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Methods of testing vulcanized rubber

Part A15. Determination of creep

Méthodes d'essais des élastomères vulcanisés
Partie A15. Détermination du fluage

Prüfverfahren für vulkanisierte Elastomere
Teil A15. Bestimmung des Krieches

British Standards Institution

Contents

Foreword

This British Standard has been prepared under the direction of the Rubber Standards Committee and is a partial revision of the 1958 edition. This revision only describes the determination of creep; the revised method for the determination of stress relaxation will be published shortly as BS 903 : Part A42. The publication of BS 903 : Part A42 will complete the revision of BS 903 : Part A15 : 1958 which will therefore be superseded and withdrawn.

This revision is restricted to tests in which the rubber is deformed in compression or in shear. It is considered that measurements in tension are not relevant to product specifications since rubber is seldom deformed in this manner in practical usage.

For tests in shear, a test piece of circular cross section is now allowed as an alternative to the test piece of square cross section and absolute dimensions of the rubber blocks are no longer specified (see **4.2**).

Where possible, it is recommended that results be presented as a creep rate.

The method described in this Part of BS 903 has been submitted to the International Organization for Standardization (ISO) for consideration as the basis for an international standard.

British Standard

Methods of testing vulcanized rubber

Part A15. Determination of creep

0. Introduction

When a constant stress is applied to rubber the deformation is not constant but increases gradually with time; this behaviour is called 'creep'. Conversely, when rubber is subjected to a constant strain, a decrease of the stress in the material takes place; this behaviour is called 'stress relaxation'.

The processes responsible for creep may be either physical or chemical in nature, and under all normal conditions both processes will occur simultaneously. However, at normal or low temperatures and/or short times creep is dominated by physical processes whilst at high temperatures and/or long times chemical processes are dominant. In general, physical creep is found to be directly proportional to logarithmic time, and chemical creep to linear time. Hence it is neither safe to extrapolate time/creep curves in order to predict creep after periods considerably longer than those covered by the test, nor to use tests at higher temperatures as accelerated tests to give information on creep at lower temperatures.

In addition to the need to specify the temperature and time intervals in a creep test, it is also necessary to specify the initial strain and the previous mechanical history of the test piece since these may influence the measured creep particularly in rubbers containing fillers.

1. Scope

This Part of BS 903 specifies test conditions and procedures for the determination of creep in rubber subjected either to compressive forces or to shear forces.

2. References

The titles of the standards publications referred to in this standard are listed on the inside back cover.

3. Definitions

For the purposes of this Part of BS 903, the following definitions apply.

3.1 strain. The total time-dependent deformation produced in a test piece by the application of a constant compressive or shear stress, expressed as a percentage of the initial thickness.

3.2 creep. The increase in deformation which has occurred after a specified time interval, during application of a constant force, expressed as a percentage of the test piece deformation at the commencement of that time interval.

NOTE. This definition of creep has been retained in this Part of BS 903 because it is widely used in the rubber industry. It has the advantage of being reasonably independent of the initial deformation of the test piece. This is not, however, the definition commonly used for non-rubber-like materials, e.g. plastics and metals. For these materials the creep is usually defined as the percentage deformation relative to the original thickness of the test piece, i.e. the percentage strain at any given time.

In view of this disparity of definition, care should be taken when comparing rubber data with that quoted for other materials.

3.3 creep rate. The ratio of creep to the common logarithm of the time interval over which it is measured, expressed as percent per decade.

NOTE. When it is found that the deformation shows a linear dependence when plotted against the logarithm of time, it is preferable to express the results as a creep rate.

4. Test piece

4.1 Test piece for measurements in compression. The test piece shall be a cylindrical disc prepared either by moulding or by cutting from flat sheet of the required thickness.

Cutting shall be carried out by means of a sharp rotating circular die or revolving knife, lubricated with soapy water, and brought carefully into contact with the rubber.

The rubber may be mounted on suitable backing material and the cutting pressure shall be kept small enough to avoid 'cupping' of the cut surface.

Either of the following types of test piece* shall be used, with the following dimensions for the rubber discs.

Type 1 shall have a diameter of 13.0 ± 0.5 mm and a thickness of 6.3 ± 0.3 mm.

Type 2 shall have a diameter of 29.0 ± 0.5 mm and a thickness of 12.5 ± 0.5 mm.

The plane surfaces of the rubber disc shall be bonded to rigid end pieces (see figure 1)†. Bonding to the end pieces shall be carried out either during moulding or subsequently by using a suitable adhesive. The thickness of the end pieces shall be determined prior to bonding.

NOTE. Since rigidity is only required in a radial direction, the end pieces may be of thin metal sheet, with a minimum thickness of 0.25 mm.

4.2 Test piece for measurements in shear. The test piece shall consist of two rubber blocks bonded to three steel plates (see figure 2); it shall be of either circular or square cross section. The diameter (or side, in the case of a square cross section) of the rubber blocks shall be at least four times the thickness.

*These test piece sizes correspond to those specified in BS 903 : Part A6 and the type 2 dimensions also correspond to the dimensions of the test piece specified in BS 903 : Part A4.

†This measure improves the reproducibility of the test method.

NOTE 1. It is not proposed to specify absolute dimensions for the rubber blocks because of the range of sensitivities of the testing machines available. However, the difficulties of ensuring uniform vulcanization in thick rubber blocks suggest that thicknesses of more than 12 mm should be avoided.

NOTE 2. The ratio of diameter (or side) to thickness will ensure that the deformation is essentially simple shear of the calculated magnitude and that the apparent shear modulus differs by less than 3 % from the true value.

A typical test piece contains rubber blocks with a circular cross section of 25.0 ± 0.1 mm diameter, and a thickness of 5.0 ± 0.1 mm.

The test pieces shall be prepared preferably by moulding directly on to steel plates prepared and treated in accordance with a normal adhesion system. Alternatively the rubber blocks shall be separately moulded, or cut from vulcanized sheet of the appropriate thickness, and then bonded to the steel plates using a suitable adhesive. The thickness of the steel plates used shall be determined prior to moulding or bonding.

NOTE 3. A suitable form of mould for producing test pieces with circular cross section is detailed in figure 3.

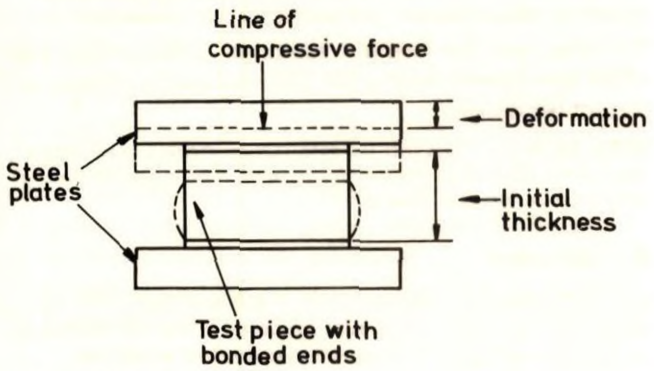


Figure 1. Test piece in compression

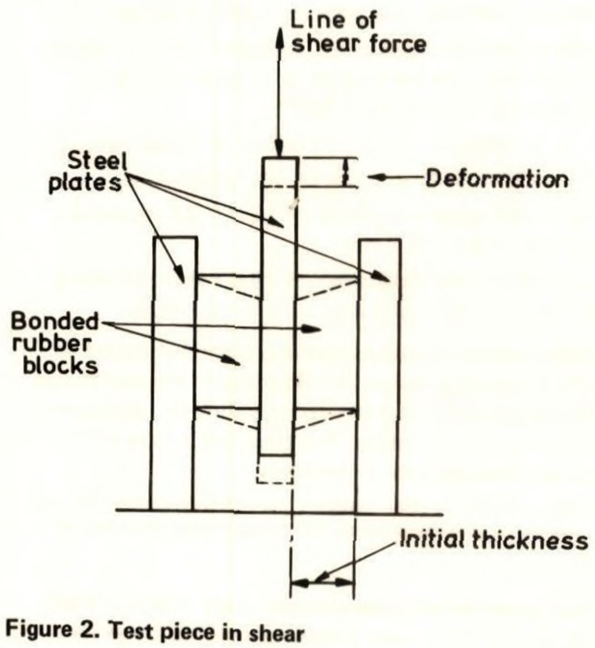


Figure 2. Test piece in shear

5. Apparatus

The following apparatus is required.

5.1 *Compression device,* consisting of

(a) a fixed, flat steel plate;

(b) a moveable, flat steel plate, parallel to the fixed plate;

(c) means of applying a force to the moveable plate;

(d) means of determining the deformation of the test piece.

The steel plates shall be sufficiently rigid to withstand the applied force without bending and of sufficient size to ensure that the whole of the test piece, when compressed between the plates, remains within the area of the plates. The fixed plate shall be rigidly mounted so that it does not move in any direction under the action of the compressive force. The moveable plate shall be able to move in a friction-free manner in one direction only, i.e. in the direction coincident with the axis of the test piece (see figure 1).

The mechanism for applying the force shall be designed so that the full force can be applied within 6 s, with negligible overshoot, and so that the applied force can then be maintained constant at the required value. The mechanism shall also ensure that the line of action of the applied force remains coincident with the axis of the test piece for the duration of the test.

The means of determining the deformation of the test piece shall be such that measurements, to an accuracy of ± 0.1 % of the test piece thickness, can be made at different times after the full force has been applied.

5.2 *Shear device,* consisting of

(a) a fixture to hold the test piece;

(b) means of applying a shear force to the test piece;

(c) means of determining the deformation of the test piece.

The fixture for holding the test piece shall be designed so that either the outer steel plates are rigidly mounted, with the shear force being applied to the central plate, or the central plate is rigidly mounted, and the shear force is applied to the outer plates. (A typical fixture is shown in figure 4.)

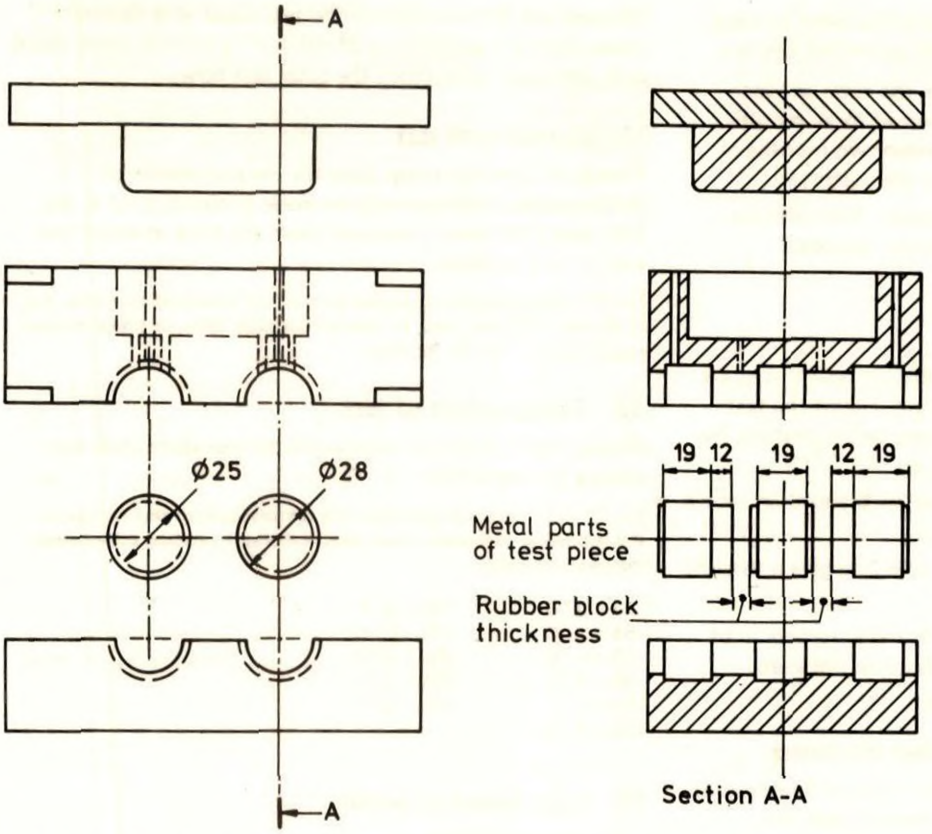
The mechanism for applying the force shall be designed so that the full force can be applied, at an approximately even rate, within 6 s, with negligible overshoot, and so that the applied force can then be maintained constant at the required value. The mechanism shall also ensure that the line of action of the applied force is in the plane of the central plate of the test piece, passing through the centre of the plate in a direction perpendicular to the undeformed rubber blocks (see figure 2), and that this line of action is maintained for the duration of the test.

The fixture and the force applying mechanism shall ensure that the movement of the central plate of the test piece relative to the outer plates (or vice versa) occurs in a friction-free manner and only in the direction of the line of action of the applied force.

The means of determining the deformation of the test piece shall be capable of measuring the relative deflection of the central plate with respect to the outer plates, to an accuracy of ± 0.1 % of the test piece thickness, at different times after the full force has been applied.

5.3 *Temperature controlled chamber,* complying with the requirements of BS 903 : Part A32 and capable of maintaining the required air temperature within the tolerances given in clause **12**. Circulation of the air shall be achieved by means of a fan.

If it is not possible for the complete compression device, or the complete shear device, as appropriate, to be enclosed in the temperature controlled chamber, the chamber and the device shall be designed so as to minimize changes in the temperature of the test piece caused by conduction through metal parts to outside the chamber.



All dimensions are in millimetres

Figure 3. Suitable mould for typical double bonded shear test piece

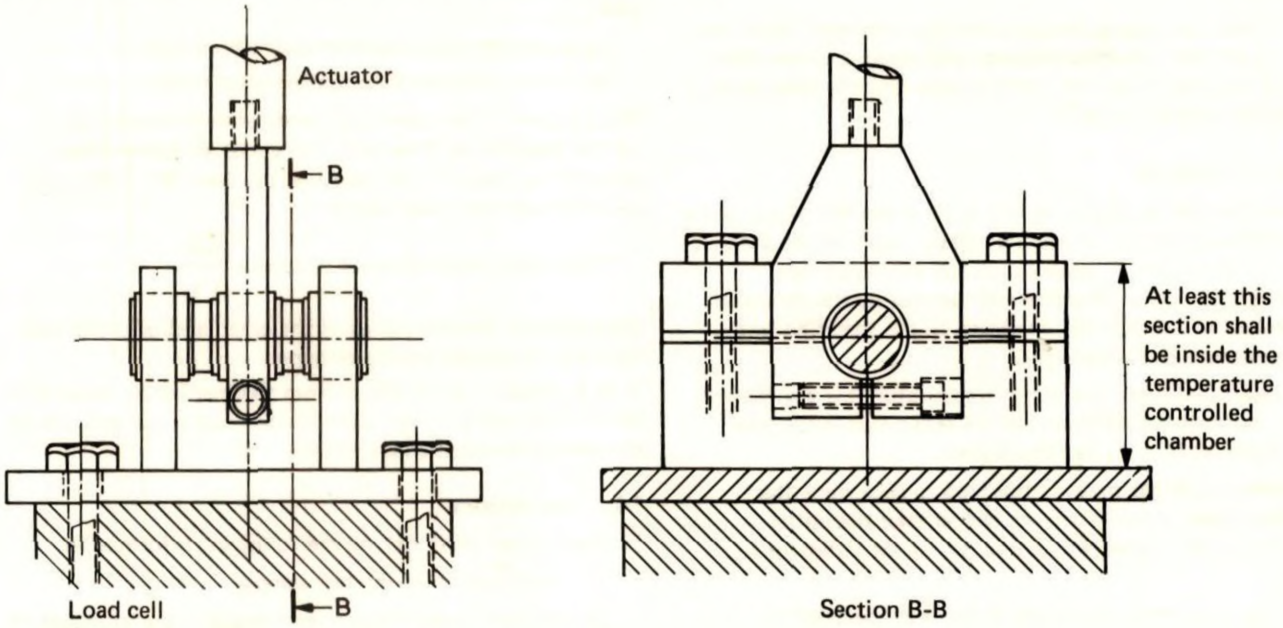


Figure 4. Typical testing fixture for double bonded shear test piece

5.4 *Micrometer dial gauge and stand.* The gauge shall have a scale graduated in unit divisions of 0.01 mm and shall comply with the relevant requirements of BS 907, particularly the requirements concerning the accuracy of calibration. It shall be firmly held in a rigid stand over a flat baseplate of diameter at least 50 mm. The gauge shall be fitted with a flat contact perpendicular to the plunger and parallel to the base-plate, and shall operate with a foot pressure of 22 ± 5 kPa.

5.5 *Stop-clock.*

6. Number of test pieces

Three test pieces shall be used.

7. Time interval between vulcanization and testing

For all test purposes, the minimum time between vulcanization of the rubber and mechanical conditioning of the test pieces (see clause 8) shall be 16 h.

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

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

Samples and test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing.

8. Mechanical conditioning

8.1 It is known that the reproducibility of results may be improved by previous mechanical strain. Hence the test piece shall be mechanically conditioned before testing, by the following procedure, carried out at 23 ± 2 °C.

8.2 Compress the test piece by about 25 % and then return it to approximately zero deflection.

8.3 Repeat the operation described in **8.2** to give a total of five deformations.

8.4 A minimum of not less than 16 h and a maximum of not more than 48 h at 23 ± 2 °C shall occur between mechanical conditioning and testing.

9. Determination of initial rubber thickness

9.1 Compression test piece. Measure the total thickness of the complete test piece, either after mounting in the compression device, or separately using the micrometer dial gauge and stand. Determine the initial thickness of the rubber by subtracting the thickness of the end pieces (see **4.1**).

9.2 Shear test piece. Measure the total sandwich thickness using the micrometer dial gauge and stand and determine the thickness of the rubber by subtracting the thickness of the steel plates (see **4.2**).

10. Procedure

Assemble the apparatus so that at least the flat plates of the compression device, or the test piece holder of the shear device (see figure 4), as appropriate, are inside the temperature controlled chamber, and bring the device, or that part of it inside the chamber, to the specified test temperature (see clause 12).

Keep the test piece at the test temperature for a sufficient time to reach equilibrium (see BS 903 : Part A32), and then mount it in the testing device.

Depending on the means employed to determine the deformation of the test piece, either take the initial reading of the measuring device, or set the indicator to zero.

Choose an appropriate series of times as specified in clause **11**.

Apply the force to the test piece so that the full force is is reached in not more than 6 s (0.1 min) and without significant overshoot.

Start the stop-clock.

The required force shall be such that it will give a strain of 20 ± 2 % when measured after 1 min from the moment that the full force is reached.

NOTE. A trial run on a separate test piece may be necessary to determine the required force.

Measure the deformation of the test piece at different times after the application of the full force, this force being held constant throughout the total test time.

11. Duration of test

For short duration creep tests the measurements of deformation shall normally be made at times, t, of 1, 10, 100, and 1000 min, measured from the time at which the full force is applied.

NOTE. Measurements may also be made at intermediate times, e.g. 3, 30, and 300 min, and, for longer duration tests, at times in days, e.g. 1, 2, 4, 7, 14, 21, 28 days.

12. Temperature of test

During any one particular test the temperature shall not change by more than ± 1 °C.

NOTE. It is recommended that the temperature of test be one of the following, although other temperatures, including sub-normal, may also be used.

23 ± 2 °C	150 ± 2 °C
55 ± 1 °C	175 ± 2 °C
70 ± 1 °C	200 ± 2 °C
85 ± 1 °C	225 ± 2 °C
100 ± 1 °C	250 ± 2 °C
125 ± 1 °C	

13. Calculation of results

Calculate the creep as

$$\text{Creep (\%)} = \frac{(D_t - D_1)}{D_1} \times 100$$

where

D_1 is the test piece deformation after 1 min

D_t is the test piece deformation after t min

Plot a graph of the measured values of deformation (D_t) against logarithmic time and, if it yields an approximate straight line, measure the slope of the line, $\Delta D_t / \Delta(\log_{10} t)$, and calculate the creep rate as

$$\text{Creep rate (\% per decade)} = \text{slope} \times \frac{100}{D_1}$$

Calculate the median values of the creep and/or creep rate from the individual results obtained.

If an individual result differs from the median by more than 10 % of the median, test three further test pieces and report the median value of all six results.

14. Test report

The test report shall include the following information.

(a) Identification of the test sample.

(b) Whether measurements were made in compression or shear, and, if compression, whether type 1 or type 2 test pieces were used, or, if shear, the dimensions of the test pieces.

(c) The temperature and duration of test.

(d) The median values of the creep and/or creep rate after the chosen durations.

(e) The number of test pieces measured.

(f) The method used, i.e. BS 903 : Part A15.

(g) The date of test.

Standards publications referred to

BS 903 Methods of testing vulcanized rubber
Part A4 Determination of compression stress-strain
Part A6 Determination of compression set after constant strain
Part A32 General directions for achieving elevated or sub-normal temperatures
Part A42* Determination of stress relaxation in compression
BS 907 Dial gauges for linear measurement

*In course of preparation and referred to in foreword only.

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