INTERNATIONAL STANDARD

ISO 4666-1

Second edition
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Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing —

Part 1: **Basic principles**

Caoutchouc vulcanisé — Détermination de l'élévation de température et de la résistance à la fatigue dans les essais aux flexomètres —

Partie 1: Principes fondamentaux

Reference number
ISO 4666-1:2010(E)

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4666-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 4666-1:1982), which has been technically revised.

ISO 4666 consists of the following parts, under the general title *Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing*:

— *Part 1: Basic principles*

— *Part 2: Rotary flexometer*

— *Part 3: Compression flexometer (constant-strain type)*

— *Part 4: Constant-stress flexometer*

Introduction

All rubbers show viscoelastic behaviour. When subjected to cyclic deformation, they absorb a part of the deformation energy and convert this into heat. The heat generated leads to a temperature rise, which can be considerable in the interior of relatively thick components because of the low thermal conductivity of rubbers. In cases where the cyclic deformation is large or the temperature reaches high values, it is possible for damage to the rubber to occur through fatigue-initiated breakdown. The breakdown begins in the interior of the rubber, spreads outwards, and can finally lead to the complete breakdown of the component.

The tests specified in the various parts of 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, 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 or bending. In the fatigue 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[1] specifies tests for the determination of flex cracking and cut growth using the De Mattia-type machine. The determination of resistance to tension fatigue is specified in ISO 6943[3].

Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing —

Part 1: Basic principles

1 Scope

This part of ISO 4666 establishes general principles for flexometer testing and defines the terms used.

Flexometer testing makes possible predictions regarding the durability of rubbers in finished articles subject to dynamic flexing in service such as tyres, bearings, supports, V-belts, and cable-pulley insert rings. However, owing to the wide variations in service conditions, no simple correlation between the accelerated tests specified in the various parts of this International Standard and service performance can be assumed.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4664-1:—[1)], *Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 1: General guidance*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4664-1 and the following apply.

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

3.2
pre-stress
σ_p
constant static stress to which the test piece is subjected during the test

NOTE 1 Pre-stress is expressed in pascals.

NOTE 2 Pre-stress can be used to simulate product requirements or simply to hold the test piece in the apparatus.

NOTE 3 The term “mean stress” (ISO 4664-1:—) is equivalent or near equivalent to “pre-stress”.

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1) To be published. (Revision of ISO 4664-1:2005)

3.3
pre-strain
ε_p
constant static strain to which the test piece is subjected during the test

NOTE 1 Pre-strain can be used to simulate product requirements or simply to hold the test piece in the apparatus.

NOTE 2 The term "mean strain" (ISO 4664-1:—) is equivalent or near equivalent to "pre-strain".

3.4
cyclic stress amplitude
σ_a
τ_a
ratio of the force amplitude (cyclic force) superimposed upon the pre-strain or pre-stress to the appropriate cross-sectional area of the unstressed test piece

NOTE 1 Cyclic stress amplitude is expressed in pascals.

NOTE 2 The term "maximum stress amplitude" (ISO 4664-1:—) is equivalent or near equivalent to "cyclic stress amplitude".

3.5
cyclic strain amplitude
ε_a
γ_a
deformation amplitude (cyclic deformation) superimposed upon the pre-strain or pre-stress

NOTE 1 For certain flexometers, the cyclic strain is smaller than the pre-strain.

NOTE 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 axial compression pre-stress, σ_p.

NOTE 3 The term "maximum strain amplitude" (ISO 4664-1:—) is equivalent or near equivalent to "cyclic strain amplitude".

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

NOTE "Heat generation" 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.

3.7
temperature rise
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
change in chemical structure, physical structure or composition of the test piece under the simultaneous action of stress and temperature

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

3.10
fatigue deformability
cyclic strain amplitude corresponding to a given fatigue life

3.11
fatigue stress
cyclic stress amplitude corresponding to a given fatigue life

3.12
limiting fatigue deformability
ε_∞
γ_∞
cyclic strain amplitude at which the fatigue life curve becomes essentially parallel to the log N axis

See Figure 1.

3.13
limiting fatigue stress
σ_∞
τ_∞
cyclic stress amplitude at which the fatigue life curve becomes essentially parallel to the log N axis

See Figure 1.

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;

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 sufficient to be discriminating, but not high enough to cause breakdown.

In tests for fatigue life, choose the loading that yields results capable of discriminating between materials.

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

5 Test pieces

5.1 Form and dimensions

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

5.2 Preparation

Test pieces shall be prepared and stored in accordance with ISO 23529. Test pieces may be prepared by vulcanization in moulds, or from slabs or finished parts by cutting, boring, and buffing. If test pieces are cut from a finished part, this shall be mentioned in the test report.

5.3 Time interval between vulcanization and testing

See ISO 23529.

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.

5.4 Conditioning

Before testing, test pieces shall be conditioned for at least 3 h at one of the standard laboratory temperatures specified in ISO 23529.

5.5 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 are needed if confidence limits are to be established. For plotting fatigue life curves, at least five and preferably 10 test pieces should be provided.

6 Apparatus

6.1 General

Only general requirements for test machines (flexometers) are considered here. Typical machines are described in ISO 4666-2, ISO 4666-3 and ISO 4666-4, but other machines may be used provided that they fulfil the basic requirements of this part of ISO 4666 and provided that all comparative tests are carried out on the same type of 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.

6.2 Temperature-controlled cabinet

For tests conducted at elevated temperature, an adequately thermostated enclosure meeting the requirements of ISO 23529 shall be provided.

6.3 Measurement of temperature

For measurement of the temperature rise, heat losses by conduction into the test apparatus should be kept low, e.g. by insulation of the surfaces in contact with the test pieces.

Two methods of measuring the temperature of the test piece are available. The permissible error in the temperature measurement is ±1 °C. The method to be used is specified in the relevant part of this International Standard.

The temperature in the interior of the test piece may be measured. Temperature can be measured by the insertion of a fine needle probe temperature sensor, or the probe may be inserted at particular times during the test when the test piece is free from stress so that the temperature sensor is closely surrounded by the rubber. It may also be embedded continuously in the test piece.

Alternatively, the temperature may be measured at the test piece surface (or on a surface in contact with the test piece).

These two methods of temperature measurement differ fundamentally in that the thermal conductivity and surface emissivity of the rubber enter into the measured values in a different manner.

7 Procedure

7.1 General

The exact procedures depend on the particular test method used: the rotary flexometer is described in ISO 4666-2; the compression flexometer is described in ISO 4666-3; and the constant-stress flexometer is described in ISO 4666-4. The basic principles in 7.2 to 7.7 are applicable to all test methods.

7.2 Temperature of test

The tests shall normally be carried out at one of the temperatures specified in ISO 23529.

7.3 Measurement of temperature rise

The initial temperature of all test pieces to be compared shall be the test 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.

7.4 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.

7.5 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 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, ε_a, or cyclic stress amplitude, σ_a (see Figure 1). 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 can 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.

7.6 Determination of creep

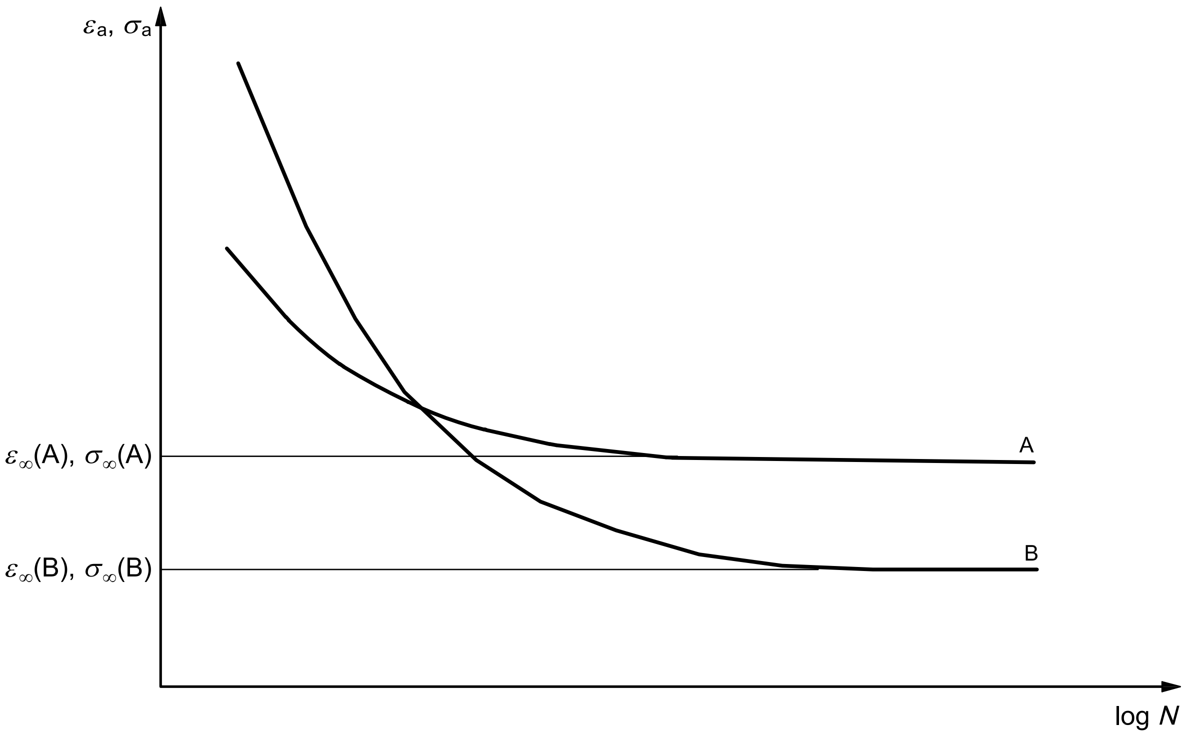
When it is appropriate and when it is required, creep shall be measured or registered by a recorder as the change in height of the test piece or the movement of a contact platen during the test, recorded at suitable intervals of time. The creep is calculated relative to the initial height of the undeformed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

7.7 Determination of set

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.

8 Test report

The results shall be reported as specified in the relevant part of this International Standard.



Key

ε_a	limiting fatigue deformability	σ_a	limiting fatigue stress
ε_∞(A)	limiting fatigue deformability of rubber A	σ_∞(A)	limiting fatigue stress of rubber A
ε_∞(B)	limiting fatigue deformability of rubber B	σ_∞(B)	limiting fatigue stress of rubber B
A	rubber A	B	rubber B
N	fatigue life		

Figure 1 — Fatigue life curves for two rubbers, A and B

Annex A
(informative)

Explanatory notes

A.1 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 eventually reaches 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 “blow-out” in truck tyres. Fatigue failure also results if the stresses imposed during deformation exceed the mechanical strength of the rubber. Other undesirable features of repeated cycling are creep and set.

A.2 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 4664-2[2], where fatigue and temperature rise effects are not taken into account.

The properties covered in the various parts of ISO 4666 and ISO 4664-2[2] do, however, have one important feature in common, namely that, because of the complex viscoelastic behaviour of rubbers, dynamic properties, e.g. 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, e.g. brought about by an increase in the level of carbon black filler, is accompanied by an increase in hysteresis, which results 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 deforms less than a softer one and so normally generates 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 Figure 1 show the shortcomings 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 begin 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.3 Notes on test procedure

A.3.1 Temperature rise

For the measurement of the temperature rise, it is generally recommended that the test time chosen be sufficiently long for the equilibrium condition between heat generation and heat dissipation to be reached, as measurements made while the temperature is still rising can lead to sources of error. Sufficiently low stresses and strains should be applied 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 of the test piece. An example is the compression flexometer described in ISO 4666-3, in which the temperature is continuously measured in the supporting surface of the test piece. Another example is the constant-stress flexometer described in ISO 4666-4, in which the needle probe temperature sensor is kept inserted in the centre of the test piece by using the positioning feedback system.

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 depends 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 has the lower temperature in the centre of the test piece because of the more rapid heat losses.

A.3.2 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 be estimated.

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 examined for discoloration, porosity or softening.

Bibliography

[1] ISO 132, *Rubber, vulcanized or thermoplastic — Determination of flex cracking and crack growth (De Mattia)*

[2] ISO 4664-2, *Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 2: Torsion pendulum methods at low frequencies*

[3] ISO 6943*, Rubber, vulcanized — Determination of tension fatigue*

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