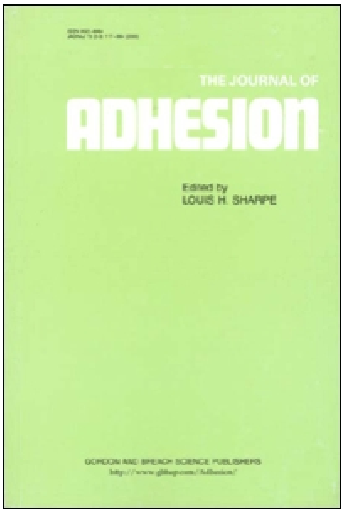

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Discussion of Bond Strength Test Results in Terms of Mathematical Statistics

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INTRODUCTION

In the course of the evaluation of bond strength test results, the following observations are made:

1) The results show a relatively high scattering.
2) The number of lower values exceeds the number of higher values.
3) The tensile strength per unit area depends on the size of the bonded area.

These findings will be discussed in terms of mathematical statistics. This seems to be the more important since those observations are the reason for the relatively high number of samples demanded by testing standards and for standardizing the size of the bonded areas.

In rubber industry, analogous findings are well known. They lead to statistical evaluations, e.g. by Kase[1] with quite a good success.

Our idea was to use this knowledge for the interpretation of bond strength test results. This intention is justified by the fact that the breakage mechanism in both cases, rubber and adhesive bonded test specimens, is the same: the breaking process starts at the weakest points caused by little defects. The treatment of these cases in the light of the extreme value theory should therefore lead to analogous results.

EXPERIMENTAL

For our purposes brass to brass bonds according to ASTM D 897–68 (Tensile Properties of Adhesive Bonds) and ASTM D 1002–64 (Strength Properties of Adhesives in Shear by Tension Loading, Metal-to-Metal) have been used. The adhesive applied was a high viscosity version of cyanoacrylate.† This adhesive belongs to the class of reactive adhesives. They are applied as monomers and polymerize very rapidly *in situ* when the specimens to be bonded are brought into close contact. Setting times are in the order of 2 to 5 seconds.

The test specimens were pretreated in the following way: Burrs were removed carefully. The specimens were degreased by treating them with perchloroethylene in an ultrasonic bath for 5 to 10 minutes. This treatment was followed by an etching process according to DIN 53281/1 (Pickling Process). The solution used was a mixture of 27.5% H_2SO_4, 7.5% $Na_2Cr_2O_7 . 2 H_2O$, and 65% H_2O (percentages by weight). The test specimens were placed in a mixture of one part (vol.) of fresh solution with nine parts (vol.) of used solution for 5 minutes at room temperature. The specimens were rinsed with distilled water and dried at 30 to 35 °C. The specimens treated in this way have to be bonded within the next 2 hours.

The tensile test specimens were prepared using a simple apparatus which allowed an exact arrangement of the specimens to be bonded and the application of a defined contact pressure. The bonding area amounted to 6.46 cm^2. An adhesive drop of constant size was placed on one of the specimens to be bonded and spread over the bonding area by use of a polyethylene spatula. The adhesive layer must be as uniform and thin as possible. The specimens to be bonded were put together immediately and loaded with 4.52 kg (= 0.7 kg cm^{-2}) for 2 minutes. The bonded test specimens were then removed and stored for 24 hours at 23 °C and 50% R.H. before being tested.

The tensile shear test specimens were prepared in the same way. The bonding area amounted in this case to 1×0.5 $inch^2 \mathrel{\hat{=}} 3.23$ cm^2, the load applied for 2 minutes to 2.26 kg ($\mathrel{\hat{=}}$ 0.7 kg cm^{-2}).

Also these tensile shear tests were performed after a storage for 24 hours at 23 °C and 50% R.H.

These rather laborious pretreatment and bonding processes have been chosen in order to ensure that the test results are as precise as possible and the expected effects are not covered by poor precision. The tensile and tensile shear tests were performed using an Instron Testing Machine at a crosshead speed of 10 mm/min.

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† Bostik Chemical Group, USM Corporation.

Tensile strength

Twenty-five bonds were prepared and tested. In order to check if these values belong to a Gaussian (normal) distribution they were plotted on a Gauss paper (Figure 1). It can be seen that the plotted values do not lie on a straight line as would have been found in the case of a normal distribution. The values, on the contrary, indicate concave curve which is typical for a negatively skewed distribution. One distribution of this type is the Kase distribution, which is based on a double exponential function $F(x) = (-\exp(-x))$. If we plot our 25 test values on a Kase paper we should find them on a straight line if our interpretation of the Gauss plot is correct. Figure 2 shows that this is the case.

Before drawing any conclusions from this fact, it would be advantageous to have a short discussion of the properties of normal and skewed, especially negatively skewed, distributions for those readers who may not be familiar with these functions.

The two distribution curves in question are given as rough drafts in Figure 3.

A normal distribution is characterized by two values: the mean μ for characterization of the position of the center of distribution and the standard deviation σ for the characterization of the width of this distribution. The distribution of numbers is formed by the observed test results. The range $\mu-\sigma$ to $\mu+\sigma$ includes 68 % of the values. μ and σ can be calculated only from an infinite number of values. Limited numbers of values lead to approximations $\bar{X}$ for μ and s for σ. s can be converted into confidence levels using Student's t factors.

In the case of a skewed distribution the measure of location can be characterized by three values: The mean, the median and the mode. The mean is the usual arithmetic mean; the median is the central value in the row of values arranged according to their amount (as many values below the median as above); and the mode is the most probable value (i.e. the abscissa of the maximum). In the case of a negatively skewed distribution the median lies between the mean and the mode. The width of the distribution may be characterized also by the standard deviation s, but it does not have the quantitative meaning as in the case of a normal distribution. It is especially important that the confidence limits are not calculated in the same manner as for a normal distribution. Mean, median and mode are identical in the case of a Gauss distribution.

The question arises which of the values, mean, median or mode, shall be declared as tensile strength when having tested, for instance, 5 specimens. The most frequently given value is the mean—but is it the best characterization of the distribution curve that stands behind those 5 values? The

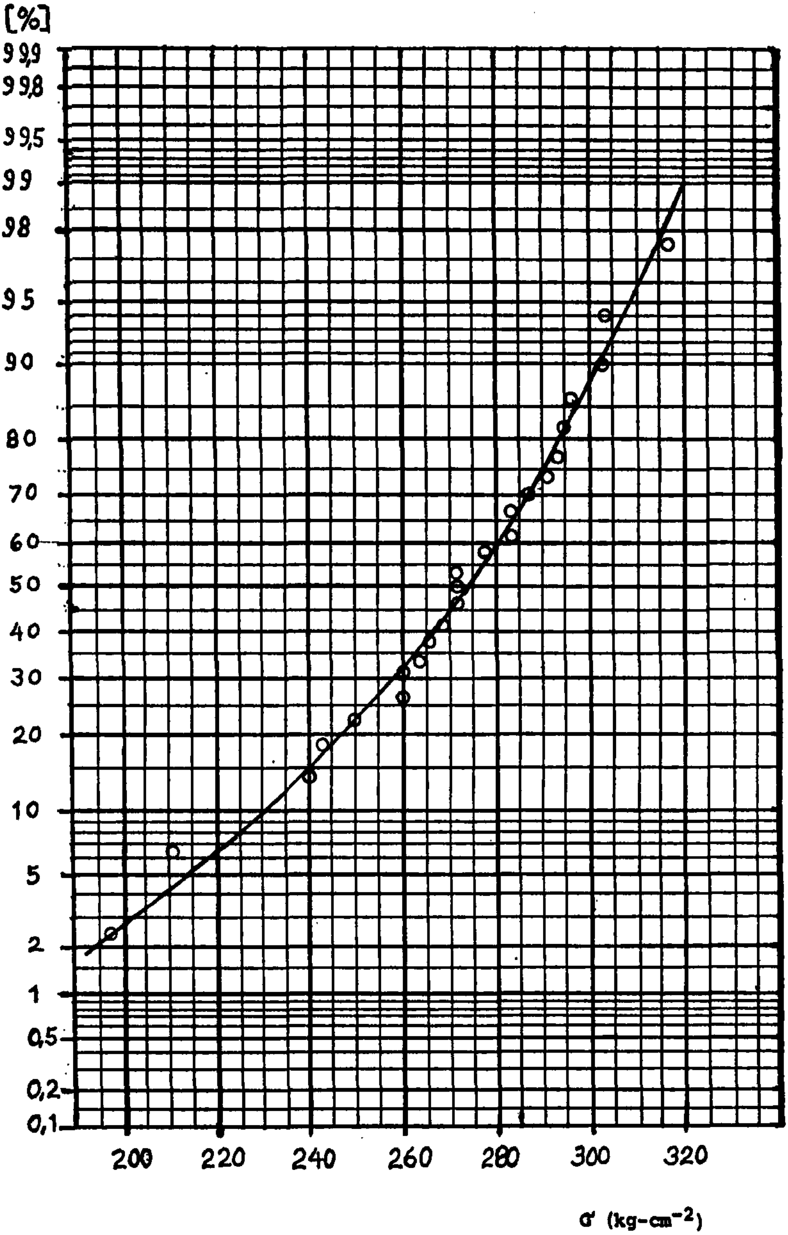

FIGURE 1 Tensile strength values plotted on a Gauss paper.

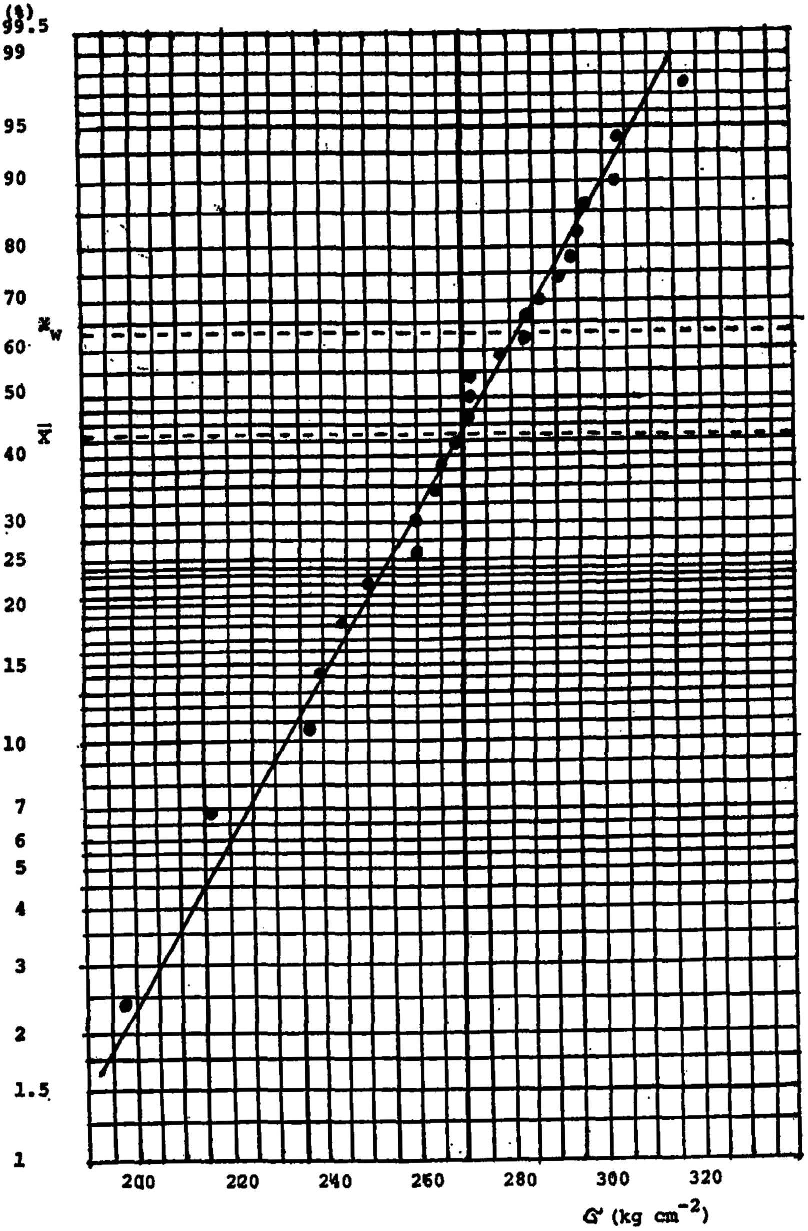

FIGURE 2 Tensile strength values plotted on a Kase paper.

answer is no. The mean is too low and the variance too great (see Figure 3) because of its sensitiveness to the low values. The mode represents the most probable value and therefore should be able to give a more realistic measure of location. The true mode can be calculated only from an infinite number of values. For practical work approximations—called estimators—can be calculated which are based on a restricted number of values.

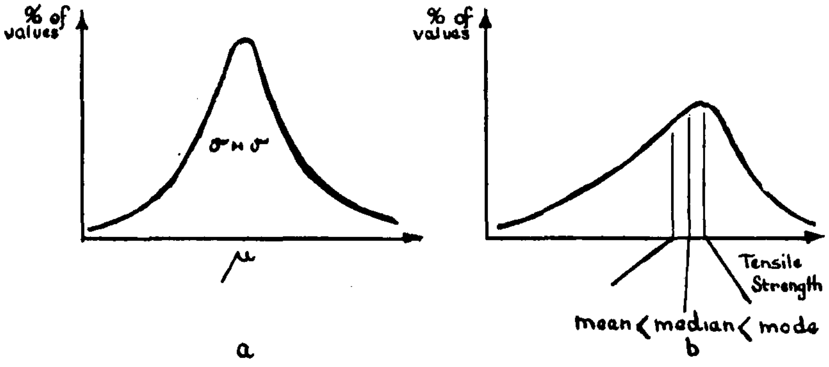

FIGURE 3 a) Normal (Gaussian) distribution.
b) Negatively skewed Kase distribution.

Heap[2] showed that estimators based on the mode give results with the lowest coefficient of variation, estimators based on the mean give the highest coefficient of variation and estimators based on the median give a coefficient of variation between those two coefficients. Consequently we should prefer the use of an estimator based on the mode for the characterization of the tensile strength.

Heap gives different estimators for the mode based on publications of Kase. These estimators may be applied, of course, also in our case, since we were able to show that our test values obey to the Kase function. These estimators are formed by taking the weighed sum $X_M = \Sigma\, a_i\, x_i$ where the weights a_i are tabulated and the x_i are the test values arranged in descending order of magnitude $x_i \geqslant x_2 \geqslant x_3 \geqslant \ldots$. In the case of three observations a more simple though not so efficient estimator for the mode may be formed by averaging the highest two values.

We usually perform 5 parallel tests in the course of tensile strength measurements. We found out that an estimator formed by averaging the three highest values gives quite good results which differ not more than 2 rel. % from those results which are based on the use of the exact weights a_i given by Kase. Since calculation of the mean of the three highest values is more simple and accuracy is good enough for practice we prefer this simplified estimator.

Tensile shear strength

Twenty-five bonds with an overlap of $\frac{1}{2}$ inch were prepared and tested. The type of distribution of the values was checked by plotting on both Gauss and Kase paper (Figures 4 and 5). Also in this case the values conform to the Kase function.

The conclusions which may be drawn from this result are the same as in the case of tensile strengths.

Dependence of bond strength of size of bonded area

Higuchi, Leeper and Davis[3] gave the following expression for the dependence of rubber tensile strength on the volumes of the specimens tested:

$$-d\sigma = k \,.\, \frac{dV}{V} \tag{1}$$

i.e. the decrease of tensile strength $d\sigma$ is proportional to the relative increase of the value dV/V. This equation has to be understood in the light of a statistical consideration of the breakage process.

Applying equation (1) to the tensile strength of bonded specimens, we can write

$$-d\sigma = k \,.\, \frac{dA \,.\, h}{A \,.\, h} \tag{2}$$

with A = bonded area (cm^2).

dA = increase of bonded area (cm^2).

h = thickness of adhesive layer, assumed to be constant.

The influence of the bond area upon the unit bond strength may be attributed to several conditions, among which are:

1) Local stress increases with the bonding area and causes decreasing bond strengths per unit area.

2) The number of microscopic defects of the bonding increases with the bonding area and increases the probability of breakages at lower bond strengths per unit area.

Integration of Eq. 2 leads to

$$\sigma = \sigma_0 - k \,.\, 2.303 \,.\, \lg\frac{A}{A_0} \tag{3}$$

with σ_0 = Tensile strength of the test specimen with the smallest bonded area A_0.

σ = Tensile strength of the test specimen with the bonded area A.

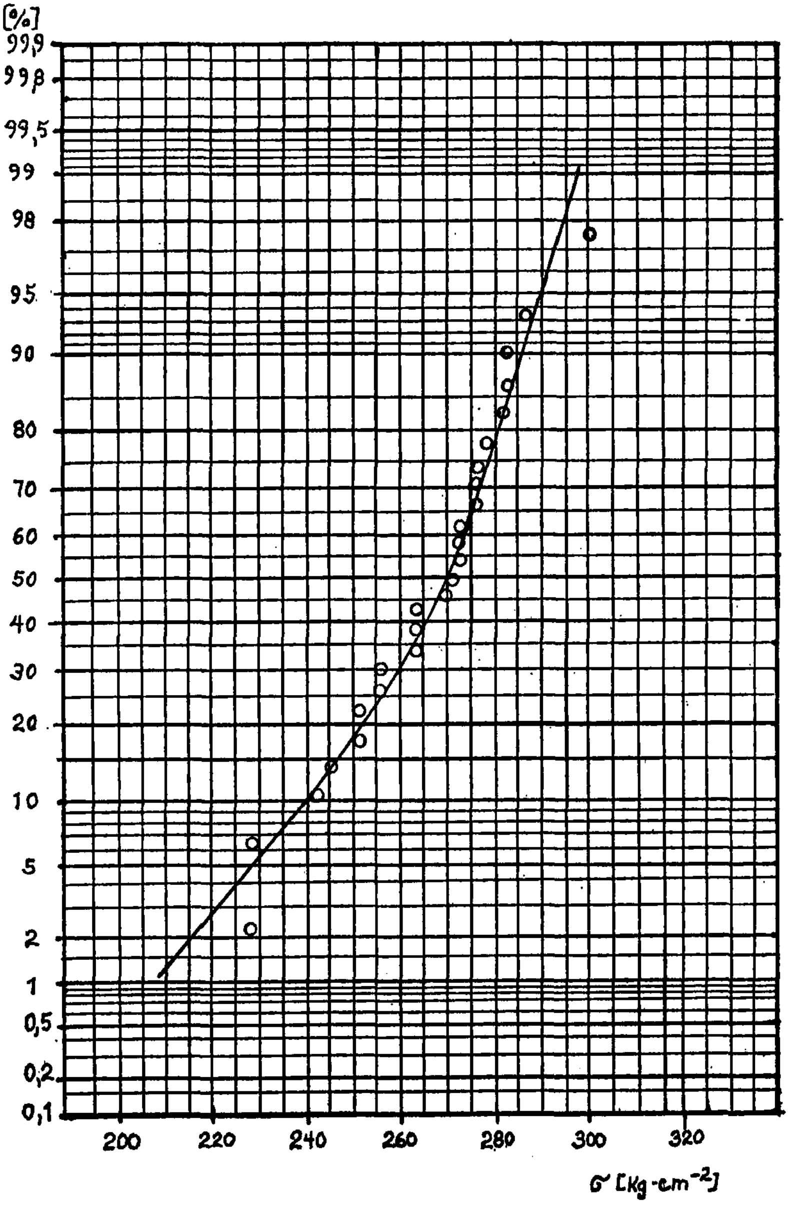

FIGURE 4 Tensile shear strength values plotted on a Gauss paper.

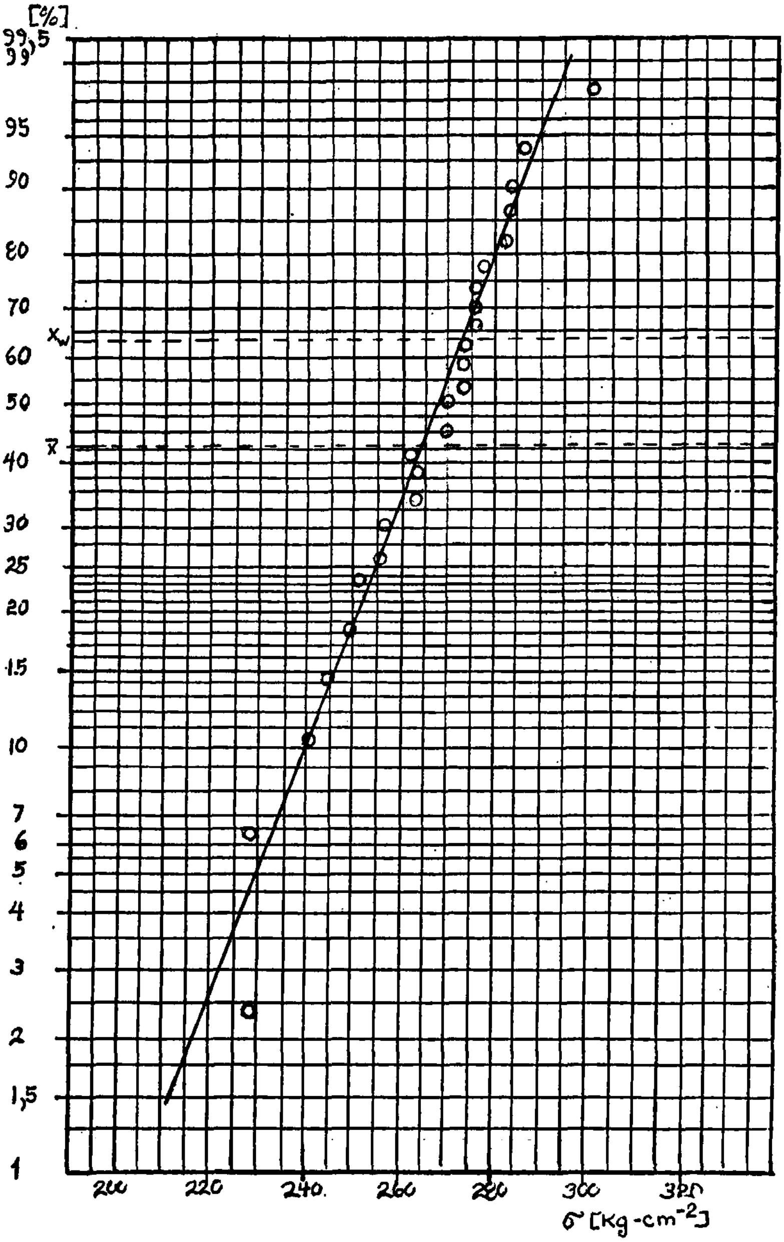

FIGURE 5 Tensile shear strength values plotted on a Kase paper.

Equation (3) demands a linear relationship between σ and $\lg(A/A_0)$, which was checked experimentally.

Tensile strength test specimens were prepared with different sizes of areas to be bonded: 1.96; 3.43; 4.95 and 6.29 cm^2. The general shape of these test specimens compiled with the ASTM Standard D 897. The material of specimens was brass and the surfaces were pretreated as mentioned above.

Five parallel tests were performed; the modes of these test results were plotted against $\lg(A/A_0)$, A_0 being 1.96 cm^2.

ASTM tensile shear test specimens were prepared with different overlaps: ¼", ½", ¾", 1" and 1 ¼ inch. The material of test specimens was brass and the surfaces were pretreated as mentioned above. Five parallel tests were performed; the modes of these test results were plotted against $\lg(A/A_0)$, A_0

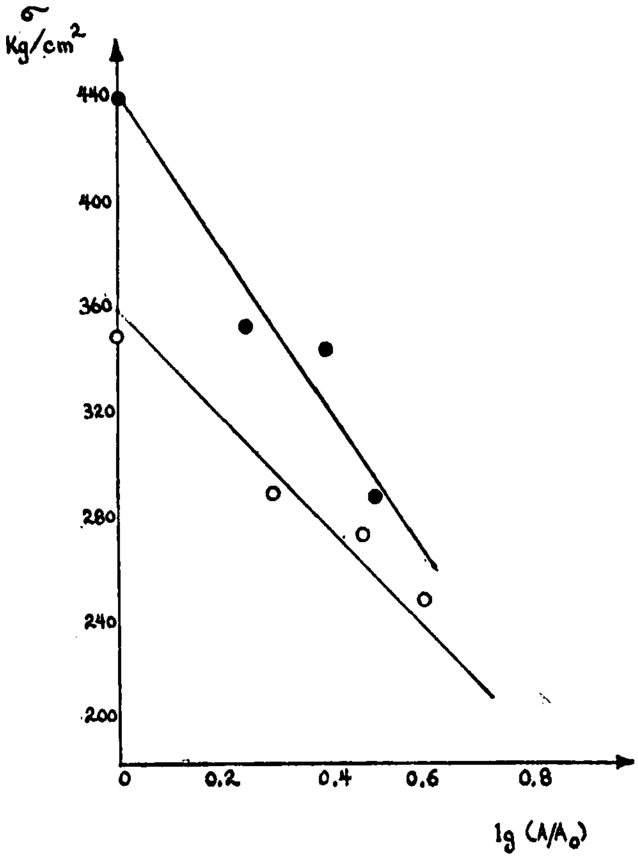

FIGURE 6 Dependence of Tensile Strength (●) and Tensile Shear Strength (○) on Bonded Area.

being in this case $\frac{1}{4}\times 1$ square inches = 1.61 cm^2. The results are shown in Figure 6.

A statistical evaluation of these test results leads to the following:

	Coefficient of linear correlation	Linear correlation at confidence level
Tensile strength	$r = 0.95$	95%
Tensile shear strength	$r = 0.98$	99%

It can be stated therefore that the linear relationship demanded by Eq. (3) has been confirmed experimentally.

SUMMARY

It is shown that tensile strength and tensile shear strength test values of adhesive bonds do not conform to the Gaussian normal distribution, but to the negatively skewed Kase distribution. As a consequence of this, the bond strength is preferably characterized by the mode instead of the mean. The standard deviation can be used for characterization of the variance of test values only with certain restrictions. A mathematical relationship between tensile strengths or tensile shear strengths per unit area and the size of bonded areas is given.

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