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Flex Life and Long-Term Strength of Composite Materials

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Synopsis

The relationship is shown between the flex life and long-term tensile strength of flexible composite materials. The long-term strength is interpreted in terms of a kinetic theory of failure. However, it is not possible to predict the flex life from tensile strength or even long-term strength data unless the stiffness or modulus is considered along with the thickness of the sample. With these factors it becomes possible to predict the flex life of a material and to compare various flex-testing instruments utilizing a new concept, which we call "effective force." The usefulness of the concept of effective force is explained.

INTRODUCTION

For several years we have worked on tests that could be used to predict the long-term usefulness of flexible materials, including foamed structures, laminates, microporous plastics, and impregnated webs. These studies have had one common element: the difficulty of relating the results of laboratory tests to performance during actual use. The present work was done during an evaluation of porous cellulose and polymer webs, which were being considered as components of synthetic shoe upper material. We examined two types of flex-testing machinery to see whether they could predict wear life in shoes or in other applications in which long flexural life is important.

There is no question that the best test is a use test, since the sample fails at the right time by the right mechanism. However, there are many drawbacks to use testing, such as the time, the wide scatter of field test data, and the availability of enough samples.

Substitution of a laboratory test can be successful, but only to the extent to which the laboratory test approximates the end use of the product. There have been many studies of the flex life of materials,[1-18] and the conclusions are varied. In many cases flex tests have not correlated at all with actual performance, because the test did not duplicate the geometry or force of the flexing during use. We were faced with this problem in testing our flexible composite webs. The main question in the use of such machines is whether they will give the same action that will be encountered in use. We have used creep failure measurements to predict the useful lifetime of materials in tension with great success, but in these cases the force acting on the material is known. In flexed samples the geometry is very complicated, and the actual force at work on the sample is unknown. We thought it should be possible to relate lifetime in tension to lifetime during flexing, thereby increasing the amount of information which could be obtained from both types of test.

Correlation of field test results with tensile properties or with data from an MIT fold tester was not satisfactory; in fact, the materials that appeared strongest by this method often failed first when worn in shoe uppers. The MIT fold flex endurance test operates at a rate of 150 flexes per minute with a load of 1,000 grams on the sample. For a sample 0.03 in. thick this is a static load of about 150 psi and is much higher while the sample is flexing because of inertial forces that could not be readily analyzed. There is also a heat buildup in the sample in the MIT test.

Since the MIT test was not appropriate for our use, we examined other types of flex testers. This work led to the concepts that are the subject of this paper.

SAMPLES

The samples for this study were hand sheets made in a Valley sheet mold. They were bulky, porous webs made from chemically and mechanically modified cotton linters having a sheet density of approximately 0.3 g/cm^3. These were dry-saturated by being dipped into a nitrile rubber latex. A percentage of 77–94 of the free volume was filled by the rubber.

Variables in the samples were the degree of beating before and after chemical treatment and rubber uptake.[19,20] After saturation the samples were oven-cured at 193°C for 2 min to cure a urea–formaldehyde additive. Some "fiber lubricants" were also present in the latex. These composite materials were reasonably soft, feeling stiffer than Corfam but softer than most leathers.

EXPERIMENTAL

Creep Testing

Creep failure data were obtained by hanging a load on the sample and waiting for it to break. The time to fail was detected by the falling weight's tripping a wire, which stopped a clock (Fig. 1). This method was used to determine the time to fail up to 2 wk. After that a failure was noted by daily inspections. This test is now ASTM D2648-67T. Graphs of the logarithm of time to fail versus force were linear. There was a mechanism change, as evidenced by the two straight-line segments in each graph.

Flex Testing

The Bally G-Flexometer-160 (Bally Shoe Factories Ltd., Schoenewerd, Switzerland) is a flex tester that operates at a rate of 100 flexes per minute.

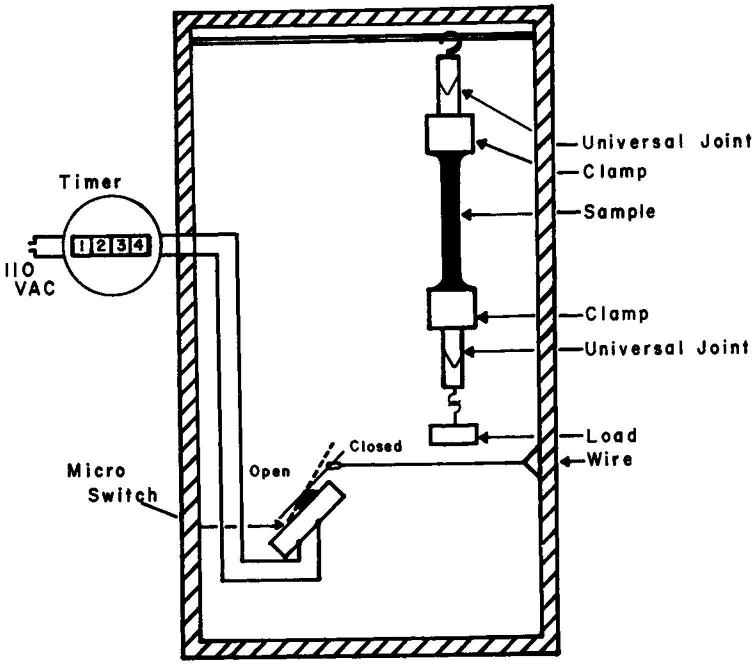

Fig. 1. The creep failure apparatus. When the weight falls, the wire is pulled off the microswitch which stops the time clock. This is on version of the ASTM D 2648-67T creep failure test.

It gives a rolling flex motion, which is shown in Figure 2. Samples were folded as shown in Figure 3. The Bally Flex Tester gives a reciprocating motion to the upper clamp, moving it back 2.2 cm and down 1.0 cm. The samples were periodically inspected at logarithmic intervals of a factor of 2 between inspections, until they had been on the flex tester several days; then they were inspected morning and evening. After 2 wks they were inspected daily. Failure was evidenced by a crack or, more usually, a hole in the area of sharpest curvature.

The other flex tester used in this study was a Lin Tronics No. W4 (Lin Tronics Co., Burlington, Mass.). It was patterned after an International Shoe Co. test machine and gave a different geometric flexing action to the sample than the Bally Tester. This machine flexed 90 times per minute; it is shown in Figure 4. Samples are 3 $\times$ 4 in. rectangles, which are folded in half and clamped into the machine. As the machine is started (by hand for the first flex), the sample is guided into the shape shown in Figure 4. The motion of the movable side is from an angle of 180°–145°C. The distance between the clamps is 5 cm, and in the flexed position it is 2.5 cm.

Neither machine gave an appreciable temperature rise to the sample as measured either by thermocouple or by touch. The Lin Tronics tester was also run for a short period at 250 flexes per minute, and this did give a measurable temperature rise ($\approx$10°C). However, no acceleration in

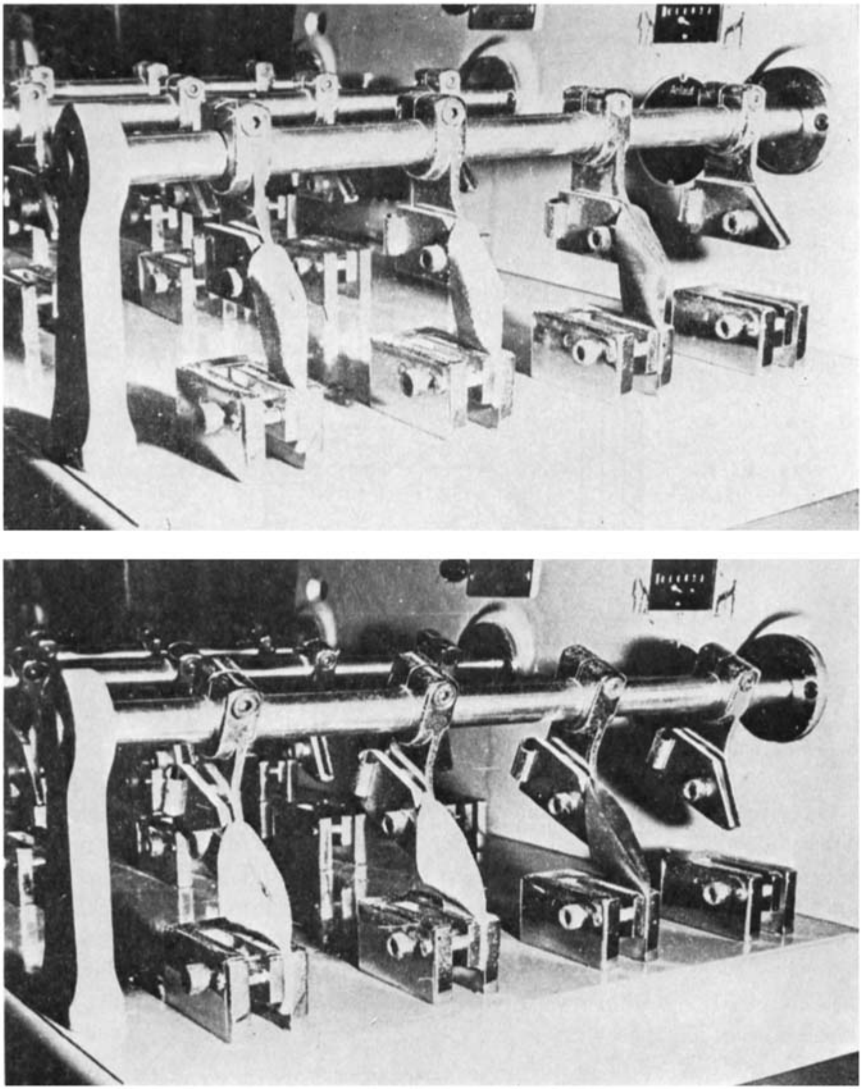

Fig. 2. The Bally Flexometer in the open and closed positions. There is a rolling flex motion at the tip of the sample. Failure usually occurred at the point of sharpest curvature which is shown in the upper part of the photograph.

failure was noted, so all the flex testing was done at 100 flexes per minute with the Bally Flex Tester, and the Lin Tronics Flex Tester was run at 90 flexes per minute. Duplicate runs on both machines gave a reproducibility of about a factor of 2, which somewhat more scatter if the sample failed during the first night of operation, when it could not be observed by an operator. Both machines gave a rolling type of flex action.

Results are expressed in time to fail rather than cycles, since Coleman[21,22] has shown that time to fail under cyclic loading is independent of frequency.

His conclusion is that a cyclic load may be approximated by a static dead load by the use of a zeroth-order, inverse, hyperbolic Bessel function. This is analogous to the independence of power in alternating-current voltage in relation to frequency. This is true as long as there is no appreciable heat buildup; the latter is discussed by Tauchert and Afzal[23] and Riddell et al.[24]

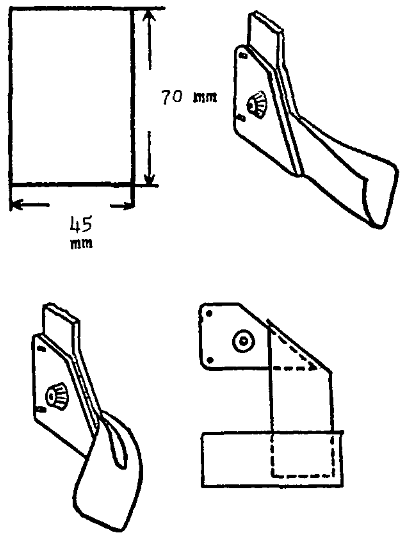

Fig. 3. The sample for the Bally Flex Tester; how it is folded and placed in the flexometer.

In our thin samples heat buildup is not appreciable, because of thinness, pumping action of the air around the sample, and moderate flex rates.

Modulus

Modulus was calculated from the Gurley stiffness measurements of the samples, which take into account the thickness of the sample. The Gurley stiffness is a good indicator of modulus if thickness is absolutely uniform. Since our samples did not have uniform thickness, the modulus had to be used.

Gurley modulus was calculated by a method developed by Biggs and Jacobs,[25] in which the following equation is used:

$$E = WRGL^3/(5{,}670T^3w \sin \theta_2)$$

where W is weight used, R is distance from axis, G is Gurley dial reading, L is length of sample, T is the sample thickness, w is width of sample, and θ is the angle formed by the loading arm and lever arm.

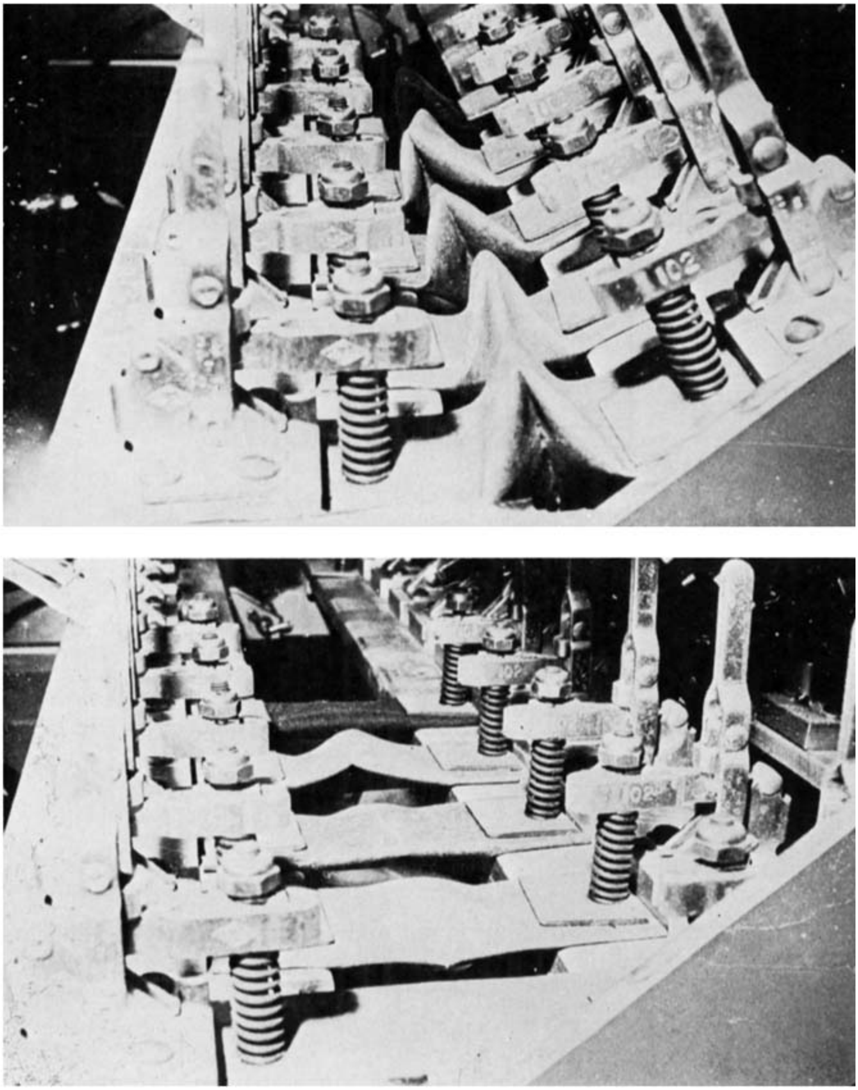

Fig. 4. The Lin Tronics Flex Tester in the open and closed positions. A rolling motion of the flex was observed for most of the samples tested, although it was not present in all cases.

RESULTS

Creep failure curves were plotted as log time to break versus force. Figure 5 shows two typical curves. It is especially to be noted that for these materials short time test results, such as tensile property measurements, can give highly misleading results for applications that require long-term properties. All graphs showed two straight-line segments. This indicates a mechanism change for failure.

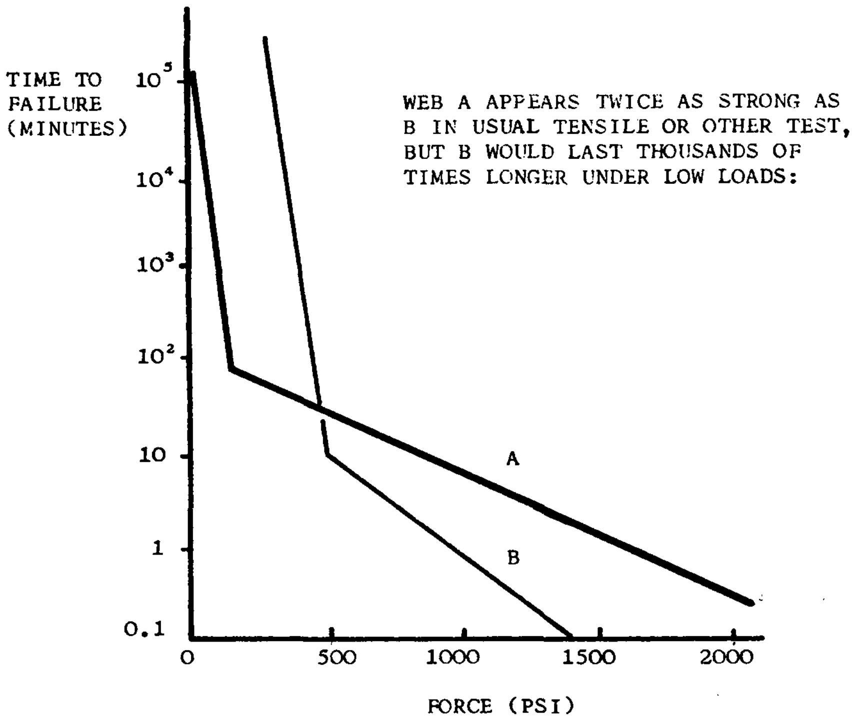

Fig. 5. Fallacy of using short-term tests to predict long-term performance. Typical creep failure curves showing how easily one could be misled by short time single point data.

Coleman[21,22] and others[26,27] have shown that failure can be described by kinetic theory. The failure curves have the form

$$\ln t_f = a - bF$$

where a is a constant, F is the applied load, and t_f is the time to fail. Coleman's derivation gives significance to the terms a and b:

$$a = \gamma_B h/\lambda kT \exp \{\Delta F/RT\}$$
$$b = (1 + E_b)\delta/2KT$$

where $\gamma_B h$ is the creep not recoverable in a slow stress strain test, λ is the separation between the positions of minimum potential force, h is Planck's constant, T is the temperature in degrees Kelvin, $1 + E_b$ is a term that corrects for the change in cross-sectional area, k is Boltzman's constant, δ is the size of the volume element in motion for the flow process, R is the gas constant, and ΔF is the free energy.

At 23°C these equations can be simplified by rearrangement and condensation to

$$\delta = (2.73 \times 10^{-18})/\bar{B}(1 + E_b)$$

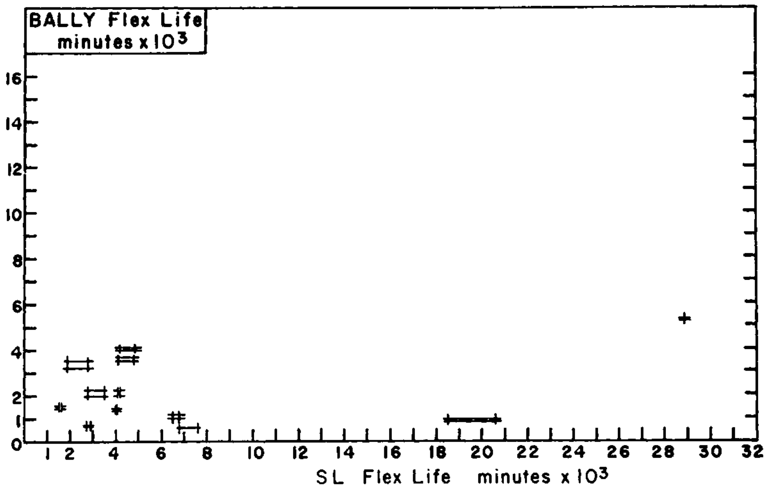

Fig. 6. The Bally flex life plotted against the Lin Tronics flex life. The lack of correlation is probably due to the inconsistent geometry of the flexed area in the Lin Tronics Tester.

where $\bar{B}$ is in pounds per square inch per decade, δ is in cubic centimeters, and ΔF is 1.354 log $3.696 \times 10^{14}a$, in which a is the zero force intercept in minutes and ΔF is in kilocalories.

The mechanism changes observed occurred anywhere between 1 min and 1 hr, so a satisfactory, simulated use test should have a failure time greater than 1 hr.

The creep failure curves of the samples used in this study were very interesting in themselves and gave new information about failure processes in cellulosic webs, which will be the topic of another paper.[28] Subsequent work has led to identification of the two main failure processes as viscoelastic flow of the fibers (at low forces) and failure of the interfiber bonded regions (at high forces).

In the high-force interfiber mechanism the free energy of activation has a range of 22–28 kcal, depending on the degree of bonding, so it appears that six hydrogen bonds are breaking simultaneously. In the low-force intrafiber mechanism the values are from about 33 kcal in highly purified pulp to an upper limit of about 51 kcal in heavily beaten pulp or when wet-strength agents are present. These free energies are equivalent to a minimum of eight and a maximum of twelve hydrogen bonds.

Displacement volumes calculated from the slopes of the lines are highly sensitive to web bulk density and to the degree of stress distribution obtained by beating or latex saturation. When the webs are considered as single fibers or, that is, when slopes are adjusted to what they would be if all webs had the density of the fiber wall, displacement volumes for the interfiber mechanism are 1 to 1.5×10^{-21} cm^3, or about two unit cell volumes. In the intrafiber mechanism values are much higher, about 20 or 30×10^{-21} cm^3, and may represent slippage of a whole microfibril.

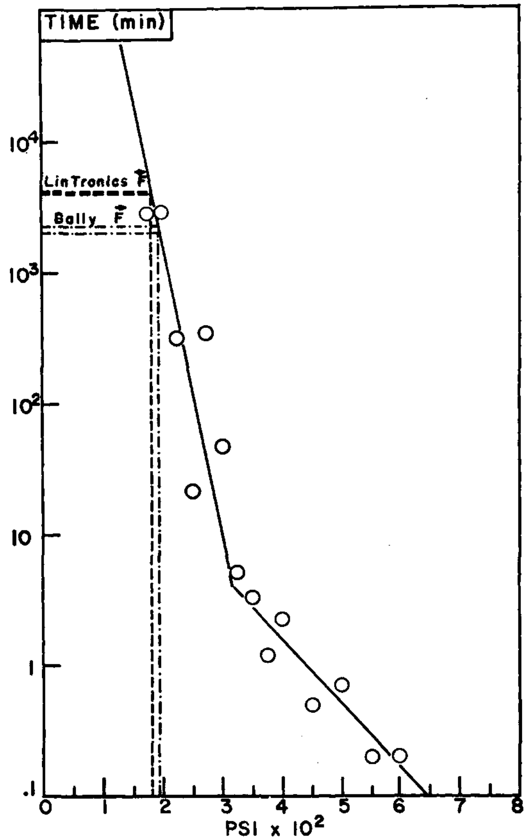

Fig. 7. Determining the effective force F. The life time in the flex test is entered on the graph on the time axis and the "effective force" is then determined from the force axis.

These results are consistent with recent work of others on the structure and failure processes in paper. The details of this work and the effects of process variables and latex saturation will be described later.

The results discussed above are applicable only to load bearing in tension. Flexural lifetime is another problem. Figure 6 shows the flex life of the Bally Tester plotted against the flex life measured on the Lin Tronics tester. There is little correlation, because the geometry of the flexed regions are dissimilar and samples vary in thickness.

In examining the geometry we gave up trying to calculate what was going on. Coleman[22] indicated that periodic forces can be approximated by a dead load. Having determined the creep failure curves and the flex life time, we thought that we could combine these measurements and experimentally estimate the "effective force" on the samples produced by the flexing machine. By using the creep failure curve the time to fail in flexure is entered on the curve and the corresponding effective force is read. This is shown by the dashed lines in Figure 7. The utility of this approach lies in the fact that the effective force can be estimated when it cannot be calculated. We have used this principle previously in a study of poly-

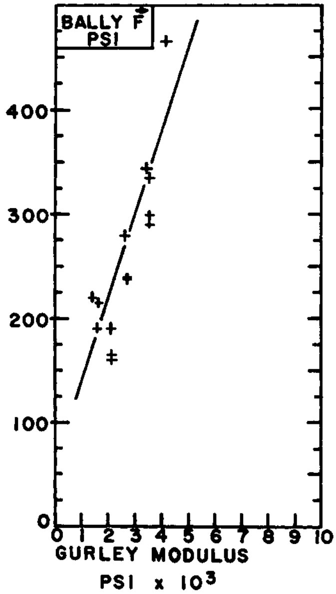

Fig. 8. The effective force for the Bally Flexometer plotted against the Gurley modulus. This indicated that the effective force is very sensitive to stiffness.

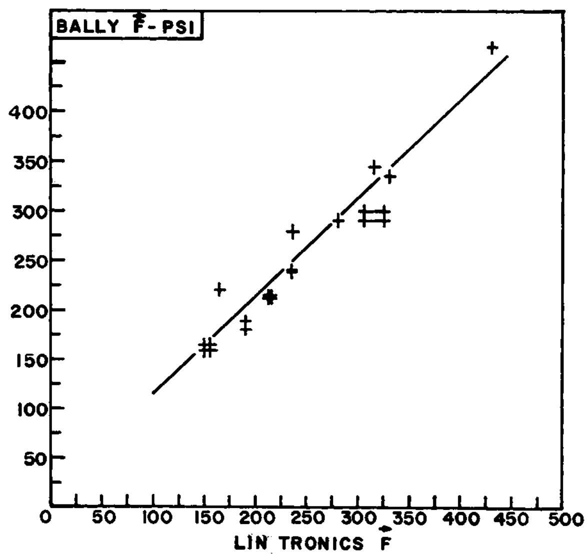

Fig. 9. The Bally "effective force" plotted against the Lin Tronics "effective force." This shows that both machines give a comparable "effective force."

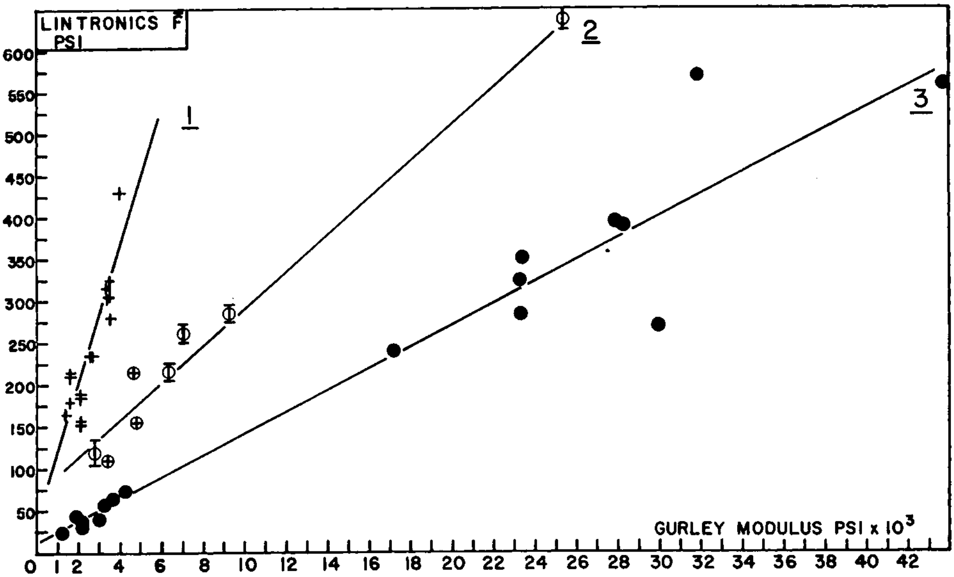

Fig. 10. The Lin Tronics "effective force" plotted against the Gurley modulus. Three lines are present for different classes of materials. These materials also had different thicknesses, which suggested a correction of the equations for thickness.

ethylene bottles under a static pressure, when the geometry would not allow the calculation of the stress.[29] When calculation was possible, as for cylindrical bottles, the effective force equaled the hoop stress.

Even though one material may be stronger than another in tension, as determined by a creep failure curve, it may fail sooner when flexed, because it has a higher effective force F. Figure 8 is a plot of the effective force F of the Bally Flex Tester versus the modulus determined by a Gurley stiffness tester. This was thought to be significant, since the force in flexure must be transmitted to the failure point by the stiffness of the material. There is a good relationship between the effective force and the modulus of the material. Figure 9 is a plot of the Bally effective force versus the Lin Tronics effective force as determined similarly. Here, a good correlation exists with little scatter.

Effective force for the Lin Tronics tester is also dependent on the class of material, as may be seen in Figure 10. The exact reason for this was not certain. We thought that thickness might have been a part of the reason, since the materials had different thicknesses. Figure 11 is a plot of failure lifetime versus thickness for one material, where only the thickness was varied. This may be described empirically by the relation

$$\log F = 2.29 \log X + 5.133 \quad \text{or} \quad F = 1.36 \times 10^5 X^{-2.29}$$

where F is the failure time and X is the thickness of the sample in mils. By using this relationship the values for c in the equation

$$\mathbf{F} = G - cX^{-2.29}$$

were calculated from the modulus and the thickness by means of the -2.29 exponent. For the first set of samples shown in Figure 8, the only case in which both Bally and Lin Tronics flex life data were available, values of c were calculated from the failure time $\mathbf{F}$; G is the Gurley modulus and X is the thickness in mils. For the Bally Flex Tester this gave the equation

$$\mathbf{F} = G - 2.18 \times 10^7 X^{-2.29}$$

with a standard deviation of 6.81×10^6 for the set of samples labeled 2 in Figure 10.

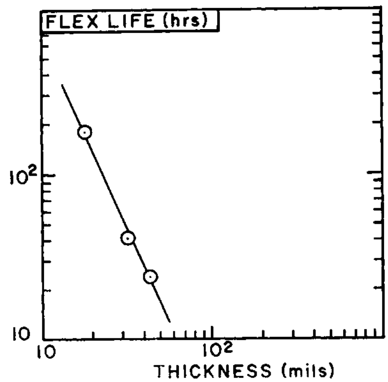

Fig. 11. Effect of thickness where thickness was the only variable. Flex life depends on the thickness to the -2.29 power, showing the necessity for correcting for the thickness of the samples.

The relationship for the Lin Tronics Flex Tester was found to be

$$\mathbf{F} = G - 1.219 \times 10^7 X^{-2.29}$$

with a standard deviation of 9.2×10^6. For the set of samples labeled 3 in Figure 10,

$$\mathbf{F} = G - 2.296 \times 10^7 X^{-2.29}$$

with a standard deviation of $c = 2.2 \times 10^7$. These standard deviations, though they seem large, contain a summation of all the errors of all of the measurements, including flex life, Gurley modulus, and those involved in drawing the creep failure curve. Much of the error is in the creep failure curve, which is quite steep in the region used. The flex life data are good to about a factor of 2 because of reliance on a visual inspection at periodic intervals. The good correlation shown in Figure 9 suggests that most of the residual error was the result of uncertainty in drawing the curves of creep failure time versus force. These are notoriously poor, as may be seen in the literature.[1–18]

Effective force, as described here, contains the variables of modulus and thickness. It may well contain other variables that we did not identify, such as a slight temperature rise and polyaxial forces, which we know to be present, but with constant machine geometry these variables may be treated as a single constant.

SUMMARY

1. A correlation was established between the lifetime of flexible materials in flexure and creep failure data; in a sense, flex life on a given machine may be considered one point on a creep failure plot in the same manner that a tensile test is.

2. We have provided a demonstration that lifetime in flexure is independent of cycling rate and can be approximated by an equivalent dead load.

3. Once the effective force in flexing has been determined for a given flex tester and a given type of material, it can be used, together with the flexural modulus and thickness of other samples of similar materials, to predict flex lives of similar samples. This method is especially useful when one wishes to know the effective force and the effective force cannot be calculated directly because of geometric considerations.

4. The two testers evaluated were not appreciably different, in that the effective force in each method gave an equivalent dead load.

The samples were prepared by J. Wrzesinski. The physical tests were performed by W. Holman, J. Berzanskis, D. Bryant, W. Callis, and J. Arnreich. Valuable assistance in this work resulted from many fruitful discussions with John Dash, Richard Hoch, Harold M. Sonnichsen, and Robert W. Penn. Mrs. C. Jenkins and Miss E. J. Skelly provided valuable assistance in digesting the data and in drawing the figures.

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