British Standard

Methods of testing vulcanized rubber

Part A49. Determination of temperature rise and resistance to fatigue in flexometer testing (basic principles)

0 Introduction

Under cyclic deformation, all rubbers absorb a part of the deformation energy and convert this into heat as a result of their visco-elastic behaviour. The heat generated leads to a temperature rise which can be very considerable in the interior of relatively thick components because of the low thermal conductivity of rubbers. In cases where the cyclic deformation or the temperature rise reaches high values, it is possible for damage to the rubber to occur through fatigue-initiated breakdown. This begins in the interior of the rubber, spreads outwards and can finally lead to the complete breakdown of the component.

The tests described in this International Standard yield either temperature rise data or the fatigue life of the rubber under given test conditions. Measurement of fatigue life over a range of conditions can be used to determine the limiting fatigue deformability or limiting fatigue stress of the rubber. The instruments used, commonly called flexometers, may subject test pieces to cycles of either constant stress amplitude or constant strain amplitude.

A distinction should be made between flexometer tests and fatigue tests conducted on thin test pieces undergoing tensile deformation. In the latter tests, the temperature rise is generally negligible owing to the rapid dissipation of heat generated, and failure results from the initiation and growth of cracks which ultimately sever the test piece. ISO 132 and ISO 133 describe tests for the determination of flex cracking and cut growth, respectively, using the De Mattia-type machine. The determination of resistance to tension fatigue will be described in ISO 6943.

1 Scope and field of application

This part of ISO 4666 lays down general principles for, and defines the terms used in, flexometer testing. It gives directions for carrying out measurements which make possible predictions regarding the durability of rubbers in finished articles (tyres, bearings, supports, V-belts, cable-pulley insert rings and similar products subject to dynamic flexing in service). However, owing to the wide variations in service conditions, no simple correlation between the accelerated tests described in this International Standard and service performance can be assumed.

2 References

ISO 132, *Vulcanized rubbers — Determination of resistance to flex cracking (De Mattia type machine).*

ISO 133, *Rubber vulcanized — Determination of crack growth (De Mattia).*

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1826, *Rubber, vulcanized — Time-interval between vulcanization and testing — Specification.*

ISO 2856, *Elastomers — General requirements for dynamic testing.*

ISO 3383, *Rubber — General directions for achieving elevated or sub-normal temperatures for tests.*

ISO 4666/2, *Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing — Part 2 : Rotary flexometer.*

ISO 4666/3, *Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing — Part 3 : Compression flexometer.*[1)]

3 Definitions

For the purpose of this International Standard, the following definitions apply. For associated terms, refer to ISO 2856 which gives the general requirements for dynamic testing.

3.1 loading : Subjection of the test piece to a predetermined stress or strain, either static or cyclic.

3.2 pre-stress, ϱ_p : The constant static stress to which the test piece is subjected during the test.

NOTE — This may be used to simulate product requirements or simply to hold the test piece in the apparatus.

1) At present at the stage of draft.

3.3 pre-strain, ϵ_p : The constant static strain to which the test piece is subjected during the test.

NOTE — This may be used to simulate product requirements or simply to hold the test piece in the apparatus.

3.4 cyclic stress amplitude, σ_a or τ_a : The ratio of the force amplitude (cyclic force) superimposed upon the pre-strain or pre-stress to the appropriate dimension of the unstressed test piece.

3.5 cyclic strain amplitude, ϵ_a or γ_a : The ratio of the deformation amplitude (cyclic deformation) superimposed upon the pre-strain or pre-stress to the appropriate dimension of the unstrained test piece.

NOTES

1 For certain flexometers, the cyclic strain amplitude has to be smaller than that of the pre-strain.

2) In a compression flexometer, the pre-stress, σ_p acts in the same direction as the cyclic strain amplitude, ϵ_a. In a rotary flexometer, a cyclic shear strain γ_a or cyclic shear stress τ_a acts at right angles to an axial compression pre-strain ϵ_p or compression pre-stress σ_p.

3.6 heat generation : The total heat generated in the test piece by energy absorption during the test.

NOTE — This term should be distinguished from the deprecated, but sometimes used, expression "heat build up", which is normally associated with the temperature rise in the test piece and which therefore may not take account of heat losses.

3.7 temperature rise : The increase in temperature of the test piece.

NOTE — The temperature rise is taken as the difference between the temperature measured at a given point in the test piece at a given time during the test and either the temperature at the beginning of the test or the ambient temperature.

3.8 fatigue breakdown : The change in chemical and/or physical structure or composition of the test piece under the simultaneous action of stress and temperature.

3.9 fatigue life, N : The number of cycles required to produce failure or breakdown under a given static and cyclic loading.

3.10 fatigue deformability : The cyclic strain amplitude corresponding to a given fatigue life.

3.11 fatigue stress : The cyclic stress amplitude corresponding to a given fatigue life.

3.12 limiting fatigue deformability, ϵ_∞ or γ_∞ : The cyclic strain amplitude at which the fatigue life curve becomes essentially parallel to the log N axis (see the figure).

3.13 limiting fatigue stress, σ_∞ or τ_∞ : The cyclic stress amplitude at which the fatigue life curve becomes essentially parallel to the log N axis (see the figure).

4 Test conditions

The relative ratings of rubbers having different moduli depend upon the type of loading used to evaluate them :

a) σ_p and σ_a or τ_a constant;

b) σ_p and ϵ_a or γ_a constant;

c) ϵ_p and σ_a or τ_a constant; or,

d) ϵ_p and ϵ_a or γ_a constant.

Both the type and magnitude of loading should be governed by the intended use of the rubber. In tests for heat generation, the magnitude should be high enough to generate a temperature rise that is sufficiently discriminating, but not high enough to cause breakdown.

In tests for fatigue life, the loading should be chosen that will yield results capable of discrimination.

NOTE — It is also possible to conduct tests under constant strain energy conditions.

5 Test pieces

5.1 Form and preparation

Test pieces for flexometer testing shall be cylindrically shaped. Dimensions will differ according to the test method used.

Test pieces may be prepared by vulcanization in moulds, or from slabs or finished parts by cutting, boring and grinding. If test pieces are cut from a finished part, this shall be mentioned in the test report.

5.2 Time-interval between vulcanization and testing (see ISO 1826)

For all test purposes, the minimum time between vulcanization and testing shall be 16 h.

For non-product tests, the maximum time between vulcanization and testing shall be 4 weeks and for evaluations intended to be comparable, the tests, as far as possible, should be carried out after the same time interval.

For product tests, whenever possible, the time between vulcanization and testing should not exceed 3 months. In other cases, tests shall be made within 2 months of the date of receipt of the product by the customer.

5.3 Conditioning

Before testing, test pieces shall be conditioned for at least 3 h at one of the standard laboratory temperatures specified in ISO 471.

5.4 Number

Two test pieces of each rubber shall be used for measurement of either the temperature rise under a specified loading or the fatigue life under a specified loading. More test pieces will be needed if confidence limits are to be established. For plotting fatigue life curves, at least five and preferably ten test pieces should be provided.

6 Test machines

Only general requirements for test machines (flexometers) are considered here. Typical machines are described in ISO 4666/2 and ISO 4666/3, but other machines may be used provided that they fulfil the basic requirements of this International Standard and provided that all comparative tests are carried out on the same machine.

Construction shall be sturdy and precise. The imposed test conditions shall be constant for any single test series, but adjustments shall be possible from one series to the next.

Readings or recordings, whether by mechanical, optical or electrical means, shall have adequate sensitivity. For tests conducted at elevated temperature, an adequately thermostatted housing operating in accordance with ISO 3383 shall be provided.

7 Procedure

The exact procedures depend on the particular test method used; the rotary flexometer is described in ISO 4666/2 and the compression flexometer is described in ISO 4666/3. The following basic principles are applicable to all test methods.

7.1 Measurement of temperature rise

For measurement of the temperature rise, heat losses should be kept low, for example by insulation of the surfaces in contact with the test pieces.

The initial temperature of all test pieces to be compared shall be either an elevated temperature held constant to within 1 °C or a standard laboratory temperature. The temperature rise of a specific point in the test piece shall be determined after a running time that is long enough to produce thermal equilibrium.

The temperature in the interior of the test piece may be measured by the insertion of a thin needle probe temperature sensor. The test piece shall be free from stress while the needle is inserted, so that the temperature sensor is closely surrounded by the rubber. In order to read the temperature continuously during the test procedure, it shall be measured at the test piece surface (or on a surface in contact with the test piece), and the temperature measured by this means may be plotted against the test time.

These two methods of temperature measurement differ basically in that the thermal conductivity and surface emissivity of the rubber enters into the measured values in a different manner. The permissible error in the temperature measurement is ± 1 °C.

7.2 Determination of fatigue life

Test pieces shall be subjected to the type and magnitude of loading specified (see clause 4) and the number of cycles to breakdown shall be recorded in each case. A suitable means of assessing the onset of breakdown shall be used and shall be the same for all comparative tests. In all cases, after the termination of the test, a check shall be made by a visual examination of the sectioned test pieces to ensure that the level of damage is comparable.

Test conditions shall not be altered during the course of the test and shall be the same when comparing several rubbers. If fatigue life is to be determined under one set of test conditions only, the imposed cyclic strain amplitude or stress amplitude shall normally be greater than the limiting fatigue deformability or limiting fatigue stress of the rubber.

7.3 Determination of limiting fatigue deformability and limiting fatigue stress

For the estimation of the limiting fatigue deformability or limiting fatigue stress of the rubber, the fatigue life shall be determined over a range of cyclic strain amplitudes or cyclic stress amplitudes, including that region in which it becomes very long and virtually indefinite. To accomplish this, several test pieces shall be subjected, one after the other, to suitably spaced loadings and the corresponding numbers of cycles to breakdown shall be measured.

The fatigue lives so obtained shall be plotted as a function of the cyclic strain amplitude or cyclic stress amplitude (see the figure). The limiting fatigue deformability and the limiting fatigue stress shall be taken as, respectively, the cyclic strain amplitude and the cyclic stress amplitude at which the fatigue life curve becomes essentially parallel to the fatigue life axis.

If required, a plot of fatigue life against cyclic strain amplitude or cyclic stress amplitude may be used to calculate the fatigue deformability or fatigue stress for a given fatigue life. In this case, it is not necessary to undertake tests at cyclic strain amplitudes or cyclic stress amplitudes where the fatigue life approaches infinity.

7.4 Measurement of creep

When it is appropriate and when it is required, creep shall be measured or registered by a recorder as the change in height of the test piece or the movement of a contact platen during the test, recorded at suitable intervals of time. The creep is calculated relative to the initial height of the undeformed or unstressed test piece.

7.5 Set calculations

When it is appropriate and when it is required, set shall be calculated from the difference between the initial height of the test piece in the undeformed or unstressed condition and the height of the test piece at the end of the test after a specified recovery period; it shall be reported as a percentage of the initial height. The recovery period chosen shall be sufficiently long for the recovery to reach substantially its asymptotic value.

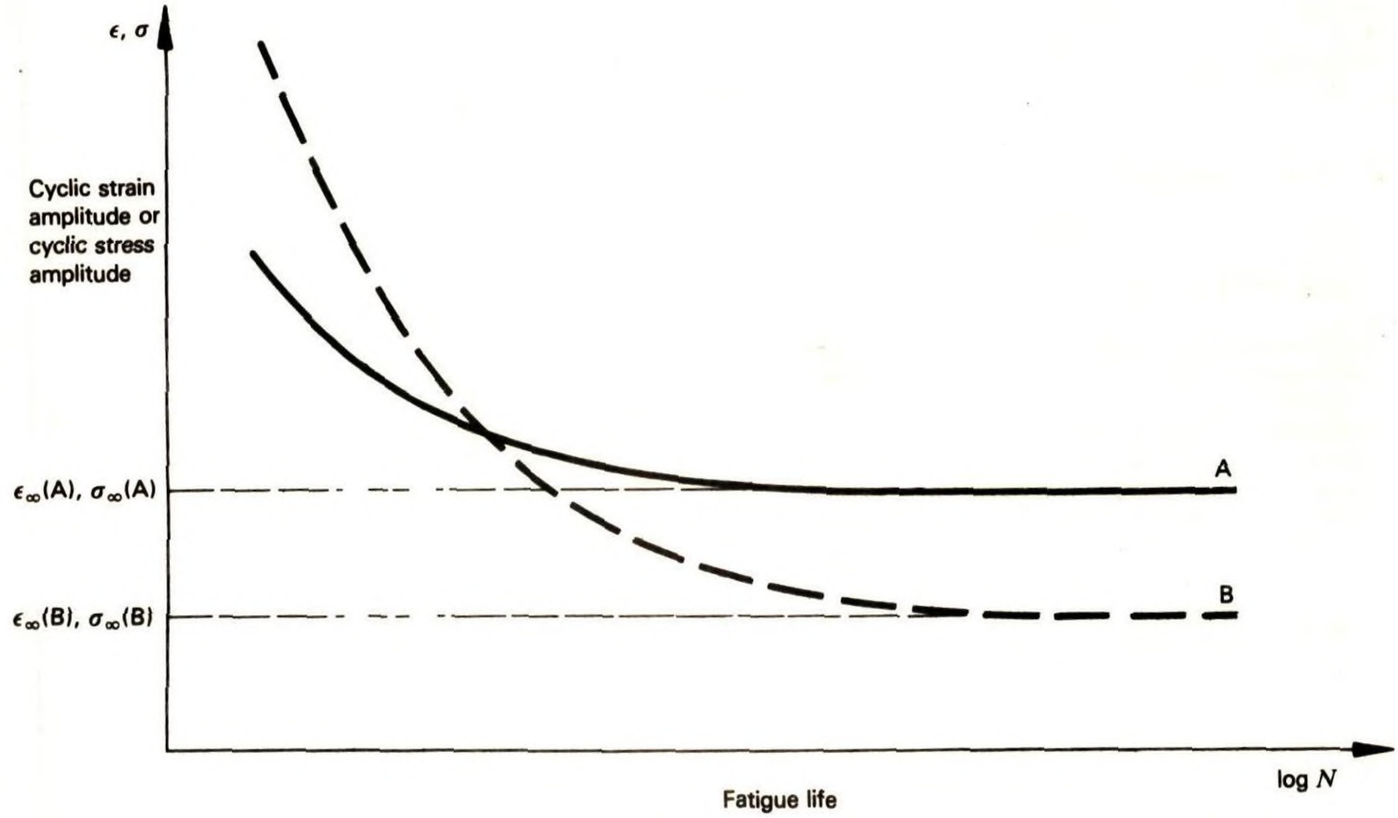

Figure — Fatigue life curves for two rubbers, A and B

Annex

Explanatory notes

(This annex does not form part of the standard.)

A.0 Introduction

When a rubber component is subjected to repeated deformation, heat is generated as a result of energy losses or hysteresis. In very thin components, this heat can be conducted away rapidly without undue effect on the rubber despite the poor thermal conductivity of rubber, but in relatively thick components the heat cannot be easily dissipated and so the rubber heats up. In the absence of degradation, the temperature will eventually reach an equilibrium level at which as much heat is generated as is dissipated by conduction, convection and radiation. If, however, the temperature increase is sufficiently high to cause the rubber to degrade, the component may become progressively hotter and eventually break down as a result of poor high temperature strength and fatigue resistance. A practical example is a "blow out" in truck tyres. Fatigue failure will also result if the stresses imposed during deformation exceed the mechanical strength of the rubber. Other undesirable features of repeated cycling are creep and set.

A.1 Use of flexometer tests

Flexometer tests are designed to determine the resistance shown by a rubber to temperature rise or fatigue breakdown under dynamic test conditions. As such, they differ from, yet complement, the tests described in ISO 2856 where fatigue and temperature rise effects are not taken into account.

The properties covered in this International Standard and ISO 2856 do, however, have one important feature in common, namely that, because of the complex visco-elastic behaviour of rubbers, dynamic properties, such as resistance to temperature rise and fatigue breakdown, are highly sensitive to test conditions such as temperature, frequency and the amplitude of applied static or cyclic stress or strain. If different rubbers are being compared, their ranking order may well vary according to the test conditions used. For this reason, it is always advisable to carry out tests over a range of conditions and to try to identify the condition relevant for a particular use or service application. A primary consideration is the decision to conduct the test at a constant cyclic stress amplitude or at a constant cyclic strain amplitude. This is especially important when comparing the behaviour of rubbers varying in hardness or modulus. An increase in hardness, for example, brought about by an increase in the level of carbon black filler, will be accompanied by an increase in hysteresis, which will result in a higher temperature rise in tests carried out at constant strain amplitude. If, however, a constant stress amplitude is being used, a hard vulcanizate will deform less than a softer one and so normally generate less heat.

A similar effect may be observed when determining resistance to fatigue breakdown. This property is strongly dependent on the amplitude of the applied cyclic stress or strain. The fatigue life curves illustrated in the figure show the short-comings of single tests. At high severities, rubber A is inferior to rubber B, but at low severities B is inferior to A. It is also found that as the test severity is progressively reduced, the fatigue lives of both rubbers begins to increase rapidly and become infinite as long as the applied stress or strain amplitude does not exceed a characteristic value. The value represents the limiting mechanical condition for fatigue failure and is appropriately expressed as the limiting fatigue deformability, ϵ_∞ or γ_∞ or limiting fatigue stress, σ_∞ or τ_∞ of the rubber, depending on the type of loading used.

A.2 Notes on test procedure

A.2.1 Temperature rise

For the measurement of the temperature rise, it is, in general, recommended that the test time chosen should be sufficiently long for the equilibrium condition between heat generation and heat dissipation to be reached.

Measurement whilst the temperature is still rising can lead to sources of error. Sufficiently low applied stresses and strains should be used to ensure there is no fatigue breakdown within the test piece.

The temperature rise test is most suited to those methods that allow a continuous recording of the temperature in the supporting surface of the test piece. An example is the compression flexometer described in ISO 4666/3, although this only allows testing to be carried out at constant pre-stress and constant cyclic strain amplitude. With other flexometers, such as the rotary type described in ISO 4666/2, the temperature rise is normally measured by the insertion of a needle thermocouple into the centre of the test piece after stopping the machine. In both cases, the temperature recorded will depend on the thermal conductivity of the rubber under test and on the rate of heat loss from the surface. If two rubbers under comparison have the same level of hysteresis, the one having the higher conductivity will have the lower temperature in the centre of the test piece because of the more rapid heat losses.

A.2.2 Fatigue life

Whenever possible, fatigue life should be determined over a range of severities, and it is always advisable that the limiting fatigue deformability or limiting fatigue stress should be estimated. A machine most suited for this purpose is the rotary flexometer described in ISO 4666/2. A compression flexometer is more limited.

An important consideration in fatigue testing is the method used for the assessment of the beginning of breakdown. This has to be indirect since destruction usually starts in the interior of the test piece and thus cannot be seen. The sudden advent of creep or temperature change is an indication of breakdown and, if either occurs, the test should be stopped immediately and the interior of the test piece should be examined for discolouration, porosity or softening.

RUBBER RESEARCH INSTITUTE
OF INDIA LIBRARY

Acc No S 126

Date