Determination of tensile stress/strain properties of rubber

DIN 53 504

ICS 83.060

Supersedes March 1985 edition.

Prüfung von Kautschuk und Elastomeren; Bestimmung von Reißfestigkeit, Zugfestigkeit, Reißdehnung und Spannungswerten im Zugversuch

In keeping with current practice in standards published by the International Organization for Standardization (ISO), a comma has been used throughout as the decimal marker.

See Explanatory notes for connection with draft International Standard ISO/DIS 37: 1989 published by the International Organization for Standardization.

All dimensions are in mm.
For general tolerances, accuracy grade m as specified in ISO 2768 shall apply.

1 Scope and field of application

The test method specified in this standard serves to determine the tensile strength at break, tensile stress at yield, elongation at break and stress at a given strain of rubber test pieces of specified shape when these are stretched to rupture at a constant rate of traverse.

It is advisable that the full force-extension curve or at least part of it be plotted, since the behaviour of rubber when subjected to tensile stress cannot be adequately characterized by the tensile strength and elongation at break.

2 Concepts

2.1 Tensile strength at break

The tensile strength at break, σ_R, is the ratio of the force at break, F_R, to the initial cross-sectional area of the test piece, A_0.

2.2 Tensile stress at yield

The tensile stress at yield, σ_{max}, is the ratio of the maximum measured force, F_{max}, to the initial cross-sectional area of the test piece, A_0.

NOTE: If the tensile testing of rubber is carried out at or above ambient temperature, force F_R is generally equal to force F_{max}.

2.3 Elongation at break

The elongation at break, ε_R, is the ratio of the change in length at break, $L_R - L_0$, to the initial gauge length of the test piece, L_0. For ring test pieces, L_0 is the internal circumferential length, and for dumb-bell test pieces, the distance between two gauge marks.

2.4 Stress

The stress at a given strain, σ_i, is the ratio of the tensile force applied to achieve a given elongation, F_i, to the initial cross-sectional area, A_0.

NOTE 1: Where ring test pieces are used, the elongation shall be referred to the initial mean circumferential length of the ring, U_m, not its internal circumferential length.

NOTE 2: The use of the term 'modulus' (e.g. modulus of elasticity) in this context is incorrect and should be avoided since it normally applies only to materials for which there is a proportional relationship between stress and strain, which is not the case for rubber, even where the strain is low.

NOTE 3: In addition to the stress as defined here, the elongation at a given stress, as specified in ISO/DIS 37, may be determined.

2.5 Force-extension curve

The force-extension curve represents the relationship between tensile force and extension of a test piece during tensile testing up to rupture.

3 Designation

Designation of a tensile test on rubber carried out as specified in this standard using an R 1 ring test piece:

Test DIN 53 504 – R 1

Continued on pages 2 to 7.

4 Apparatus

4.1 Instrument for measuring test piece thickness

For measuring the test piece thickness, a thickness gauge as specified in subclause 5.2 of DIN 53 534, May 1994 edition, shall be used.

4.2 Instruments for determining the initial cross section of ring test pieces

For determining the mean initial cross section of ring test pieces, a balance permitting the test pieces mass in air and in water to be established shall be used.

4.3 Tensile testing machine

The tensile testing machine shall conform to class 1 as specified in DIN 51 221. For tests to be carried out at other than ambient temperature, a controlled-atmosphere test chamber shall be used. The temperature in the chamber shall be measured in the vicinity of the test piece and shall be kept constant to within ± 2°C during the test. For further details, see subclause 6.1.

For testing ring test pieces, the testing machine shall be equipped with one power-driven and one freely rotatable pulley. The distance traversed by the former shall be about 50 mm for one rotation. It shall be possible to move the pulleys close enough together to permit the test pieces to be fitted without undue strain.

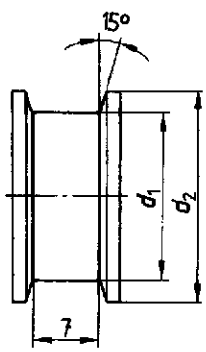

Figure 1: Testing machine pulley

Table 1: Pulley dimensions

Test piece	d_1	d_2	Spacing of pulley axes at start of test
R 1	22,3	26	35,0
R 2	18,3	22	28,7

For testing dumb-bell test pieces, the machine shall be equipped with two grips designed to enable the longitudinal axis of the test piece to be aligned to coincide with the strain axis of the machine at any time, which can be effected using a template. The test piece shall be held so as to prevent slip relative to the grips as far as possible. Suitable grips are those which maintain or even increase the pressure on the test piece as the stress increases. The grips shall not cause premature rupture of the test piece.

The testing machine shall permit both force-extension curves or stress-strain curves to be plotted. The extensometer shall be suitable for determining the change in gauge length of the test piece at any moment during the test and shall have as low an inertia as possible. When an extensometer attached to the test piece is used instead of a contact-free extensometer, there shall be no sign of damage to the test piece nor any slippage between the extensometer grips and the test piece.

5 Test pieces

5.1 Shape

Test results obtained for test pieces of different shape are not comparable.

5.1.1 Ring test pieces

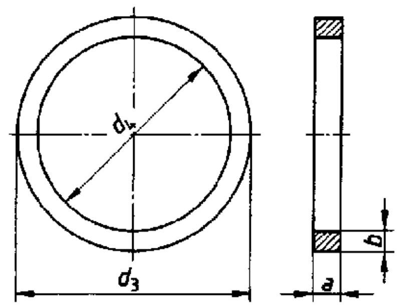

Figure 2: Ring test piece

Table 2: Dimensions of ring test pieces

		Ring	
		R 1	R 2
External diameter, d_3	$^{+0,05}_{0}$	52,6	44,6
Internal diameter, d_4	$^{+0,05}_{0}$	44,6	36,6
Width, $b = \frac{d_3 - d_4}{2}$	±0,1	4,0	4,0
Thickness, a		4 ± 0,2 or 6,3 ± 0,3	4 ± 0,2 or 6,3 ± 0,3
Initial gauge length, L_0: a) internal circumferential length (for elongation at break): $U_i = \pi \cdot d_4$		140,1	115,0
b) mean circumferential length (for stress): $U_m = \pi \left(\frac{d_3 + d_4}{2}\right)$		152,7	127,5
Dimensions d_3 and d_4 relate to the dies. Type R 1 test pieces should preferably be used.			

5.1.2 Dumb-bell test pieces

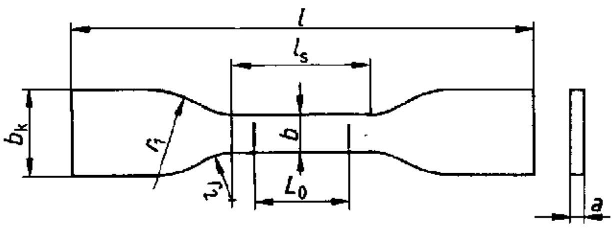

Figure 3: Dumb-bell test piece

Table 3: Dimensions of dumb-bell test pieces

	Test piece type			
	S 1	S 2	S 3	S 3A
Minimum overall length, l	115	75	35	50
Width of ends, b_k	25	12,5	6	8,5
Length of narrow parallel portion, l_s	33	25	12	16
Width of narrow parallel portion, b (± 0,05)	6	4	2	4
Small radius, r_1	25	12,5	3	10
Large radius, r_2	14	8	3	7,5
Thickness, a	2 ± 0,2	2 ± 0,2	1 ± 0,1	2 ± 0,2
Initial gauge length, L_0	25	20	10	10
The dimensions given relate to the cutters. Although the width of the ends of dumb-bells and the small and large radii may deviate from the dimensions specified, the symmetry shall be maintained. S 2 dumb-bell test pieces shall preferably be used.				

5.2 Preparation of test pieces

Where test pieces are prepared separately, choose the sheet form. The thickness of the sheets shall be equal to the thickness of the final test pieces.

If the test pieces cannot be cut directly from finished components, cut out sheets of appropriate thickness so that test pieces can be taken from these.

Remove any unevenness by grinding.

The test piece thickness shall not deviate by more than ± 2,5 % from the median from three individual measurements.

Prepare the test pieces as described in ISO 4661-1, preferably using a circular cutter in the case of ring test pieces.

It should be noted that test results obtained for test pieces of a thickness other than specified in tables 2 and 3 (e.g. where the test pieces are to be taken from finished components) cannot be simply compared with those obtained for test pieces whose thickness lies within the specified range. The former results are to be identified in the test report.

5.3 Number of test pieces

Use at least three test pieces, or at least seven in arbitration cases. For dumb-bell pieces, take three (or seven) test pieces each from two directions at right angles to each other, choosing the directions parallel and perpendicular to the grain of the material if the latter can be established.

5.4 Reference marks

When using non-contact extensometers to measure the elongation of dumb-bell test pieces, make reference marks on the test pieces to define the gauge length, taking care that the marks and the marker used do not adversely affect the material to be tested. The marker lines shall be as narrow as possible and approximately equidistant from the centre. Determine the distance between them to within 1 % or less.

6 Procedure

6.1 General

Carry out the test at (23 ± 2) °C ('ambient temperature', for short) or, subject to agreement, at one of the following temperatures (cf. ISO 471: 1983): −70, −55, −40, −25, −10, 0, 40, 55, 70, 85, 100, 125, 150, 175, 200, 225, or 250 °C.

Prior to the test, condition the test pieces for a least 20 minutes at the test temperature. Testing shall not be carried out less than 16 hours, and in arbitration cases, less than 72 hours, after vulcanization.

> NOTE: When testing ring test pieces below ambient temperature, replace the steel pulleys by those made of a material with a low coefficient of friction (e.g. tetrafluoroethylene) to ensure a uniform distribution of stresses in the ring.

6.2 Determination of test piece dimensions

Prior to testing, measure the test piece thickness at ambient temperature at not less than three points, that of dumb-bell test pieces within L_0, preferably at the two ends and in the centre. Calculate the initial cross-sectional area from the mean thickness and the width of the narrow portion (distance between the cutting edges) of the die. In arbitration cases, measure the width of the narrow portion.

The initial cross-sectional area of ring test pieces may alternatively be determined from the density using Archimedes' principle by weighing the sample in air and in water.

Calculate A_0 using equation (1):

$$A_0 = \frac{W_1}{\varrho \cdot \pi \cdot (d_3 + d_4)/2} \qquad (1)$$

where

- W_1 is the mass of the test piece, in g;
- ϱ is the density of the test piece, in g/cm^3;
- d_3 is the external diameter of a type R 1 test piece, in mm;
- d_4 is the internal circumferential length of a type R 1 test piece, in mm.

Determine the density, ϱ, by the same method, using equation (2):

$$\varrho = \frac{W_1}{W_1 - W_2} \cdot \varrho_W \quad (2)$$

where

W_1 is the mass of a type R 1 test piece weighed in air, in g;

W_2 is the mass of a type R 1 test piece weighed in water, in g;

ϱ_W is 0,9975 g/cm^3 (density of water at 23 °C).

6.3 Dumb-bell test piece gripping

A template should be used to insert the test piece in the grips in such a way that its longitudinal axis coincides with the strain axis. Ensure that as large an area as possible of the ends is gripped, but not any regions having a width less than b_k and that slippage relative to the grips is prevented.

6.4 Tensile testing

6.4.1 Dumb-bell test pieces

Use of a contact-free method of extension measurement is to be given preference, since here the origin of the force-extension curve is not shifted as a result of the initial stress.

Adopt the following procedure when using a contact-free extensometer:

a) mark the initial gauge length, L_0, as described in subclause 5.4, without stressing the test piece;

b) insert the piece in the grips, as described in subclause 6.3;

c) apply an initial stress of less than 0,1 MPa at a rate of less than 50 mm/min and stop the machine;

d) switch on the extensometer system to find the reference marks and to measure the L_0, as changed by the initial stress;

e) start the machine at an appropriate rate (200 mm/min for S 2, S 3 and S 3A type test pieces, and 500 mm/min for an S 1 type test piece) and plot the force-extension curve. The position of origin of the curve is a function of the initial stress and the change in initial gauge length.

Adopt the following procedure when using an extensometer attached to the test piece:

a) insert the test piece in the grips, as described in subclause 6.3;

b) apply an initial stress of less than 0,1 MPa at a rate of less than 50 mm/min and stop the machine;

c) bring the extensometer into contact with the test piece with a spacing of contact point equal to L_0;

d) start the machine at an appropriate rate of traverse (see above) and plot the force-extension curve.

This method causes a slight falsification of results owing to the initial stress applied. Accordingly, the initial stress selected shall be as small as possible while still sufficient to tauten the test piece.

Do not evaluate tests where the rupture occurs outside the gauge length.

6.4.2 Ring test pieces

For testing ring test pieces, extension measurement shall be based on the spacing of pulley axes (as specified in table 1), any change in extension being recorded by the system for measuring the distance traversed.

After fitting the test piece on the pulleys without tension, the pulley spacing shall be set at the value specified in table 1, using a rate of traverse of 50 mm/min. Then start the machine, with the rate set at 500 mm/min and plot the force-extension curve.

6.5 Determination of stress/strain properties

If it is not possible or not required to plot the force-extension curve in its entirety, measure the tensile force at two of the following elongation values: 50, 100, 200, 300, 500 %.

Where the stress is to be determined for ring test pieces, the elongation (cf. subclause 2.4) is to be based on the initial mean circumferential length, and the elongation at break on the internal circumferential length. Thus, in order to obtain the elongation values given in the 1st paragraph, select the following elongation at break (ε^*) values:

type R 1 test pieces: 54,5; 109; 217,9; 326,9; 544,8 %;

type R 2 test pieces: 55,5; 110,9; 221,9; 332,8; 554,6 %.

Calculate these values, ε^*, as percentages, using equation (3):

$$\varepsilon^* = \varepsilon = \frac{U_m}{U_i} = \varepsilon \frac{d_3 + d_4}{2\, d_4} \quad (3)$$

where

ε is the required elongation (based on U_m), as a percentage;

U_m is the mean circumferential length of the test piece, in mm;

U_i is the internal circumferential length of the test piece, in mm;

d_3 is the test piece external diameter, in mm;

d_4 is the test piece internal diameter, in mm.

7 Evaluation

7.1 Tensile strength at break

Calculate the tensile strength at break, σ_R, in MPa or N/mm^2, from the initial cross-sectional area measured as in subclause 6.2 for each test piece, using equation (4) for ring test pieces and equation (5) for dumb-bell test pieces:

$$\sigma_R = \frac{F_R}{2\, A_0} \quad (4)$$

$$\sigma_R = \frac{F_R}{A_0} \quad (5)$$

where

F_R is the force at break, in N;

A_0 is the initial cross-sectional area, in mm^2.

NOTE: Measurements of the tensile strength at break made on S 2 type test pieces generally yield higher values than those made on R 1 type test pieces.

7.2 Tensile stress at yield

Calculate the tensile stress at yield, σ_{max}, in MPa or N/mm^2, using equation (6) for ring test pieces and equation (7) for dumb-bell test pieces:

$$\sigma_{max} = \frac{F_{max}}{2\,A_0} \qquad (6)$$

$$\sigma_{max} = \frac{F_{max}}{A_0} \qquad (7)$$

where

F_{max} is the maximum force, in N;

A_0 is the initial cross-sectional area, in mm^2.

7.3 Elongation at break

Calculate the elongation at break, ε_R, as a percentage, using equation (8):

$$\varepsilon_R = \frac{L_R - L_0}{L_0} \cdot 100 \qquad (8)$$

where

L_R is the gauge length at break, in mm;

L_0 is the initial gauge length, in mm.

Calculate the gauge length (internal circumferential length) at break, L_R, in mm, for ring test pieces, using equation (9):

$$L_R = 2\,D_R + U \qquad (9)$$

where

D_R is the spacing of pulley axes at break, in mm;

U is the circumferential length of one pulley, in mm.

7.4 Stress

Calculate the stress at a given strain, F_i, in MPa or N/mm^2, using equation (10) for ring test pieces and equation (11) for dumb-bell test pieces:

$$\sigma_i = \frac{F_i}{2\,A_0} \qquad (10)$$

$$\sigma_i = \frac{F_i}{A_0} \qquad (11)$$

where

F_i is the force at strain i (cf. subclause 6.5);

A_0 is the initial cross-sectional area, in mm^2.

Use the numerical value of the given strain as a subscript for symbol σ, e.g. σ_{100} to denote 100 % elongation.

7.5 General

Calculate the median[1]) and the range[1]) on the basis of the individual values.

NOTE: The measured values of tensile strength at break and elongation at break can be evaluated using the Weibull distribution, while the stress values can be represented as a normal distribution.

[1]) See DIN 53 598 Part 1.

8 Test report

The test report shall refer to this standard and include the following details:

a) type and designation of the product tested;

b) method used to prepare test pieces;

c) type and thickness of test pieces and cross-sectional area in the case of ring test pieces;

d) direction of sampling;

e) strain measuring system used, with details of the resolution and accuracy;

f) initial stress used, expressed in MPa or N/mm^2;

g) tensile strength at break, σ_R, or tensile stress at yield, σ_{max}, in MPa or N/mm^2, to the nearest 0,1 MPa or 0,1 N/mm^2;

h) elongation at break, ε_R, as a percentage, to the nearest integer;

i) stress at a given strain, σ_i, in MPa or N/mm^2, to the nearest 0,1 MPa or 0,1 N/mm^2;

(for g) to i): Specify median, range, number of test pieces or all the individual values.)

j) any deviations from the specifications of this standard;

k) date of testing.

9 Precision

To evaluate the test results, table 4 lists precision data for three different tensile strength levels. The data shown were determined on S 2 type test pieces and cover tensile strength at break, elongation at break and stress at 100 % strain. Seventeen laboratories participated in 1989 in a national interlaboratory test in which the short-term precision was determined, i.e. the repeatability limit and reproducibility limit apply to measurements taken within one week. The test was organized and evaluated as specified in ISO 5725.

Table 4: Data on precision

Mean tensile strength at break, $\bar{x}$, in MPa	Repeatability limit		Reproducibility limit	
	r MPa	r_{rel} %	R MPa	R_{rel} %
22,4	2,5	11,2	2,6	11,6
31,9	1,3	4,1	1,9	6,0
33,8	5,2	15,4	5,3	15,7
Mean elongation at break, $\bar{x}$, as a percentage	**Repeatability limit**		**Reproducibility limit**	
	r %	r_{rel} %	R %	R_{rel} %
390	27,5	7,1	53,1	13,6
537	55,6	10,4	76,8	14,3
671	57,4	8,6	97,2	14,5
Mean stress at 100 % strain, $\bar{x}$, in MPa	**Repeatability limit**		**Reproducibility limit**	
	r MPa	r_{rel} %	R MPa	R_{rel} %
3,5	0,6	17,1	0,9	25,7
2,7	0,5	18,5	0,6	22,2
2,4	0,3	12,5	0,5	20,8

Repeatability limit
(same operator, same apparatus)

The difference between two individual test results obtained by the same operator using the same apparatus on identical test material within an interval of one week shall not exceed the repeatability limit, r, on average more than once in 20 cases.

Reproducibility limit
(different operators, different apparatus)

The difference between two individual and independent test results obtained by two operators, working in different laboratories, on identical test material shall not exceed the reproducibility limit, R, on average more than once in 20 cases.

Standards referred to

DIN 51 221 Part 1	Tensile testing machines; general requirements
DIN 53 534	Determination of the linear dimensions of rubber test pieces and finished components
DIN 53 598 Part 1	Statistical evaluation of random samples with examples from tests on rubber and plastics
DIN EN 61	Glass reinforced plastics; tensile test
ISO 471 : 1983	Rubber; standard temperatures, humidities and times for the conditioning and testing of test pieces
ISO 527-1 : 1993	Plastics; determination of tensile properties; general principles
ISO 527-2 : 1993	Plastics; determination of tensile properties; test conditions for moulding and extrusion plastics
ISO 2768-1 : 1989	Tolerances for linear and angular dimensions without individual tolerance indications
ISO 4661-1 : 1993	Rubber, vulcanized or thermoplastic; preparation of samples and test pieces
ISO 5725 : 1986	Precision of test methods; determination of repeatability and reproducibility for a standard test method by interlaboratory tests
ISO 5893 : 1993	Rubber and plastics test equipment; tensile, flexural and compression types (constant rate of traverse); description
ISO/DIS 37	Rubber, vulcanized and thermoplastic; determination of tensile stress-strain properties

Previous editions

DIN DVM 3504 = DIN 53 504 Part 1: 11.38; DIN DVM 3504 = DIN 53 504 Part 2: 12.40; DIN 53 504: 10.65, 05.69, 08.75, 03.85.

Amendments

The following amendments have been made to the March 1985 edition.

a) The specifications of this standard have been harmonized with draft International Standard ISO/DIS 37, this involving a restructuring of the text.

b) Test equipment (grips, extensometer) has been described in more detail.

c) Tolerances have been specified for the test pieces thickness (± 2,5%).

d) Specifications regarding reference marks have been included (cf. subclause 5.4).

e) Temperatures for testing at a temperature other than ambient temperature have been specified.

f) The points at which the thickness of dumb-bell pieces is to be measured have been specified.

g) Supplementary specifications have been given in subclause 6.2 with regard to the buoyancy method of determining the cross-sectional area of ring test pieces.

h) Additional information on the gripping of dumb-bell test pieces has been given in subclause 6.3.

i) The tensile testing procedure has been described in more detail.

j) An initial stress for tautening dumb-bell test pieces (0,1 MPa) has been specified.

p) The specifications regarding the test report have been amended and the clause on precision has been included.

Explanatory notes

This standard has been prepared by Technical Committee *Prüfung physikalischer Eigenschaften von Kautschuk und Elastomeren* of the *Normenausschuß Materialprüfung* (Materials Testing Standards Committee), adopting the specifications of ISO/DIS 37 in substance, except for the following points.

Test pieces of type R 2 have been retained, as has been the pulley diameter for testing type R 1 test pieces.

As in its March 1985 edition, the standard deviates from ISO/DIS 37 in specifying a rate of traverse of 200 mm/min for type S 2 test pieces in order to preclude any difficulties in the extension measurement.

No specifications have been made regarding limits of error of extensometers for measuring dumb-bell test pieces (cf. subclause 4.3), since the available strain gauges do not comply with the specifications for accuracy (1 %) given in Standards ISO 527 and DIN EN 61. Here, it does seem reasonable to adopt the limits of error given in ISO 5893 (2 %), it being the machine manufacturer's responsibility to specify the smallest value for which this value applies.

In addition to the tests on R 1 and R 2 test pieces, the *Verband der Automobilindustrie e.V.* (VDA) (German Motor Vehicle Industries Association) specifies that tests be carried out on circular and rectangular gaskets using further spacings and pulley axes at the start of test (cf. table 5).

Table 5: Pulley axes spacing as a function of the test piece internal diameter for gaskets used in the motor industry

Ring internal diameter, d_4	d_1 (± 0,2)	d_2	Spacing of pulley axes at start of test*)
34,8 to 41,4	18,3	22	$0{,}5 \cdot \pi (d_4 - d_1)$
Over 41,4 up to 51,2	22,3	26	$0{,}5 \cdot \pi (d_4 - d_1)$
Over 51,2 up to 64,3	28,3	32	$0{,}5 \cdot \pi (d_4 - d_1)$
Over 64,3 up to 82,3	36,3	40	$0{,}5 \cdot \pi (d_4 - d_1)$
Over 82,3 up to 108,4	47,3	51	$0{,}5 \cdot \pi (d_4 - d_1)$
Over 108,4 up to 142,8	61,8	65,5	$0{,}5 \cdot \pi (d_4 - d_1)$
Over 142,8 up to 192,0	83,4	87,1	$0{,}5 \cdot \pi (d_4 - d_1)$
Over 192,0 up to 257,0	112,4	116,1	$0{,}5 \cdot \pi (d_4 - d_1)$
*) In the test, the spacing is set to the nearest millimetre.			

International Patent Classification

C 08 L 021/00
G 01 L 001/00
G 01 N 033/44