**Designation: D 4482 – 99**

# Standard Test Method for
# Rubber Property—Extension Cycling Fatigue[1]

## 1. Scope

1.1 This test method covers the determination of fatigue life of rubber compounds undergoing a tensile-strain cycle. During part of the cycle, the strain is relaxed to a zero value. The specimens are tested without intentionally initiated flaws, cuts, or cracks. Failure is indicated by a complete rupture of the test specimen.

1.2 No exact correlation between these test results and service is given or implied. This is due to the varied nature of service conditions. These test procedures do yield data that can be used for the comparative evaluation of rubber compounds for their ability to resist (dynamic) extension cycling fatigue.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[2]

D 3767 Practice for Rubber—Measurement of Dimensions[2]

D 4483 Practice for Determining Precision for Test Method Standards In the Rubber and Carbon Black Industries[2]

2.2 *British Standard:*

BS5324 Guide to Application of Statistics to Rubber Testing[3]

## 3. Description of Terms Specific to This Standard

3.1 *extension ratio*—the ratio of the extended length of a specimen, $L$, to the unextended length, $L_o$, calculated as follows:

$$\lambda = \frac{L}{L_o} \tag{1}$$

3.2 *fatigue life (sample)*—the mean value of the number of cycles required to cause failure for a number of specimens of the sample.

3.3 *fatigue life (specimen)*—the total number of cycles required to cause failure of a specimen, and is defined as a complete rupture or separation of the specimen.

3.4 *strain energy*—the energy per unit of volume required to deform the specimen to the specified strain. It is measured by the area under a stress-strain curve and expressed in kJ/m3 (see Annex A1).

## 4. Summary of Method

4.1 The dumbbell test specimens are cyclically strained at a fixed frequency and a series of fixed maximum extension ratios such that little or no temperature rise is induced. This cyclical straining action is called flexing. As a result of the flexing, cracks usually initiated by a naturally occurring flaw, grow and ultimately cause failure which is defined as complete rupture of the test specimen. The number of cycles to failure (fatigue life) is recorded.

4.2 Fatigue, as used in this test method, implies a rupture failure mechanism that results from the growth of flaws in the specimen. Fatigue does not refer to the drastic alteration of the physical-chemical rubber structure characteristic of high frequency flexing tests that give rise to a substantial temperature increase.

4.3 Fatigue life may be determined at each of a number of different extension ratios and the log (fatigue life) plotted as a function of either extension-ratio or log (strain energy). A single extension-ratio or log (strain energy) may be used for limited comparisons of rubber vulcanizates having similar stress-strain properties and the same polymer system (see Annex A1).

## 5. Significance and Use

5.1 This test method covers one procedure for determining fatigue life at various extension-ratios. The strain cycle is characteristic of the type of test apparatus specified. Experience in fatigue testing shows that fatigue life may have a wide, non-normal distribution and therefore, a large standard deviation that is compound dependent. Natural rubber, for example,

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[1] This test method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.15 on Degradation Tests.

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

[3] Available from British Standards Institute, 2 Park St., London W1A 2BF, United Kingdom.

has shown a narrower distribution than many synthetic rubbers. A large number of specimens may, therefore, be required to yield the desired precision. Comparison of different rubber compounds should be made with due consideration to the standard deviation for each (see 7.1).

5.2 Fatigue data, as generated in this test method, give primarily an estimate of the crack initation behavior of a rubber vulcanizate and only a very approximate measure of the crack propagation rate. The information obtained may be useful in predicting the flex-life performance of a compound in active service; however, the user should be aware that in actual use, products are subjected to many other fatigue factors not measured in this test method.

## 6. Apparatus

6.1 *Fatigue Tester*, [4] consisting of framework capable of containing two or more sets of multi-place specimen racks or crossheads that hold specimens in a vertical position, side-by-side, in suitable grips. A crosshead or rack set is comprised of one stationary bar to which grips are attached and one moveable bar that is cycled by a cam at 1.7 ± 0.17 Hz (100 + 0 − 10 cpm). Specimens are mounted in the grips, one specimen in each set of upper and lower grips.

6.1.1 The fatigue tester shall be capable of nominal specimen extension ratios of 1.6 to 2.4. The extension ratio is controlled by the use of a cam attached to a rotating drive shaft. The eccentricity of the cam imports the characteristic strain cycle to the specimen. Each cycle consists of:

6.1.1.1 Increasing strain for one quarter of the cycle time,

6.1.1.2 Decreasing strain for one quarter of the cycle time, and

6.1.1.3 Zero imposed strain for one half of the cycle time.

6.1.2 The specimen grips shall not cause premature failure outside the restricted portion of the test specimen. This is achieved by using a dumbbell test specimen with a thick beaded edge molded at each end of the specimen. This specimen is placed into grips that loosely hold it at the bead but impose no compressive stress on it (see Fig. 1).

6.2 *Mold*, sheets, to be used to cut specimens, can be vulcanized in a single cavity compression mold of two piece construction with a hard chrome finish (see Fig. 1). The cavity is 254 mm (10 in.) × 78.54 mm, and has a 6.35-mm (0.25 in.) diameter bead along each lengthwise edge. Cutting guides should be included at 14.3 mm (0.56 in.) intervals along the beaded edges.

6.3 *Press*, as described in accordance with Recommended Practice D 3182.

6.4 *Specimen Cutter*, the cutting die shall conform to Fig. 2. The inside faces of the reduced section shall be polished and perpendicular to the plane formed by the cutting edges for a depth of at least 5 mm (0.2 in.).

6.5 *Vernier Calipers*, Calipers capable of making measurements in accordance with Practice D 3767, with a minimum range of 15 mm (0.6 in.), and precision of 0.05 mm (0.002 in.).

6.6 *Stress-Strain Measuring Apparatus*— Either of two types may be used:

6.6.1 A machine in which the actual extension is measured at a given dead-weight force. A stand enables masses to be suspended from the specimen. A set of masses that includes at least one 50, one 100, two 200, two 500, and one 1000-g mass shall be available.

6.6.2 Alternatively, a tensile testing machine may be used that is capable of extending the specimen at a rate of 0.85 mm/s 50.0 mm/min (2.0 in./min). It should automatically measure elongation to an accuracy of ±5 % of the specimen's original length.

6.7 *Micrometer*—The micrometer or thickness gage shall conform to the specifications in Practice D 3767.

6.8 *Bench Marker*, with two parallel straight marking surfaces ground smooth in the same plane. The surfaces shall be between 0.05 and 0.08-mm (0.002 and 0.003-in.) wide and 23-mm (0.9-in.) long. The angle between the marking surfaces and sides shall be at least 75°. The distance between the marking centers shall be 25± 0.50 mm) (0.984 ± 0.020 in.).

## 7. Sampling

7.1 Sampling shall be done in a way that justifies the conclusions drawn from any particular test program in declaring one compound to be superior to another. In fatigue-life measurement, a sampling variance that includes mix and curing variance components shall be used.

## 8. Specimen Preparation

8.1 Compounds shall be prepared in accordance with Recommended Practice D 3182 and vulcanized in the specified mold with the milling-grain direction parallel to the beaded edge.

8.2 The molded sheet shall be conditioned in an unstrained state for at least 24 h at test temperature before testing.

8.3 Specimens shall be cut with the die cutter at right angles to the beaded edge. The die cutter shall be sharp and free from nicks and oil prior to cutting. Support the sheets on a suitable cutting surface (cardboard, linoleum, etc.) covered with a thin plastic film to prevent inclusions. Cut the sheets with a single, smooth stroke.

8.4 Discard specimens having obvious flaws. Before testing, physically randomize the specimens from all sheets of the same compound.

## 9. Conditioning

9.1 *Test Temperature*—It is suggested that the test temperature be 23 ± 2°C.

NOTE 1—It is recommended that the laboratory room housing the fatigue tester be free of any ozone-generating equipment.

## 10. Procedure

10.1 *Fatigue Tester*:

10.1.1 Install the proper cam that will give the desired extension ratio.

NOTE 2—If previous knowledge about fatigue life of a particular compound is not available, an initial extension-ratio of 2.0 is often used.

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[4] Commercial fatigue testers, available from Monsanto, 2689 Windgate Ave., Akron, OH 44314 and Swindon, United Kingdom.

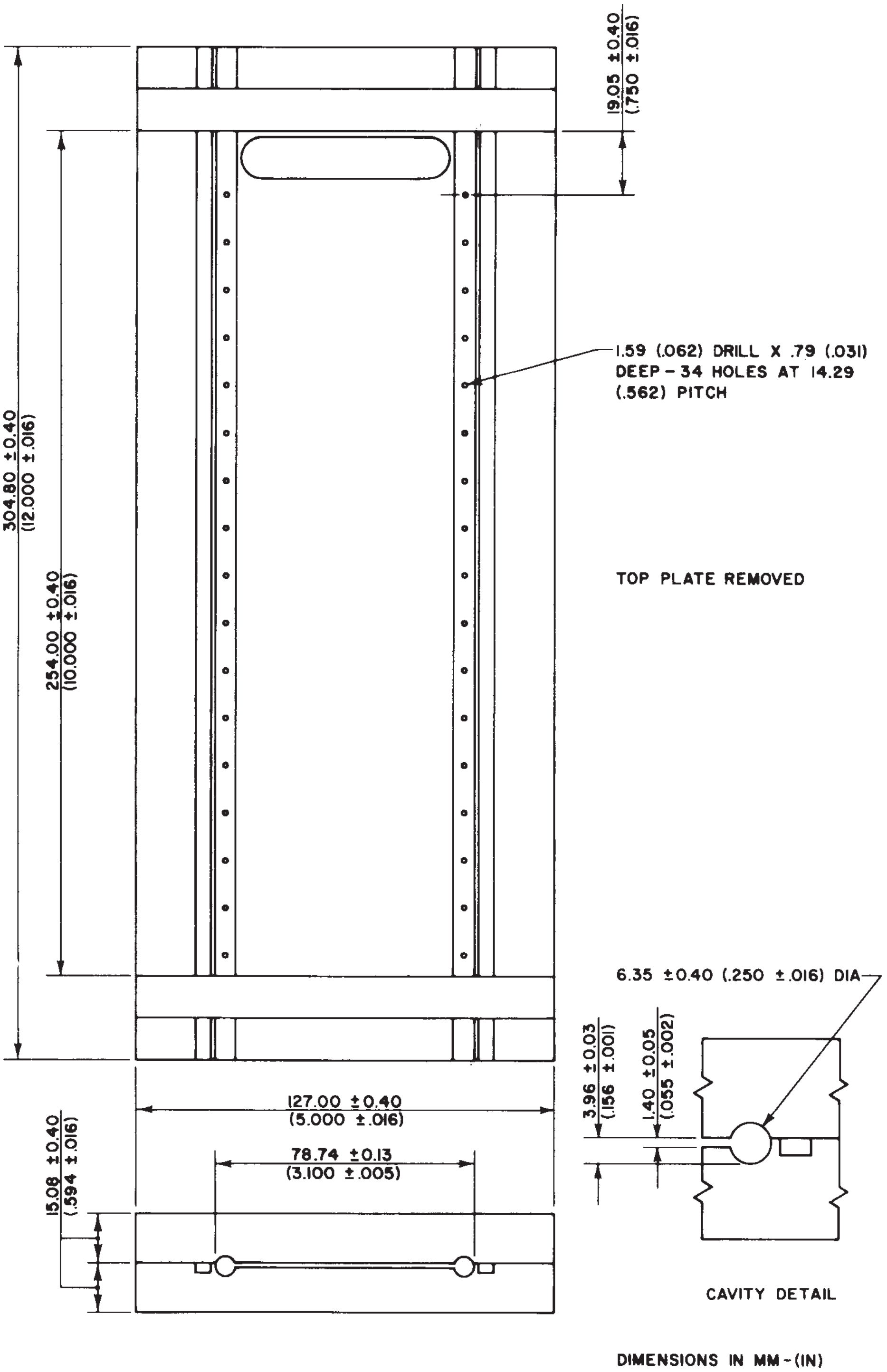


**FIG. 1 Single-Cavity Compression Mold**

10.1.2 Adjust the distance between the flat, inner surface of the grips to 6 cm. A gage block 6-cm long is convenient for this adjustment.

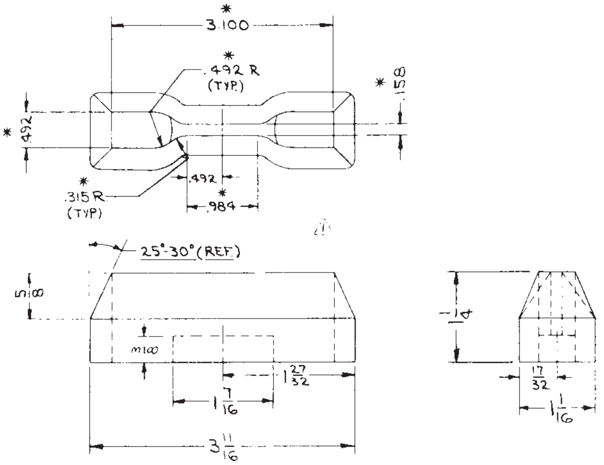
10.1.3 If permanent set and extension-ratio measurements are desired, place two bench marks approximately 25 mm apart on the reduced section of two specimens of each sample. Place the marks perpendicular to the longitudinal axis and equidistant from the center of the specimen. This distance is designated $L_i$.

10.1.4 Manually set the cam position to the minimum crosshead separation. Mount the specimens randomly in the grips.

10.1.5 Start the fatigue tester.

10.1.6 At 1000 cycles stop the tester and manually adjust the drive mechanism to produce minimum crosshead separation.

NOTE 3—If the tester is using two sets of crossheads, follow the



NOTE 1—* Indicates Cutter Edge
Cutter to be used for cutting samples from molded rubber sheets.
Ref: Dumbbell outline to conform with B.S. 903, Part , Type E.
Matl: Annealed tool steel harden and grind.

**FIG. 2 Specimen Cutter**

instructions starting with 10.1.6 for one bank of specimens at a time.

10.1.7 For each specimen, one at a time, use the grip adjustment to increase the distance between the grips until the specimen is under a slight tension, then relieve the tension until a slight bow is just perceptable in the specimen. The specimen has now been adjusted for permanent set.

10.1.8 For specimens requiring permanent set measurements, measure the distance between the bench marks and record as $L_o$ to the closest ±1 mm. Calculate the permanent set using the *Li* measurement from 10.1.3.

$$\text{Permanent set, \%} = \left(\frac{L_o - L_i}{L_i}\right)(100) \qquad (2)$$

NOTE 4—Repeatability may be improved if the specimen is not adjusted for permanent set.

10.1.9 For those specimens requiring a strain ratio measurement; after the specimens have rested at zero strain for at least 3 min, manually adjust the drive mechanism until the specimens are at maximum extension. Measure the distance between the bench marks and record as *L* to the closest ± 1.0 mm. Compute the extension ratio,λ .

$$\lambda = \frac{L}{L_o} \qquad (3)$$

10.1.10 Restart the tester, readjust the permanent set at 10 000 additional cycles, and then each 24 h, thereafter.

10.1.11 At the end of the test, record the total cycles for each specimen, note and record the number of failures. Terminate the test when all specimens have failed or an arbitrary number of cycles have been obtained, or an arbitrary number of specimens remain intact.

10.1.12 Repeat steps 10.1.1 through 10.1.11 for each desired extension ratio.

10.2 *Strain Energy Determination*:

10.2.1 If strain energy comparisons are necessary, determine stress-strain properties using either the method in 10.2.2, the Manual Machine Method, or 10.2.3, the Tensile Testing Machine Method. This is optional for single extension ratio tests.

10.2.2 *Manual Machine Method*—Measure the specimen width and thickness with the micrometer. Stretch the specimen 30 times to the maximum extension used in testing. Place bench marks just short of the end of the straight, reduced section of the specimen at maximum strain and then release to zero strain. Measure the unextended length with vernier calipers. Place sufficient force on the specimen in obtain an extension ratio approximately 1.2 within 1 min. Increase the force at 1 min intervals to achieve a maximum extension ratio of 2.5. Measure the extended length 0.5 min after each force is applied.

10.2.3 *Tensile Machine Method*—Measure the specimen width and thickness with the micrometer. Stretch the specimen 30 times to the maximum extension used in testing. Elongate the specimen at 50 mm/min (2 in./min), automatically recording the force at every 10 % elongation increment until an extension ratio of 2.5 (150 % elongation) is obtained. A constant rate of extension tensile machine may be used.

## 11. Calculations

11.1 Determine the geometric mean fatigue life for each rubber vulcanizate at each extension. When the logarithmic transformation is applied, the mean fatigue life is determined as follows:

$$G = n\sqrt{N_1XN_2XN_n} \qquad (4)$$

where
$G$ = geometic mean,
$N_i$ = fatigue life of the $_i$*th* specimen, and
$n$ = total number of specimens.

11.1.1 On a log basis:

$$\log G = \frac{\Sigma(\log N_i)}{N} \qquad (5)$$

11.2 The use of log (fatigue life) has been found to approximately normalize the distribution and reduce the standard deviation of a single compound at multiple extension ratios.

11.3 The variability of fatigue testing leads to difficulties in determining “average” fatigue lives. In addition to the use of the geometric mean, the median value of the tests has also been used. Annex A2 is taken from the British Standard BS5324, and may provide useful information for the analysis of fatigue data.

11.4 If required, determine the strain energy at each extension for each rubber compound (Annex A1).

11.5 Plot log *G* as a function of the extension ratio and log *G* as a function of log (strain energy) for multiple extension radio tests. A straight line may be fitted through these points either visually or by the least squares method.

## 12. Report

12.1 The report shall include the following information:

12.1.1 Identification of test compounds.

12.1.2 Test temperature.

12.1.3 Number of specimens tested at each extension ratio.

12.2 The fatigue life for each compound at each extension ratio and the method of calculating the fatigue life.

12.3 The average extension ratio obtained for each compound at each extension ratio.

12.4 A graph of the log *G* as a function of the extension ratio for multiple extension tests.

12.5 A graph of the log *G* as a function of the log strain-energy for multiple extension tests.

## 13. Precision and Bias

13.1 *Precision*—An interlaboratory test has been conducted and test results generated. A precision statement is being developed in accordance with Practice D 4483.

# ANNEXES

### (Mandatory Information)

## A1. CALCULATION OF STRAIN ENERGY

A1.1 Strain energy is the area under a force-deflection curve up to a fixed deflection and is expressed in joules. The area under a stress-strain curve up to fixed extension ratio becomes strain energy normalized for a specific volume based on specimen cross sectional area and gage length. This is expressed as J/m$^3$ or kJ/m$^3$.

A1.2 This normalized strain energy may be approximated by applying Simpson's Rule to the stress-strain data. A plot of stress versus strain should be drawn and a smooth curve approximated through these points. Simpson's rule:

$$\text{Strain Energy} = \frac{1}{3}\Delta \times f(xi) + 4f(x_i + \Delta x) + f(x_i + 2\Delta x) \tag{A1.1}$$

where:

$x_i$ = a given value of strain,

$f(x_i)$ = the corresponding stress value when strain = xi, and

$\Delta x$ = a small constant increment of strain.

A1.3 Successive values for strain should be chosen from the stress-strain curve so that 1.0 is the first value and is incremented by 0.1 until a strain of 2.4 is obtained. Therefore:

$$\text{Strain Energy } (1.0 \rightarrow 1.2) = \frac{1}{3}(0.1)\, f(1.0) + 4\, f(1.1) + f(1.2) \tag{A1.2}$$

$$\text{Strain Energy } (1.2 \rightarrow 1.4) = \frac{1}{3}(0.1)\, f(1.2) + 4\, f(1.3) + f(1.4) \tag{A1.3}$$

and so on until a strain energy from 2.2 to 2.4 is obtained. The accumulated strain energy is the sum of the strain-energy intervals. For example:

$$\text{Accumulated strain energy } (1.0 \rightarrow 1.4)$$

$$= \text{Strain energy } (1.0 \rightarrow 1.2) + \text{strain energy } (1.2 \rightarrow 1.4) \tag{A1.4}$$

A1.4 From a plot of strain versus strain energy, the strain energy at the actual extension ratio may be obtained. An example of this is shown in Table A1.1. The stress-strain curve representing the data in Table A1.1 is illustrated in Fig. A1.1 and Fig. A1.2.

**TABLE A1.1 Stress-Strain Deformation Measurements**

NOTE 1—Stress- extension ratio indices were obtained from Fig. A1.1.

| Extension Ratio | Stress, kPa |
|---|---|
| 1.00 | 0.0 |
| 1.03 | 170 |
| 1.16 | 507 |
| 1.43 | 845 |
| 1.74 | 1180 |
| 1.98 | 1520 |
| 2.15 | 1860 |
| 2.26 | 2200 |
| 2.29 | 2540 |
| 2.35 | 2870 |
| 2.41 | 3210 |

A1.5 Data to apply Simpson's rule are found in Table A1.2.

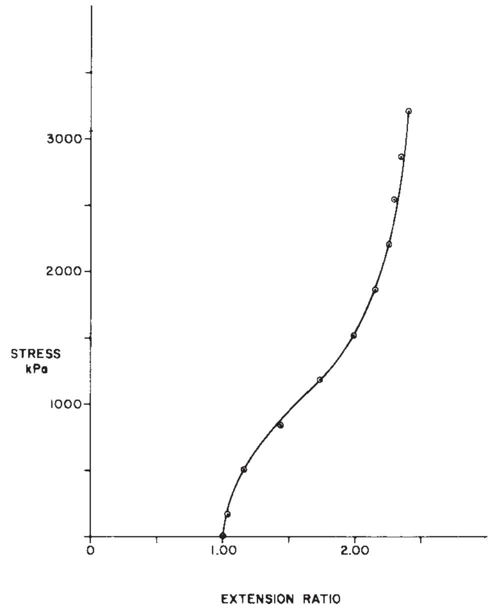


**FIG. A1.1 Stress-Strain Curve**

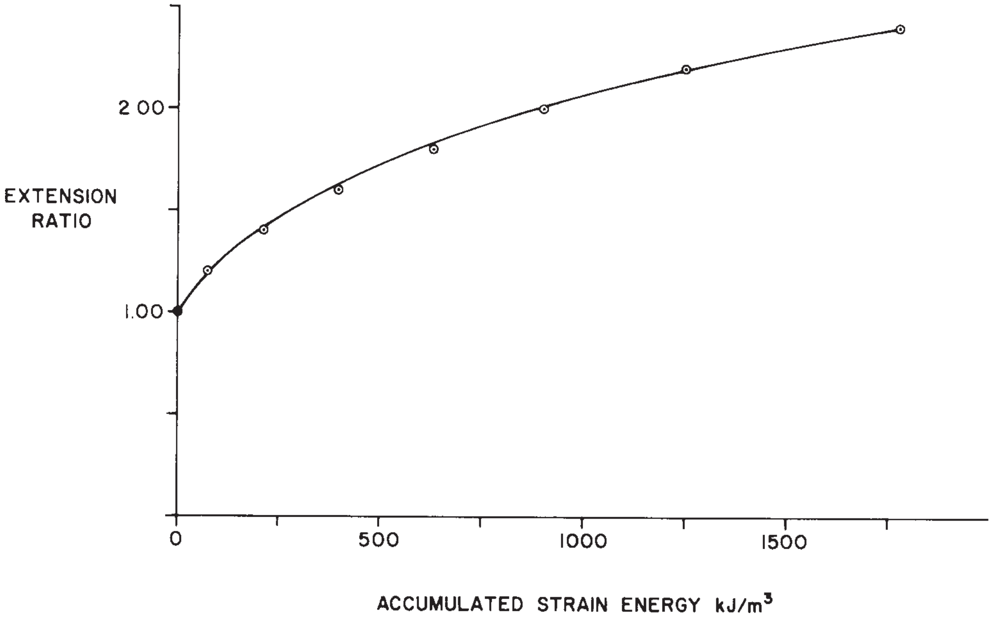


FIG. A1.2 Extension Ratio-Strain Energy Curve

TABLE A1.2 Data for Applying Simpson's Rule

| Extension Ratio, *xi* | Stress, kPa, *f(x)* | Strain Energy,[A] kJ/m3 | Accumulated Strain Energy, kJ/m$^3$ |
|---|---|---|---|
| 1.0 | ... | ... | ... |
| 1.1 | 382 | | |
| 1.2 | 579 | 70 | 70 |
| 1.3 | 716 | | |
| 1.4 | 814 | 142 | 212 |
| 1.5 | 912 | | |
| 1.6 | 1020 | 182 | 395 |
| 1.7 | 1130 | | |
| 1.8 | 1260 | 227 | 632 |
| 1.9 | 1400 | | |
| 2.0 | 1560 | 280 | 901 |
| 2.1 | 1750 | | |
| 2.2 | 2020 | 352 | 1250 |
| 2.3 | 2480 | | |
| 2.4 | 3160 | 504 | 1780 |

[A] Strain energy (1.0 → 1.2):
= 1/3(.1)[ *f* (1.0) + 4 *f* (1.1) + (1.2)]
= 1/3(.1)[0 + 4(382) + 579]
= 70
Strain energy (1.2 → 1.4):
= 1/3(.1)[579 + 4(716) + 814]
= 142
Accumulated strain energy (1.0→ 1.4)
= 70 + 142
= 212
The series of accumulated strain energy values may be plotted as a function of extension ratio, as shown in Fig. A1.2.

## A2. ANALYSIS OF FATIGUE DATA

A2.1 This brief guide to data interpretation is taken from paragraph 15.2.3 of BS 5324 (British Standard).

A2.2 *Fatigue testing*—Fatigue testing involves measuring the number of cycles to the failure (the fatigue life) of test pieces subjected to repeated deformations. A brief description of statistical aspects of fatigue behaviour is given below together with a simple method for the assessment of results.

A2.3 The inherent variability in fatigue life is very much greater than in other strength property, such as tensile strength; this reflects the greater sensitivity of fatigue life to factors that influence failure, such as flaw size. The extent of the variation depends on vulcanizate compositions, particularly the type of rubber used. For example, the overall variation, maximum or minimum life, or both, for vulcanizates of natural rubber (NR) or isoprene rubber (IR), is typically two-fold or less, whereas, for styrene-butadiene rubber (SBR) or butadiene rubber (BR) it can be an order of magnitude more.

A2.4 The nature of fatigue life distribution is also influenced by vulcanizate details and no single distribution is applicable to all rubbers. Therefore, for NR or IR vulcanizates, the distribution of fatigue lives often approximates to a Normal (Gaussian) distribution. On the other hand, for SBR the distribution of fatigue lives tends to be markedly skew, but that of their logarithms may be essentially Normal. In view of these differences in behaviour and the complexities that they present, particularly in relation to the treatment of blends of different rubbers (which are very widely used in practice), a generally applicable method of analysis is recommended. This analysis may be along the simple lines given in A2.4.1 through A2.4.4. For each set of tests, the following should be reported:

A2.4.1 Number of test pieces used,
A2.4.2 Individual fatigue lives in ascending order of magnitude,
A2.4.3 Median fatigue life, and
A2.4.4 Measure of dispersion.

A2.5 It is important that some measure of dispersion is quoted. Apart from standard techniques previously described in this guide, use of the ratio of highest to lowest life has provided a simple measure that has been found useful in the particular area of fatigue testing. In principle this ratio involves some disadvantages, but for the numbers of test pieces that are normally involved it has been found to correlate closely with the coefficient of variation and is much easier to handle. Because of the complexity of fatigue testing and the differences in the behaviour of the various rubbers, care should be taken in applying statistical tests previously described in this guide. Appropriate statistical tests are available (for example, distribution free tests), but are beyond the scope of this guide.

A2.6 Low results should be disregarded if (and only if) there is positive nonstatistical evidence that they are unrepresentative, for example, the presence of an abnormally large flaw in the fracture surface that is clearly attributable to a fault in test-piece preparation.

A2.7 For NR and IR, six test pieces should give a representative measure of the median but for SBR and rubbers that behave similarly 12 test pieces are likely to be required, particularly when only one strain cycle is used. Apart from the general reasons given earlier in this guide, the median provides a more satisfactory measure of central tendency than the arithmetic mean for rubbers such as SBR, the (fatigue) lives of which follow a skew distribution. Attention is drawn to the lowest fatigue life since this is often primarily of concern from a service viewpoint.