was discarded. This procedure was adopted to minimize the transfer of talc-like particles between strips. Once the adhesive was spread over both strips, the solvent was allowed to evaporate at laboratory conditions (approximately 22 C and 45 percent relative humidity) for approximately 30 minutes. The strips were then joined and pressed together in a hydraulic press at 700 kPa for 10 seconds forming a T-peel joint.

Replicate T-peel joints were cured to one of 12 preassigned cure times (0.5, 1.0, 1.5, 2, 4, 6, 24, 48, 96, 192, 768 and 1,320 hours). The cure time for a T-peel joint began as soon as the T-peel joint was removed from the press.

The strength of three replicate T-peel joints was determined at each cure time and at each contamination level. The strength of a T-peel joint was determined in peel at a machine speed of 50 millimeters per minute. The separated strips were then weighed and the fraction of the adhesive adhering to the clean strip (side A) and the contaminated strip (side B) determined by subtracting the initial mass of each strip from its final mass. If the adhesive was evenly distributed, the failure mode was assumed to be cohesive; if most of the adhesive adhered to the uncontaminated (side A) strip, the failure mode was assumed to be adhesive.** To supplement this determination, the failure mode of the separated strips was visually categorized as cohesive, adhesive or a mixture of cohesive and adhesive. By comparing these visual assessments of failure to their corresponding percent mass data, it was determined that in general a percent mass of 60 percent corresponded to co-

hesive failure, a percent mass of 75 percent corresponded to a mixture of cohesive and adhesive failures and a percent mass of 90 percent corresponded to adhesive failure.

RESULTS AND DISCUSSION

Percent mass of the adhesive adhering to the clean side (side A) of the separated T-peel joints versus cure time data are plotted in Figures 5 and 6 for adhesives 1 and 2, respectively. For T-peel joints composed of two cleaned strips (greyscale = 33), the joint always failed cohesively. For T-peel joints using adhesive 1 and containing a contaminated strip, the joints failed cohesively for cure times less than two hours; failed by a mixture of cohesion and adhesion for cure times between two and six hours; and failed by adhesion for cure times greater than six hours. For T-peel joints using adhesive 2 and containing a contaminated strip, cohesive failures were not observed at cure times greater than one-half hour, and instead, most of the failures were by a mixture of cohesion and adhesion for the first six hours of cure and by adhesion for cure times greater than six hours. The major difference between the two adhesives, therefore, was that adhesive 1 took a longer time to set up than did adhesive 2—two hours as opposed to less than one-half hour.

Strength data at the five levels of contamination are plotted versus cure time and contamination level in Figures 7 and 8 for adhesives 1 and 2, respectively (a more complete presentation, including raw data, is given in Martin, et al.¹⁴). Each curve can be divided into two periods. The first period starts when a T-peel joint is removed from the press (time zero on the graph) and lasts for cure times up to two hours for adhesive 1 and less than one-half hour for adhesive 2 (see Figures 7 and 8). The failure mode during this period was almost always cohesive due to the low strength of the adhesive at these early cure times; as such, strength did not depend on contamination level. During the second period, the strength of the T-peel joints was highly dependent on contamination level and the failure mode was almost always by adhesion.

The observed strength data, S , for T-peel joints made using the butyl adhesives, which cured over time, were fitted to a Gompertz growth curve model¹⁵ (equation 1 and the solid lines in Figures 7 and 8) for each of the five experimental contamination levels. The Gompertz growth curve model has the form as follows:

$$S = ka^{b^t} \quad [1]$$

or

$$\log S = \log k + (\log a)b^t \quad [1a]$$

where

t is the cure time,

k , a and b are coefficients where $0 < a < 1$ and $0 < b < 1$.

This model was found to fit the data well for both adhesives and at all five levels of contamination. The coefficients have the significance: a is related to the strength of the T-peel joint when first removed from the press; b indicates the rate of change in T-peel strength with cure time, which can be shown by taking the logarithm of eq 1a; and k is a measure of the maximum strength. The significance of the coefficients is briefly discussed below and in more detail in Martin, et al.¹⁴

** The locus of adhesive failure was expected to be between the butyl adhesive and the contaminated side B strip because that is where the talc was applied. For T-peel joints that failed by adhesion, most of the adhesive was expected to remain on the uncontaminated side A strip.

For a T-peel joint adhered with adhesive 1 and composed of at least one contaminated strip, the time-dependent strength of the T-peel joints was a mixture of two Gompertz growth curves: the first curve modeled the cohesive strength of the T-peel joints for times up to and including two hours, and the second curve modeled the strength of the T-peel joints for cure times greater than two hours. The parameters of the first curve were independent of the level of contamination, in that the same parameters could be used to model both the clean and the contaminated T-peel joints; the values of the parameters for the first curve are given in Table 1 for adhesive 1 at a greyscale level of 33. After the first two hours, another Gompertz growth curve model was fitted to the T-peel strength data. The parameters for these curves are tabulated in Table 1 at greyscale values of 60, 90, 130 and 160.

Besides fitting the strength data well,¹⁴ the Gompertz growth curve model supports many of our experimental observations. For example, at $t = \text{zero}$, eq 1 becomes

$$S = ka \quad [2]$$

which is the minimum strength of a T-peel joint or, equivalently, the strength of a T-peel joint when it is first removed from the press.

$$\text{As } t \rightarrow \infty, \text{ eq 1 becomes } S = k \quad [3]$$

the maximum strength of the adhesive joint. The minimum and maximum strengths, along with Gompertz parameters a and b are plotted in Figures 9 and 10 versus greyscale level for adhesives 1 and 2, respectively. The minimum strength, ka , (remember for adhesive 1, the initial strength was the same for all five nominal levels of contamination) and the value of parameter b remained relatively constant over the range of contamination levels studied (the average value for parameter b was 0.94). The maximum strength of the T-peel joints decreased with increasing levels of contamination. This decrease corresponded with relatively low levels of talc-like particle deposition (see Figure 2).

The significance of a constant minimum strength indicates that the strength of the T-peel joints was controlled by the cohesive strength of the butyl adhesive. A constant b parameter value for both adhesives indicates that the rate of increase in strength does not depend on the level of contamination. The decrease in the maximum strength of the T-peel joint with small increases in the amount of talc-like particles deposited indicates that the ultimate strength of a T-peel joint is highly dependent on contamination level. These results reinforce the need for strict adherence to proper seam-cleaning techniques prior to the application of the butyl adhesive and the need for quality assurance techniques for assuring satisfactory seam formation.

CONCLUSIONS

The sensitivity of butyl-adhered EPDM T-peel joints to contamination by talc-like particles was experimentally determined for two commercially available butyl adhesives and one commercially available EPDM membrane material. From our experimental results, we make the following conclusions:

- The maximum EPDM T-peel strength is achieved when the strips comprising a T-peel joint are thoroughly cleaned.
- For contaminated joints and for cure times less than two hours, the strength of a contaminated T-peel joint does not depend on contamination level; instead, the strength of the joint is controlled by the cohesive strength of the adhesive.

- For contaminated joints and for cure times greater than two hours, the strength of a butyl-adhered EPDM T-peel joint is highly dependent on contamination level.
- A Gompertz growth curve appears to fit the strength versus cure time data well for both adhesives and for all five levels of contamination. The properties of the Gompertz growth model helped to predict strength and explain many of our experimental observations.

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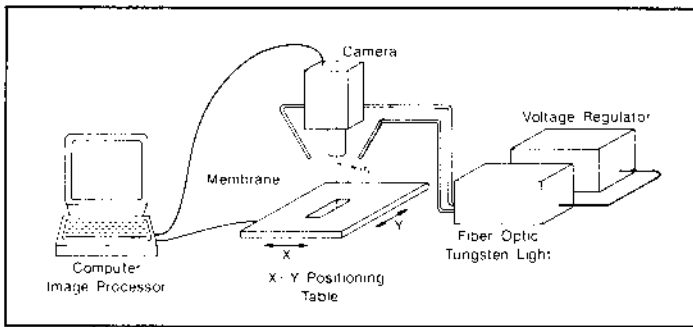


Figure 1 Schematic of computer image processing system for quantifying the greyscale of an EPDM T-peel strip

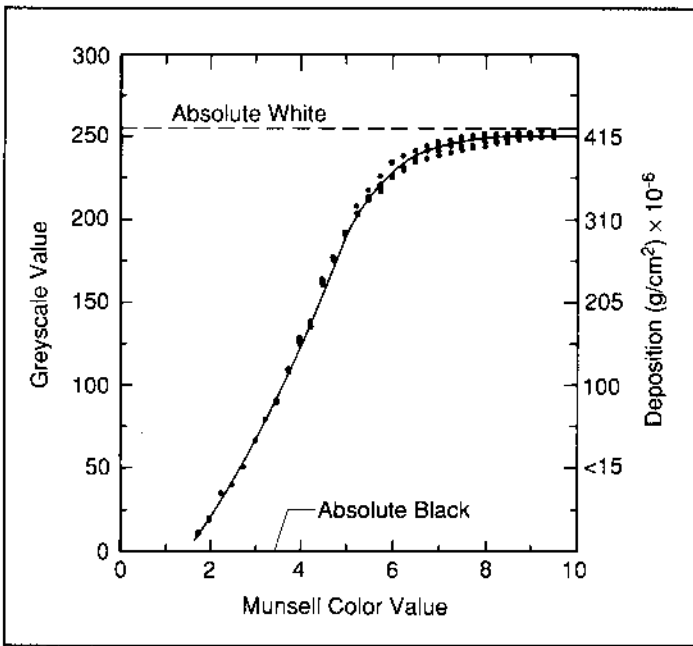


Figure 2 Calibration curve of greyscale values versus Munsell neutral color value. Right axis is in terms of talc-like particle deposition per unit area (g/cm^2) at different greyscale values.

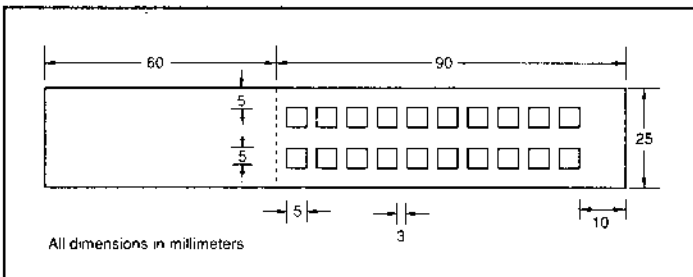


Figure 3 Location of 20 sampling areas on a membrane strip

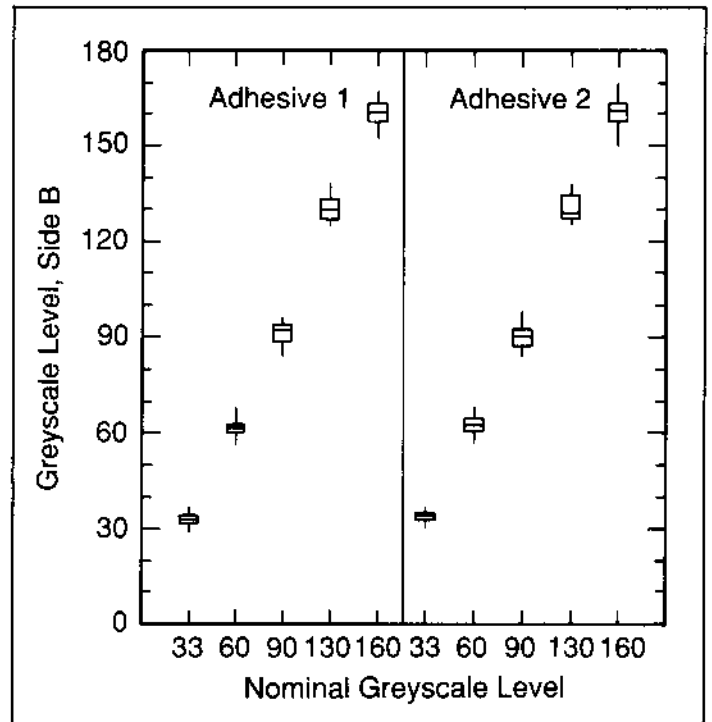


Figure 4 Box-and-whisker plot of greyscale values for the contaminated side B strips assigned to adhesives 1 and 2, which were contaminated to one of five nominal contamination levels.

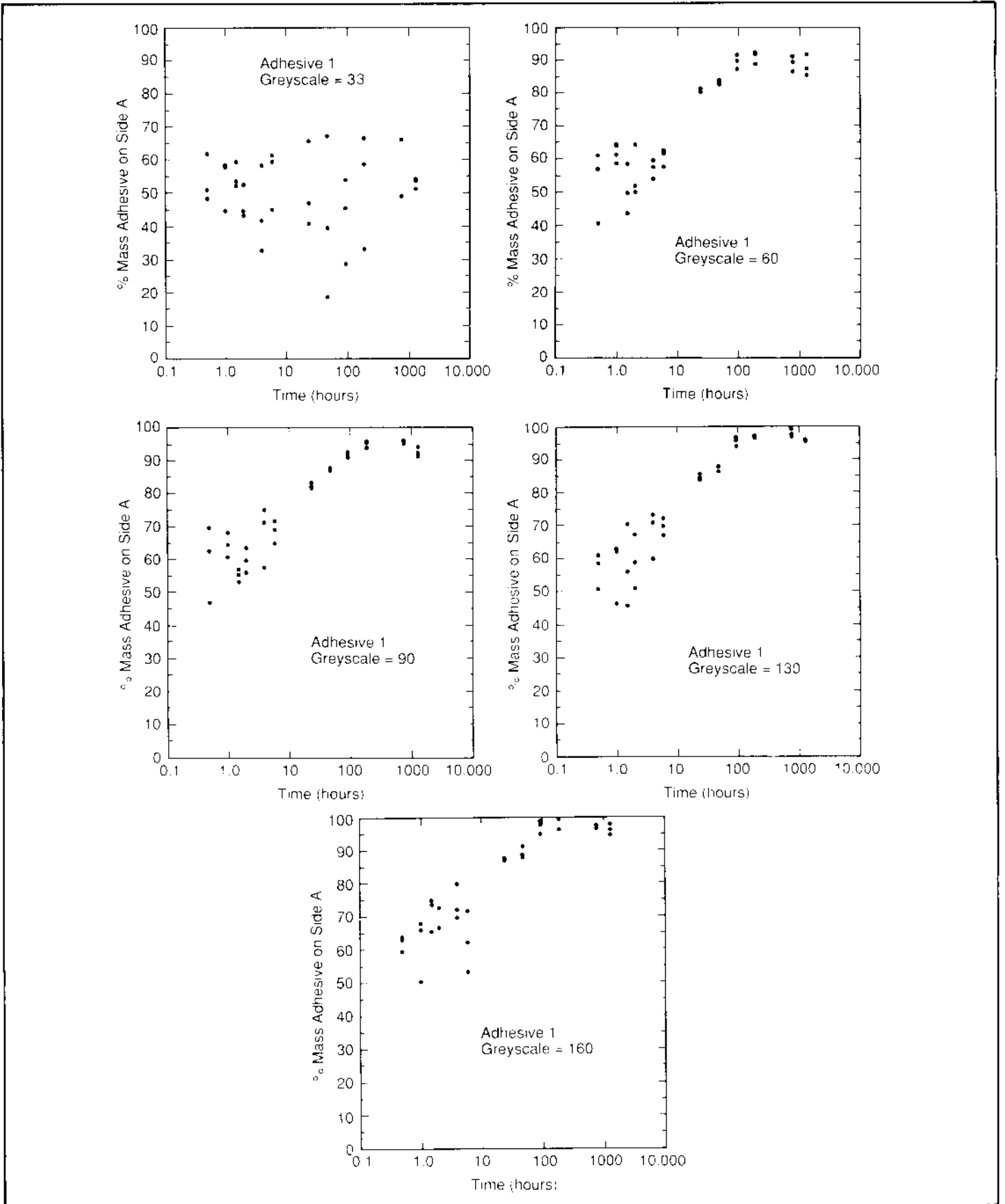


Figure 5 Proportion of adhesive adhering to the clean side of a failed T-peel joint at different cure times and contamination levels for adhesive 1

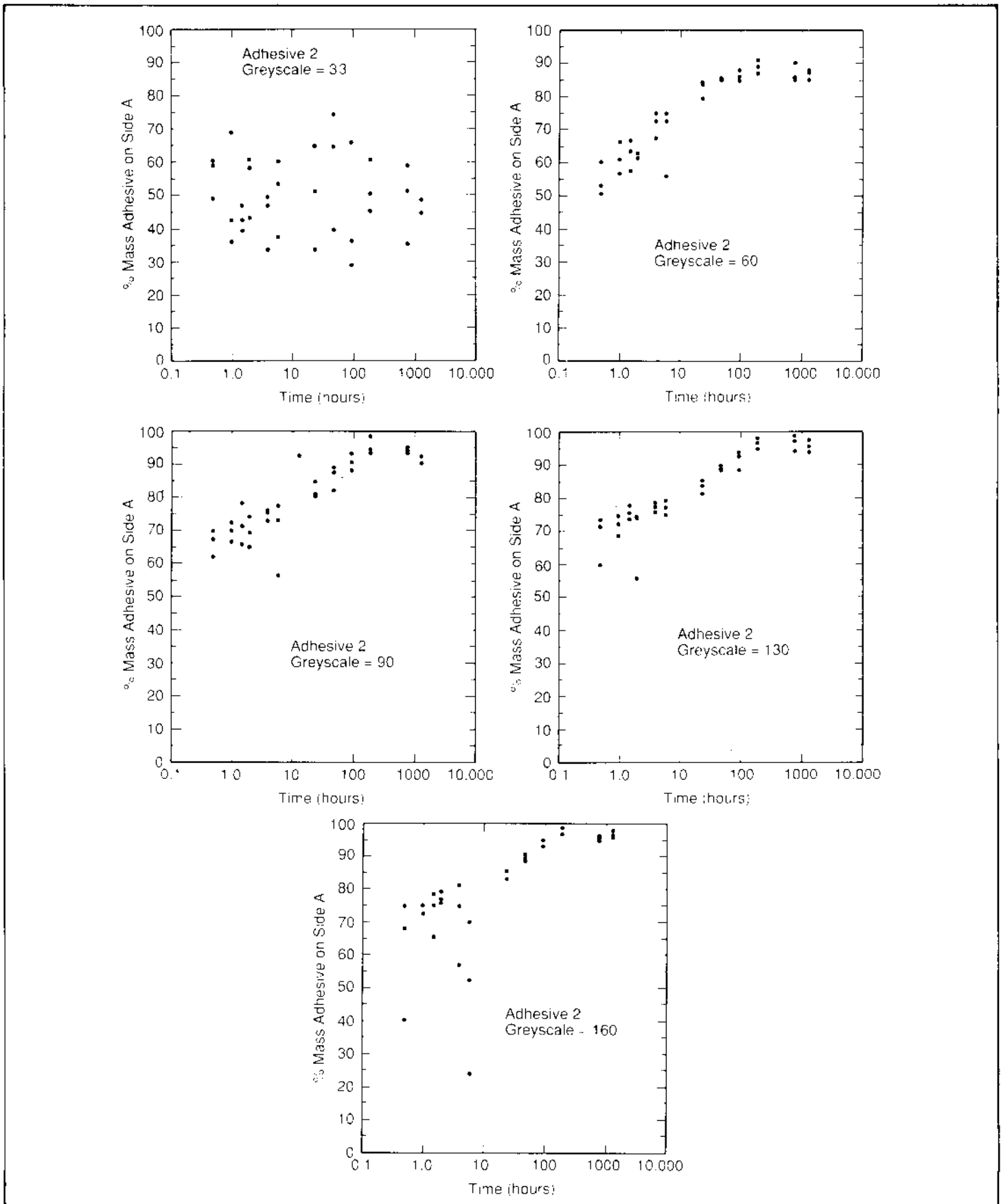


Figure 6 Proportion of adhesive adhering to the clean side of the failed T-peel joint at different cure times and contamination levels for adhesive 2. T-peel joints having a greyscale value of 33 are the controls

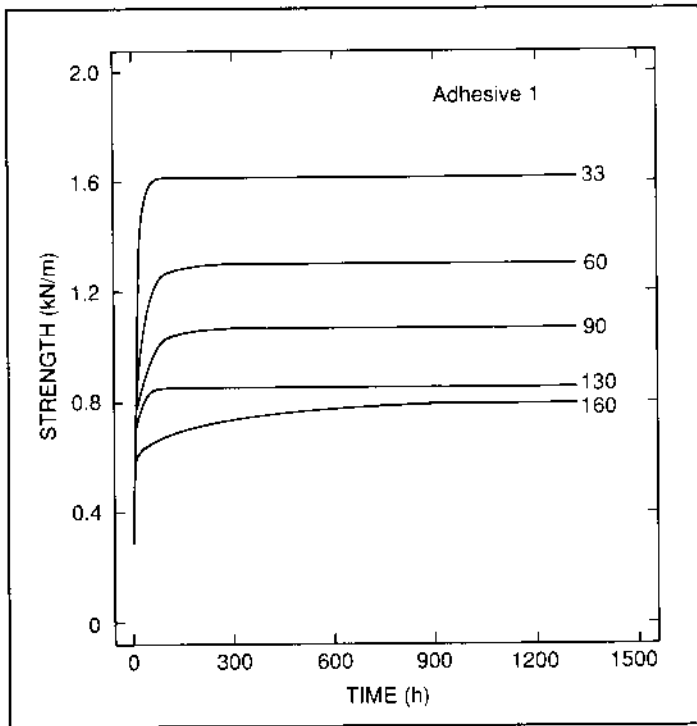


Figure 7 T-peel strength versus cure time for adhesive 1. T-peel joints having a greyscale value of 33 are the controls

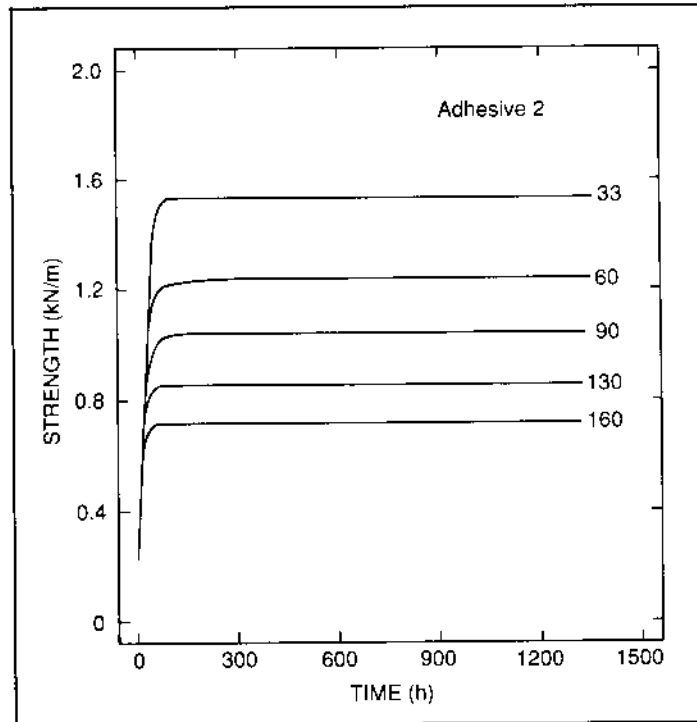


Figure 8 T-peel strength versus cure time for adhesive 2. T-peel joints having a greyscale value of 33 are the controls

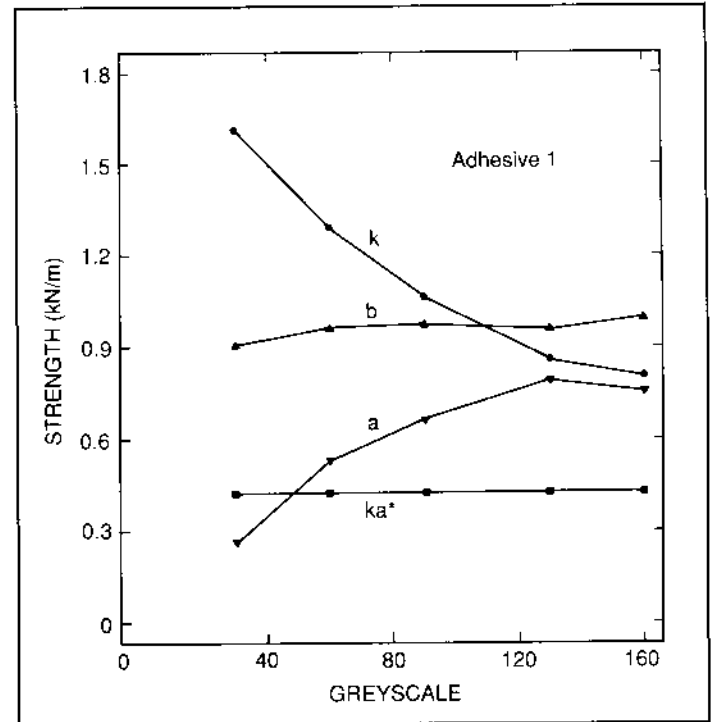


Figure 9 Gompertz growth curve parameters versus nominal contamination level for adhesive 1, where the parameter k equals the ultimate strength and ka equals the minimum strength of a T-peel joint. For adhesive 1, the minimum strength was the same at all five nominal contamination levels

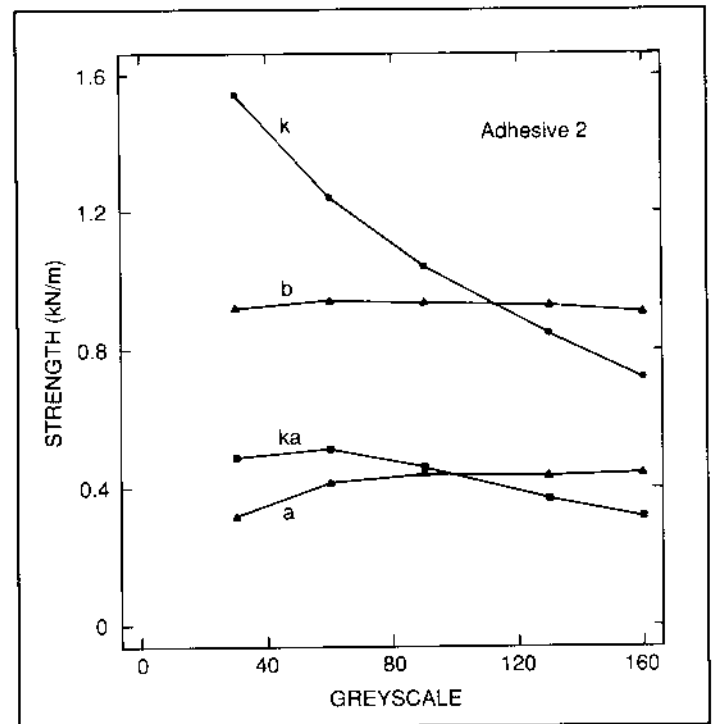


Figure 10 Gompertz growth curve parameters versus nominal contamination level for adhesive 2, where the parameter k equals the ultimate strength and ka equals the minimum strength of a T-peel joint

Adhesive	Average Greyscale	Gompertz Growth Curve Parameters			r^2
		k	a	b	
1	33	1.612	0.260	0.905	0.96
	60*	1.287	0.560	0.963	0.92
	90*	1.062	0.662	0.974	0.80
	130*	0.860	0.790	0.958	0.51
	160*	0.830	0.753	0.995	0.49
2	33	1.546	0.315	0.921	0.96
	60	1.243	0.414	0.944	0.94
	90	1.042	0.438	0.938	0.89
	130	0.949	0.433	0.929	0.89
	160	0.717	0.439	0.910	0.80

* Gompertz growth curve parameters for these nominal greyscale levels are for cure times greater than two hours (see text).

Table 1 Gompertz growth curve parameters for adhesives 1 and 2 along with the squared correlation coefficient, r^2 , at all five nominal greyscale levels.