

JIS

JAPANESE INDUSTRIAL STANDARD

**Physical testing methods
for vulcanized rubber**

JIS K 6301—1995

Translated and Published

by

Japanese Standards Association

Printed in Japan

JAPANESE INDUSTRIAL STANDARD

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May, 1996

ERRATA

Page	Position	Correct
13	Fig. 5, Type A	"<u> </u>" denotes the portion corrected. Type A Scale (degree) Distance between loading surface and tip of indenter (mm) Load applied to tip of indenter by spring (N (gf))

Remarks: This errata is for correcting the first edition of this Standard.
Japanese Standards Association

In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

JAPANESE INDUSTRIAL STANDARD

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Physical testing methods
for vulcanized rubber

K 6301-1995

1. General rules

1.1 Scope This Japanese Industrial Standard specifies physical testing methods for the vulcanized rubber (excluding ebonite and foam rubber).

Remarks 1. The following standards are cited in this Standard:

- JIS B 1501 Steel balls for ball bearings
- JIS B 4401 Reamers for morse tapers
- JIS B 7503 Dial gauges
- JIS G 3101 Rolled steel for general structure
- JIS K 2201 Gasoline for industrial purpose
- JIS K 2249 Testing methods for density of crude oil and petroleum products, and petroleum measurement tables based on a reference temperature of 15 centigrade degrees products
- JIS K 2256 Testing methods for aniline point and mixed aniline point of petroleum
- JIS K 2265 Testing methods for flash point of crude oil and petroleum products
- JIS K 2283 Crude petroleum and petroleum products — determination of kinematic viscosity and calculation of viscosity index from kinematic viscosity
- JIS K 3504 Testing method for acid value of fats and oils
- JIS K 8001 General rule for test methods of reagents
- JIS K 8530 Potassium bromate
- JIS K 8637 Sodium thiosulfate pentahydrate
- JIS K 8680 Toluene
- JIS K 8913 Potassium iodide
- JIS K 8922 Potassium iodate
- JIS K 9007 Potassium dihydrogenphosphate
- JIS K 9019 Disodium hydrogenphosphate 12-water
- JIS R 6253 Waterproof abrasive papers
- JIS Z 8401 Rules for rounding off of numerical values

2. The units and numerical values given in { } in this Standard are based on the traditional units and appended for informative reference.

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1.2 Test items Test items specified in this Standard shall be as follows:

- (1) Tensile test
- (2) Tension set test
- (3) Hardness test
- (4) Accelerated ageing test
- (5) Peeling test
- (6) Adhesion test to metal
- (7) Tear test
- (8) Compression set test
- (9) Rebound resilience test
- (10) Effect of liquids test
- (11) Low stretch stress test
- (12) Impact brittleness test at low temperature
- (13) Flex test
- (14) Resistance to ozone cracking test
- (15) Compression test
- (16) Stress relaxation test
- (17) Low temperature torsion test
- (18) Low temperature compression set test

2. General conditions for testing

2.1 Standard conditions for laboratory Temperature of the laboratory shall, as a rule, be $20 \pm 10^{\circ}\text{C}$ and the temperature of the test should be recorded in the results of the tests.

2.2 Standard conditions for sample The sample shall, as a rule, be that which has elapsed not less than 24 h after vulcanization and should be kept in a room maintained under standard conditions for not less than 1 h before test.

2.3 Rounding off of results of tests Results of each test shall be rounded off according to JIS Z 8401 and shall be denoted by the number of figures shown in Table 1.

Table 1

Test items	Measured value	Results of tests to be obtained
Tensile strength [MPa {kgf/cm ² }]	First decimal place	First decimal place
Elongation (%)	Integer only	2 significant digits
Tensile stress [MPa {kgf/cm ² }]	First decimal place	First decimal place
Tension set (%)	Integer only	Integer only
Hardness (scale)	Integer only	Integer only
Peeling [kN/m {kgf/cm}]	First decimal place	First decimal place
Adhesion to metal [MPa {kgf/cm ² } or [kN/m {kgf/cm}]	First decimal place First decimal place	First decimal place Integer only
Tear strength [kN/m {kgf/cm}]	First decimal place	Integer only
Compression set (%)	First decimal place	Integer only
Rebound resilience (%)	Integer only	Integer only
Effect of liquids (rate of change in weight and volume) (%)	First decimal place	First decimal place
Low stretch stress [MPa {kgf/cm ² }]	Second decimal place	Second decimal place
Impact brittleness temperature at low temperature (°C)	—	Integer only
Length of crack (mm)	First decimal place	First decimal place
Compression strain (%)	Integer only	Integer only
and load [MPa {kgf/cm ² }]	First decimal place	First decimal place
Rate of residual stress (%)	Integer only	Integer only
Low temperature torsion (°C)	Integer only	Integer only
Low temperature compression set (%)	First decimal place	Integer only

2.4 Record Employed conditions shall be recorded in the results of the test.

3. Tensile test

3.1 Purpose This test shall be performed to measure the maximum stress (referred to as tensile strength) at which breaking of the vulcanized rubber occurs, the elongation at the time of breaking (referred to as elongation) and the stress corresponding to the specified elongation (referred to as tensile stress).

3.2 Test piece

3.2.1 Shape and dimensions of test pieces Shapes and reference dimensions of test pieces shall be as shown in Table 2 and Fig. 1.

Table 2

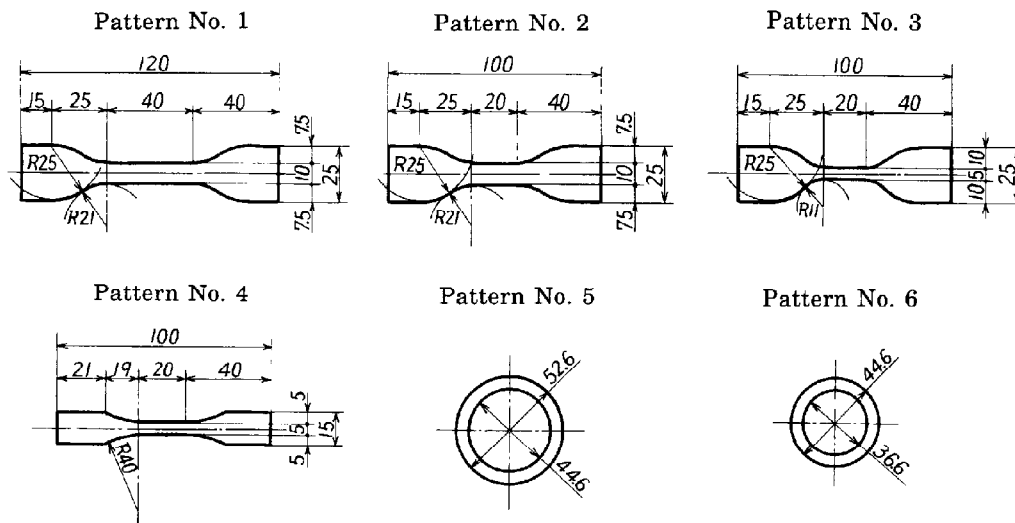
Unit: mm

Shape	Pattern	Dimensions of principal parts			
		Width of parallel portion	Length of parallel portion	Thickness of parallel portion	Gauge length
Dumbbell shape	Pattern No. 1	10	40	3 max.	40
	Pattern No. 2	10	20	3 max.	20
	Pattern No. 3	5	20	3 max.	20
	Pattern No. 4	5	20	3 max.	20

Shape	Pattern	External diameter	Internal diameter	Thickness	Length corresponding to gauge length
Ring shape	Pattern No. 5	52.6	44.6	4 to 6	70
	Pattern No. 6	44.6	36.6	4 to 6	57.5

Fig. 1

Unit: mm



3.2.2 Blanking die Dumbbell shaped test piece shown in Table 2 shall be punched out with an appropriate blanking die corresponding to each pattern. Shape and dimensions of blanking die are shown in Fig. 2 and Table 3. However, dimensions shown in Table 3 where no tolerance is given shall be reference dimensions.

Fig. 2

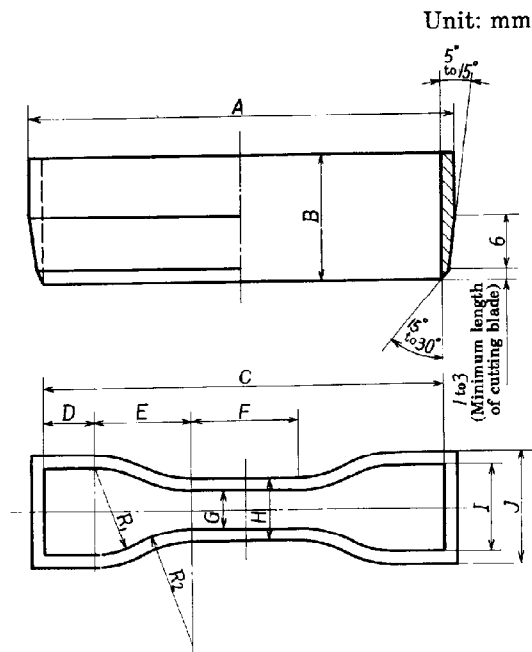


Table 3

Unit: mm

Shape	Pattern	Measured spots of dimensions											
		A	B	C	D	E	F	G	H	I	J	R ₁	R ₂
Dumbbell shape	Pattern No. 1	127	34	120	15	25	40 ⁻² ₀	10.0±0.1	17	25.0±0.5	32	25	21
	Pattern No. 2	107		100			20 ⁺² ₀						
	Pattern No. 3							5.0±0.1	12				11
	Pattern No. 4				21	19				15.0±0.5	22	—	40

3.2.3 Selection of test pieces As a rule, the dumbbell shaped pattern No. 3 test piece shall be used. The pattern No. 1 test piece shall be used for the samples having a little elongation and pattern No. 2 for the sample having a small tensile strength.

Pattern No. 4 shall be used mainly for the sheet of pure rubber stock of not more than 1 mm in thickness. In this case, it is desirable to spread the lubricant such as talc and zinc stearate on the surface of the test piece. Test piece of pattern No. 6 shall be used only when the test piece of pattern No. 5 is difficult to punch out.

Remarks: When the specified test piece is difficult to punch out, it may be permissible to use the test pieces in the forms of rectangular, thread, ring of dimensions other than those specified in this Standard and others.

3.2.4 Sampling and preparation of test pieces Test pieces shall, as a rule, be sampled and prepared by the following methods:

- (1) In case of the dumbbell shaped test piece, it shall, as a rule, be taken in the direction parallel to that of grain of rubber.
- (2) In case of a test piece to be taken directly from the product, slice off the rubber layer of a proper width from the product in the form of flat plate as far as possible according to the following method (in case of the dumbbell shaped test piece, width shall be about 30 mm) and prepare the test piece from it.

When cloth is attached, cut off the sample of a proper width with cloth as it is and slice off or strip off cloth with a small knife or any other suitable tool, while taking care not to give the strain to the rubber as far as possible and ensuring the rubber surface as flat as possible. When there is a pattern on the surface, slice off the rubber layer in the form of flat plate if possible according to the above method while taking care not to give the strain to the rubber as far as possible.

In case of preparation of the dumbbell shaped test piece, if the original thickness of the rubber layer of the sample exceeds 3 mm, the thickness of its rubber layer should be close to 3 mm as far as possible and if the original thickness of rubber layer is not more than 3 mm, its thickness should be close to the original thickness wherever practicable.

When the solvent is used from necessity for the removal of cloth, No. 1 gasoline specified in JIS K 2201 shall be used and after removal, allow it to stand for 1 h or more. Then dry the test piece thoroughly and use it for the test.

- (3) A test piece both surfaces of which are not flat shall be ground by a buffing machine to make them as flat as possible. If possible, grinding shall be performed in such a manner that no heat is generated while avoiding excessive grinding.

After grinding, the thickness of parallel portion of the dumbbell shaped test piece shall, as a rule, be 2 mm to 3 mm. However, if the original thickness of the test piece before grinding is not more than 2 mm, it shall be finished close to the original thickness as far as possible.

- (4) When the test piece is to be taken from a rubber plate vulcanized under the same conditions as the product, take the test piece of the following thickness from the press vulcanized rubber plate.

In the case of the dumbbell shaped test piece 2 mm to 3 mm

In the case of the ring-shaped test piece 4 mm to 6 mm

- (5) Test piece shall be prepared by punching with a cutting die.

When punching is performed, use an elastic sheet such as leather, rubber belt, cardboard and others as the underlay and it should be completed by one blow or by one action. However, in the case of the ring-shaped test piece, a revolving edge may be used.

Test piece shall be prepared by punching after grinding with the buffing machine. If the above operation is impossible, grinding may be performed after punching.

3.2.5 Measurement for thickness and width The thickness and width of the test piece shall be measured by the following methods:

- (1) The thickness gage shall have a scale of 0.01 mm and a flat faced loading disc 5 mm in diameter.
The pressure load applied by the gage shall, as a rule, be 0.785 N {80 gf} and should not fluctuate not less than $\pm 15\%$ in the range of measurement.
- (2) Measurement of the thickness shall be carried out at several spots (in case of the dumbbell shaped test piece, several spots on its parallel portion) and make the lowest measured value as the thickness of the test piece.
However, the measurement should not be performed when the center of the loading disc of the gage protrudes out of the edge of the test piece.
- (3) The width of a blanking die (inside width of the cutting edge) shall be taken as the width of the test piece. However, when a blanking die other than those shown in Table 3 is unavoidable to be used, width (inside width of the cutting edge) shall be measured.
- (4) The sectional area of the test piece shall be calculated by the following formula:

Dumbbell shaped test piece	Thickness (cm) $\times$ width (cm) of parallel portion
Ring-shaped test piece	Thickness (cm) $\times$ width (cm) $\times 2$

3.2.6 Bench mark for measuring elongation A dumbbell shaped test piece shall be marked with bench mark for measuring its elongation (hereafter referred to as "bench mark") by the following method:

- (1) Gauge length (distance between bench marks) shall be in accordance with Table 2.
- (2) Bench marks shall be marked accurately and clearly on the parallel portion of the test piece with the central part of it as the center of the bench marks.

3.2.7 Selection of test pieces Test pieces which are irregular in thickness (in case of dumbbell shaped test piece, thickness of the parallel portion) exceeds 0.1 mm, width (in case of dumbbell shaped test piece, width of the parallel portion) is irregular or having any foreign substance, bubble and flaw shall be discarded.

3.3 Testing apparatus Tension tester which satisfies the following requirements shall be used as the testing apparatus.

3.3.1 Mechanism of tester Tester shall be fitted with a maximum load indicator and a clamp which automatically grips a test piece for the dumbbell shaped one or a device which let a test piece revolve while drawing for the ring-shaped one.

3.3.2 Capacity of tester Tester with a capacity 15 % to 85 % of which corresponds to the maximum load at the time of test shall be used.

3.3.3 Rate of tension Moving rate of the clamp for the test piece shall, as a rule, be 500 ± 25 mm per minute for the dumbbell shaped one and 300 ± 15 mm per minute for the ring shaped one.

3.3.4 Tolerance on the load scale of the tester Tolerance on the load scale of the tester shall be ± 2 %.

3.3.5 Inspection of the tester The tester shall be set correctly and inspected at least once a year.

3.4 Testing method

3.4.1 Fitting of test piece Test piece shall be gripped correctly and accurately by the clamp so that a twist, cutting caused by the clamp and other troubles shall not occur.

3.4.2 Measurement for tensile strength and elongation Measurement for the tensile strength shall be performed by reading the maximum load up to the time of breaking of the test piece with the testing apparatus described under 3.3.

As for the measurement of elongation, in case of the dumbbell shaped test piece, measure the gauge length to mm by a proper method at the time of breaking, and in case of the ring-shaped test piece, measure the distance between clamps at the time of breaking and obtain the numerical value corresponding to the gauge length.

3.4.3 Measurement of tensile stress Tensile stress is the stress in the test piece when it is forced to elongate to the specified length and it is measured, as a rule, when the elongation is not less than 100 %. When the dumbbell shaped test piece is used for this measurement, read the load by means of a proper method at the time when the gauge length has attained the required length. When the ring shaped test piece is used, read the load at the time when the distance between clamps has attained the required length.

3.4.4 Calculation Tensile strength, elongation and tensile stress shall be calculated by formulas given hereunder:

(1) In the case of tensile strength

$$T_B = \frac{F_B}{A}$$

where, T_B : tensile strength (MPa {kgf/cm²})

F_B : maximum load (N {kgf})

A : sectional area of the test piece (mm² {cm²})

(2) In the case of elongation

$$E_B = \frac{L_1 - L_0}{L_0} \times 100$$

where, E_B : elongation (%)
 L_0 : gauge length (mm)
 L_1 : length between bench marks at the time of breaking (mm)

(3) In the case of tensile stress

$$M_n = \frac{F_n}{A}$$

where, M_n ⁽¹⁾ : tensile stress (MPa {kgf/cm²})
 F_n : load at the time of specified elongation (N {kgf})
 A : sectional area of the test piece (mm² {cm²})

Note ⁽¹⁾ n of M_n shows the numerical value of the specified elongation (%).
 For example, M_{300} shows the tensile stress at the time of 300 % elongation.

3.5 Arrangement of test results

3.5.1 Number of test pieces The number of the test pieces shall be four. When four pieces can not be prepared, it shall be three or two pieces. In this case the number of the test pieces should be recorded.

3.5.2 Test results

(1) Tensile strength and elongation Measured values of the tensile strength and elongation shall be arranged in the order of decreasing numerical value and denote respectively as $S_1 \geq S_2 \geq S_3 \geq S_4$ and express with those calculated by the following formulas:

(a) In the case of four test pieces,

$$T_B \text{ or } E_B = 0.5S_1 + 0.3S_2 + 0.1(S_3 + S_4)$$

(b) In the case of three test pieces,

$$T_B \text{ or } E_B = 0.7S_1 + 0.2S_2 + 0.1S_3$$

(c) In the case of two test pieces,

$$T_B \text{ or } E_B = 0.9S_1 + 0.1S_2$$

(2) Tensile stress Tensile stress shall be denoted by the average of the measured values.

3.6 Record Following items shall be recorded in the results of the tests.

- (1) Tensile strength (MPa {kgf/cm²}), elongation (%), tensile stress (MPa {kgf/cm²})
- (2) Capacity of the tester
- (3) Shape and pattern of the test piece

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- (4) Temperature of test
- (5) Other necessary items

4. Tension set test

4.1 Purpose Vulcanized rubber is drawn to a certain length and after keeping it in the elongated state for a certain time, let it contract rapidly and allow it to stand for a certain time. This test is performed for the measurement of the residual elongation of the vulcanized rubber after a certain time.

4.2 Test piece As a rule, the test pieces of dumbbell-shaped pattern No. 1 sampled and prepared in the same manner as described under 3.2 shall be used.

4.3 Testing apparatus

4.3.1 Tester Apparatus capable of drawing the test piece at a uniform rate and keeping at a certain length for a fixed time shall be used.

The tension tester (refer to 3.3) may be used.

4.3.2 Tension speed As a rule, rate of tension shall be uniform such that the required length can be attained in about 15 s.

4.4 Testing method

4.4.1 Portion to be measured Mark bench mark (refer to 3.2.6) for the measurement of elongation on the test piece and stretch this portion to the required length.

4.4.2 Measurement As a rule, draw the test piece to a length corresponding to about $\frac{1}{2}$ of the elongation (%) and after keeping for 10 min, allow it to contract rapidly while taking care not to allow it to bounce. After 10 min, perform the measurement.

Remarks: Length corresponding to about $\frac{1}{2}$ of the elongation (%) shall be obtained by the following formula:

$$L_X = L_0 + \frac{1}{2}(L_1 - L_0)$$

where, L_X : length corresponding to $\frac{1}{2}$ of the elongation (%)
(refer to 3.4.2) (mm)

L_1 : length between the bench marks at the time of
breaking (mm)

L_0 : gauge length (mm)

4.4.3 Calculation Tension set shall be calculated by the following formula:

$$PS = \frac{l_1 - L_0}{L_0} \times 100$$

where, PS : tension set (%)
 L_0 : gauge length (mm)
 l_1 : length between the bench marks after standing
specified time after contraction (mm)

4.5 Arrangement of test results Test results shall, as a rule, be denoted as the average of tension sets of two test pieces.

4.6 Record Following items shall be recorded in the test results.

- (1) Tension set (%)
- (2) Shape and pattern of the test piece
- (3) Temperature of test
- (4) Other necessary items

5. Hardness test

5.1 Purpose This test shall be performed for the measurement of hardness of the vulcanized rubber. Hardness tests shall, as a rule, be performed by one of the following methods:

- (1) Spring type hardness test (type A and type C)
- (2) Constant load type (Olsen) hardness test
- (3) Constant load type (Pusey-Johnes) hardness test

5.2 Spring type hardness test (type A and type C)

5.2.1 Test piece A test piece shall, as a rule, be not less than 12 mm thick in type A. In case that the thickness is below 12 mm, laminate, if possible, to obtain the thickness of not less than 12 mm. In the case of type C, a test piece not less than 6 mm thick shall be used and in case that the thickness is below 6 mm, laminate, if possible, to obtain the thickness of not less than 6 mm.

When the surface of the test pieces to be measured ⁽²⁾ is not flat, grind so as to make it flat.

Note ⁽²⁾ Surface to be measured shall be large enough to let the loading disc of the tester come within the measuring boundary.

5.2.2 Tester Type A or type C of the spring type hardness tester shown in Fig. 3 shall be used as the tester.

Fig. 3

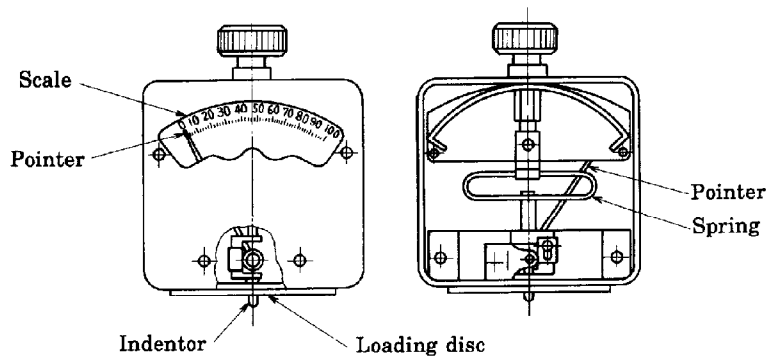
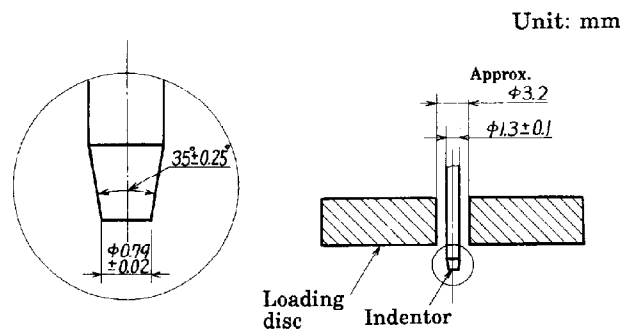


Fig. 4



When the loading disc ⁽³⁾ of this test comes into contact with the surface of the test piece indenter ⁽⁵⁾ projecting from a hole in the center of the loading disc by means of a spring force ⁽⁴⁾ is pushed back by the surface of rubber and this distance is indicated by a pointer on the scale ⁽⁶⁾ as the hardness.

The tester shall be inspected at least every three months.

Notes ⁽³⁾ The loading disc is a plane at right angles to the indenter and has a hole in its centre to pass through the indenter as shown in Fig. 4. Diameter of the hole shall be not less than 10 mm.

⁽⁴⁾ Tolerance on the reference line (refer to Fig. 5) showing the relation among the scale, movement of the indenter and force of the spring shall be $\pm 0.0785 \text{ N}$ [$\pm 8 \text{ gf}$] in case of type A, and $\pm 0.196 \text{ N}$ [$\pm 20 \text{ gf}$] in case of type C.

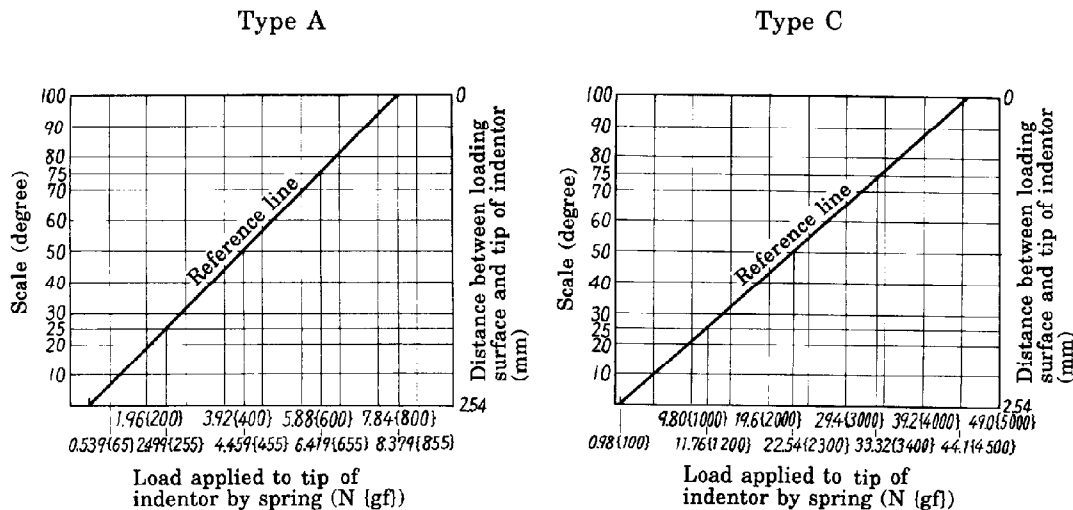
Further, there should be no play between the movement of indenter and that of pointer.

⁽⁵⁾ Material of indenter shall be highly resistant to wear and also highly resistant to rust and shape and dimensions of indenter shall be as shown in Fig. 4.

The indenter shall be fitted correctly at the center of the hole of the loading surface and when the pointer indicates zero on the scale the tip of the indenter shall be projected from the loading surface by 2.54 ± 0.05 mm and when the pointer indicates 100 on the scale the tip of the indenter shall be on the same level with the loading surface.

(6) The scale shall be divided evenly from 0 to 100.

Fig. 5



5.2.3 Testing method Keep the tester vertically and let the loading surface contact with the test piece so as to make the indenter vertical to the surface of the test piece to be measured. Then immediately read the scale and obtain the hardness of the test piece. In case that the reading is made after a certain hour elapsed from making contact with the loading surface, the tester shall be kept vertically and it is desirable to use a proper auxiliary apparatus which is constructed so as to make the indenter perpendicular to the surface to be measured before the test.

In this case vertically press type A tester with a load of 9.807 N {1000 gf} and type C tester with a load of 49.03 N {5000 gf} and read the scale.

Remarks: Type C hardness tester is suitable for the sample which shows the hardness of about not less than 70 by type A.

Moreover, it is desirable to use type C tester for the sample, measured values of which is 30 or more to and excluding 90.

5.3 Constant load type (Olsen) hardness tester

5.3.1 Test piece Shape and dimensions of the test piece should, as a rule, be 12 mm or more in thickness and a disc shape of 50 mm in diameter having flat surface and uniform thickness.

5.3.2 Testing apparatus Olsen type hardness tester shown in Fig. 6 shall be used. This tester consists of a smooth and horizontal test piece holder, a loading surface ⁽⁷⁾ to press the surface of the test piece, an indenter ⁽⁸⁾ which gives a dent on the test piece, a loading apparatus ⁽⁹⁾ capable of applying load completely without giving impact to the indenter and a dial gauge ⁽¹⁰⁾ for the measurement of the depth of depression.

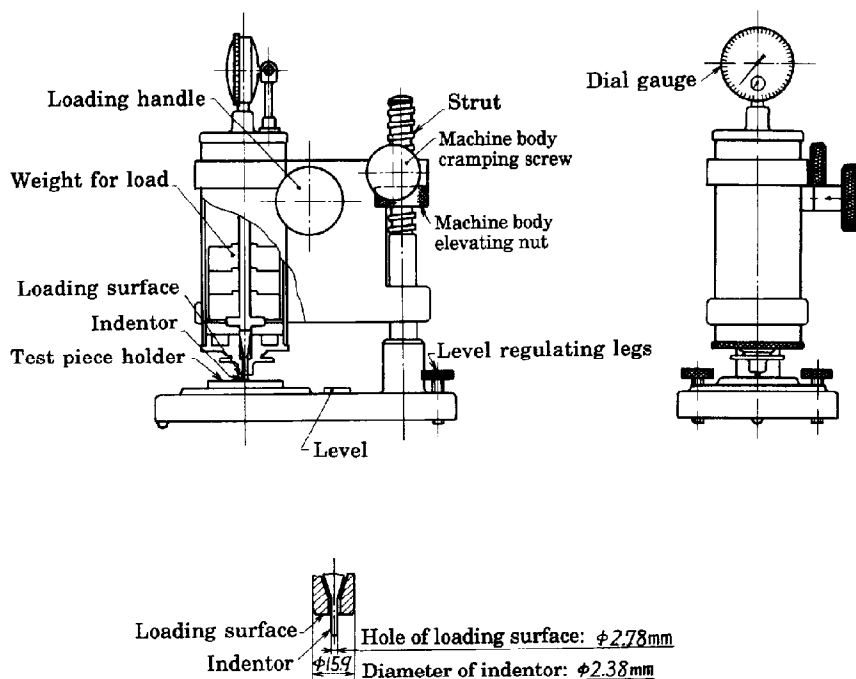
Notes ⁽⁷⁾ Loading surface shall be the load of 22.24 ± 0.21 N {2268 $\pm$ 21 gf}

⁽⁸⁾ Indenter shall be that having a tip of hemispherical form 2.38 ± 0.02 mm in diameter.

⁽⁹⁾ Loading apparatus shall be that capable of applying a load of 13.35 ± 0.07 N {1361 $\pm$ 7 gf}.

⁽¹⁰⁾ Dial gauge shall be that capable of showing the movement of indenter in the vertical direction by 0.254 mm as 10 degrees of Olsen hardness. Precision shall be within ± 0.013 mm over the whole range.

Fig. 6



5.3.3 Testing method The body of the tester shall be placed horizontally and the zero point of the dial gauge shall be corrected.

Measurement shall be performed at a spot at least 12 mm apart from each end of the test piece and the depth of depression shall be measured by the distance between the loading surface and the vertex of hemispherical part of the indenter. Then the loading surface shall be lowered until it comes in perfect contact with the surface of the test piece and the load of 13.35 N {1361 gf} shall be applied without giving any impact to the indenter. Just after 30 s scale shall be read and the hardness of the test piece shall be obtained.

5.4 Constant load (Pusey-Johnes type) hardness test

5.4.1 Test piece Shape and dimensions of the test piece shall, as a rule, be rectangular shape of 12 mm thick, 30 mm wide and 70 mm long with smooth surface and uniform thickness.

5.4.2 Tester Pusey-Johnes type hardness tester shown in Fig. 7 shall be used as a tester.

This tester consists of an indenter ⁽¹¹⁾ which gives a depression to the test piece, a weight ⁽¹²⁾ which can be independently raised or lowered in the vertical direction and a dial gauge ⁽¹³⁾ which shows the depth of depression and these are fixed on the holder.

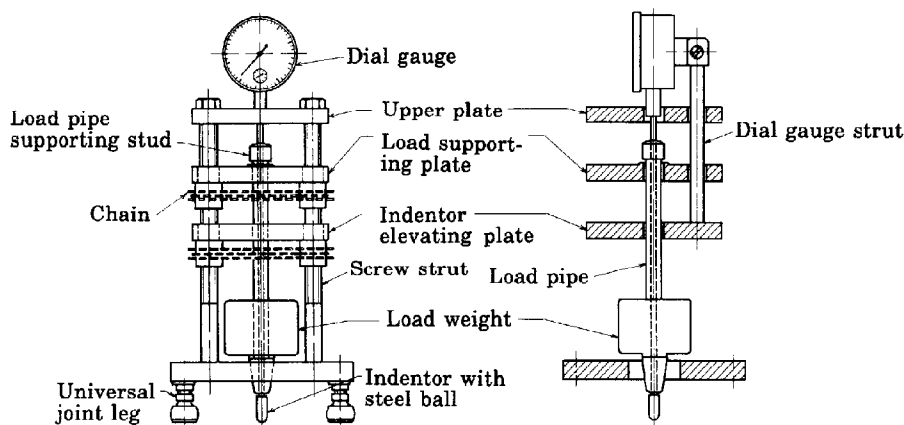
Notes ⁽¹¹⁾ Diameter of the steel ball of the indenter with a small-sized steel ball shall be $\frac{1}{8}$ of that specified in JIS B 1501.

Diameter of the indenter with large-sized steel ball shall be $\frac{1}{4}$ of that specified in JIS B 1501.

⁽¹²⁾ Weight shall be 1000 ± 1 g.

⁽¹³⁾ The smallest value of the scale of dial gauge shall be 0.01 mm.

Fig. 7



5.4.3 Testing method

- (1) **Measurement of the depth of depression** Place the test piece on a smooth and firm stand so as to make the surface of the test piece horizontal. In case of a test piece with the curved surface like a rubber roll, place it so that the central line of the axis of indenter becomes vertical to the surface at a contact point of indenter. Measurement shall be performed at a spot at least 12 mm apart from each end of the test piece.

Next, allow the apparatus to stand on the vertical position and lower the indenter until its tip comes in contact with the test piece and a pointer of the dial gauge turns round three times. Then adjust the dial gauge to read zero at this position of the indenter.

Lower the load while taking care not to give the vibration and impact and make a gap between the load supporting plate and the load pipe supporting stud not less than 5 mm so that the load shall be fully weighted on the indenter without trouble.

Employ an indenter with a small sized steel ball and read the scale after 1 min elapsed from the application of load. Then obtain the depth of the depression.

In case of a very mild test piece which shows the reading of not less than 200 by the indenter with a small sized steel ball, employ an indenter with a large sized steel ball, then read the scale after 15 ± 1 s elapsed from the application of load and obtain the depth of depression.

- (2) Expression of hardness Number expressing the depression by the unit of 0.01 mm shall be called Pusey-Johnes hardness. Kind of the steel ball employed shall be recorded in the results of the test.

5.5 Number of measurements Measurement shall be performed at five spots of new contact points of the sample every measurement.

5.6 Arrangement of results of test Results of the test shall, as a rule, be expressed by affixing the following testing methods or their marks to the average of the measured values at 5 spots.

- (1) Spring hardness, Hs (JIS A or C)
- (2) Olsen hardness, Ho
- (3) Pusey-Johnes hardness, Hp

5.7 Record The following items shall be recorded in the results of the test.

- (1) Hardness
- (2) Kind of hardness tester
- (3) Temperature of test
- (4) Other necessary matters

6. Accelerated ageing test

6.1 Purpose This test shall be performed for the measurement of the ageing of the vulcanized rubber caused by heating. Ageing test shall, as a rule, be performed by measuring the properties such as tensile strength, elongation, tensile stress, hardness and others after heating by anyone of the following methods and finding the change in the values obtained as compared with the values before heating.

- (1) Air oven ageing test
- (2) Oxygen pressure heat ageing test
- (3) Air pressure heat ageing test
- (4) Test tube heat ageing test

6.2 Test piece Test piece shall, as a rule, be prepared according to that described under 3.2 and the thickness and width shall be measured before the ageing test. Then the bench mark for the measurement of elongation shall be marked.

6.3 Air oven ageing test

6.3.1 Testing apparatus Gear system ageing tester or an apparatus corresponding to it shall be employed as the tester.

Tester is so constructed as to ventilate inside air at a rate of at least once in an hour and the tolerance on the temperature of each part of the chamber shall be $\pm 2^\circ\text{C}$ against the temperature of the central part.

Also, an automatic thermoregulator capable of adjusting the temperature in the chamber in the range of $\pm 1^\circ\text{C}$ shall be equipped.

6.3.2 Conditions of test

- (1) **Temperature of test** Temperature of the test shall, as a rule, be $70 \pm 1^\circ\text{C}$ at the central part of the chamber and when the high temperature is especially required, it shall be $100 \pm 1^\circ\text{C}$ or $120 \pm 1^\circ\text{C}$.
- (2) **Time of test** Time of the test shall, as a rule, be any one of 24 h, 48 h, 96 h, 168 h and 336 h.
- (3) **Mass of test piece** Mass of the test piece should not exceed 1 g per 10 ml of the capacity of the chamber of the tester.

6.3.3 Testing method

- (1) **Testing operation** Test piece shall be suspended in the chamber of the tester and heat to cause the ageing. At this time, the suspended test pieces shall not contact with one another or with any part of the wall of chamber of the tester.

The test pieces of different kind of rubber which are apt to interact shall not be put at the same time.

- (2) **Measuring method** Test piece shall be taken out from the chamber of the tester after subjecting to the ageing for specified hour and shall be allowed to stand as it is at room temperature. Within from 16 h to 96 h, the measurement for tensile strength, elongation, tensile stress, hardness and others shall be performed.

6.4 Oxygen pressure heat ageing test

6.4.1 Testing apparatus Bealer Davis system ageing tester or an apparatus corresponding to it shall be employed. This tester consists of an oxygen pressure container in which the test pieces are put and a chamber for the heating the oxygen pressure container. The oxygen pressure container is a cylindrical metal container which is constructed so as to withstand thoroughly the oxygen pressure while keeping the enough internal pressure and this container is steeped in the chamber filled with the heating medium to maintain it at the specified

temperature.

Oxygen pressure container shall be fitted with a safety valve or bursting diaphragm which works surely at a pressure of 3.43 MPa {35 kgf/cm²}. Heating source shall be placed outside of the oxygen pressure container and heating medium to be employed should be water or the liquids which are regarded as stable against oxygen.

Heating medium shall be stirred and circulated to keep the temperature uniform and the temperature shall be adjusted with an automatic thermoregulator.

6.4.2 Conditions of test

- (1) Temperature of test Temperature of the test shall, as a rule, be $70 \pm 1^\circ\text{C}$ or $80 \pm 1^\circ\text{C}$.

If the oxygen pressure container is fully steeped in the heating medium, the temperature of the heating medium may be regarded as that in the oxygen pressure container.

- (2) Oxygen pressure Oxygen pressure in the oxygen pressure container at the time of the test shall, as a rule, be 2.06 ± 0.07 MPa {21.0 $\pm$ 0.7 kgf/cm²}.
- (3) Time of test Time required for the test shall, as a rule, be any one of 24 h, 48 h, 72 h and 96 h.
- (4) Mass of test piece Mass of the test piece shall not exceed 1 g per 10 ml of the inside capacity of the oxygen pressure container.

6.4.3 Testing method After the oxygen pressure container has been previously heated to the specified temperature, the test pieces are inserted in it.

When the test pieces are suspended in the oxygen pressure container, they should not come in contact with one another or with the inside wall of the container.

The test pieces of different kind of rubber which are apt to interact shall not be inserted at the same time.

After insertion of the test pieces, pass oxygen sufficiently through the oxygen pressure container to expel the air in the container and raise the pressure to the specified value. When the specified pressure has been attained, it is regarded as the time of commencement of ageing test.

6.5 Air pressure heat ageing test

6.5.1 Testing apparatus Tester consists of an air pressure container and a chamber for heating it. The air pressure container shall be made of metal so as to withstand the applied pressure and fully maintain the inside pressure, and the heating chamber shall be capable of heating the air pressure container uniformly with air or liquid.

Bealer Davis system rubber ageing tester or autoclave may be used as the tester by putting it in a proper heating chamber. It is preferable that the temperature and pressure can be automatically regulated.

6.5.2 Conditions of test

- (1) Temperature of test Temperature of the test shall, as a rule, be $127 \pm 1^\circ\text{C}$. Temperature of the test shall be measured according to the method described under 6.4.2 (1).
- (2) Pressure of air Pressure in the air pressure container at the time of test shall, as a rule, be $0.55 \pm 0.02 \text{ MPa}$ ($5.6 \pm 0.2 \text{ kgf/cm}^2$).
- (3) Time of test Time required for the test shall, as a rule, be any one of 4 h, 8 h, 16 h, 24 h, 32 h and 48 h.
- (4) Mass of test piece Mass of the test piece shall not exceed 1 g per 100 ml of the capacity of the air pressure container of the tester.

6.5.3 Testing method Test pieces shall be vertically suspended in the air pressure container and they should not contact with one another or with the inside wall of the container.

When the temperature in the air pressure container has been raised to the specified temperature in a short time while keeping the specified air pressure, it is regarded as the time of commencement of the ageing test. When the specified time of the test has passed, the air pressure is gradually lowered to the atmospheric pressure in about 5 min. Then the test pieces shall be taken out and cooled to room temperature on the flat plate.

6.6 Test tube heat ageing test

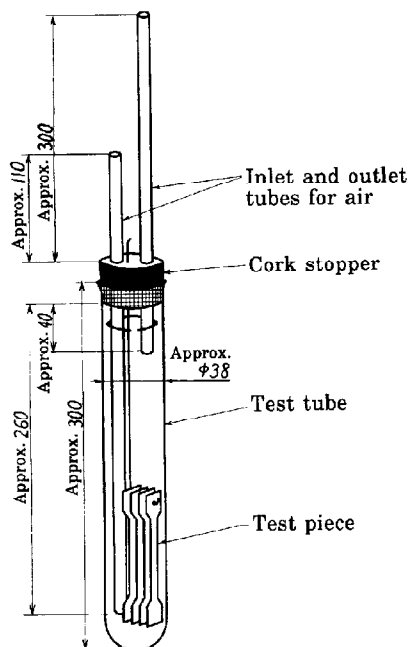
6.6.1 Testing apparatus Testing apparatus consists of a glass test tube ⁽¹⁴⁾ and a heating equipment ⁽¹⁵⁾.

Notes ⁽¹⁴⁾ Test tube Test tube shall be a glass tube about 38 mm in external diameter and about 300 mm in length and fitted with a cork stopper carrying an inlet tube and an outlet tube for air as shown in Fig. 8.

⁽¹⁵⁾ Heating equipment Heating equipment has an oil bath or a metal block as the heating medium and it shall be that which is constructed so as to maintain the tolerance on the temperature in the test tube at $\pm 1^\circ\text{C}$. In the case of the apparatus with the oil bath, care shall be taken not to let oil vapour enter the test tube from the inlet tube for air when heated.

Fig. 8

Unit: mm



6.6.2 Conditions of test

- (1) **Temperature of test** Temperature shall be measured by inserting a thermometer in an optional test tube. Temperature of the test shall, as a rule, be $120 \pm 1^\circ\text{C}$ and it shall be up to $150 \pm 1^\circ\text{C}$ according to the demand.
- (2) **Time of test** Time required for the test shall, as a rule, be any one of 8 h, 16 h, 24 h, 48 h, 72 h and 96 h.

6.6.3 Testing method

- (1) **Number of test pieces** Test pieces which are put in one test tube shall be the maximum 4 pieces and the test pieces which are put in the same test tube should be of the same blending.
- (2) **Testing operation** After adjusting the heating equipment to the specified temperature, put the test pieces, then insert the test tube fitted with the inlet and outlet tubes for air and heat for the specified time while taking care to keep the upper end of the test tube within 50 mm from the heating equipment.

Test pieces shall be suspended vertically as near as possible to the bottom of the tube so that they shall not come in contact with one another or with the sidewall of the tube. Test tube and inlet and outlet tubes for air shall be cleaned before test.

6.7 Calculation

- (1) The variation rate for the tensile strength, elongation and tensile stress shall be calculated by the following formulas:

- (a) In the case of four test pieces

$$A_R(T_B) = \frac{1}{4} \left(\frac{S_{21}}{S_{11}} + \frac{S_{22}}{S_{12}} + \frac{S_{23}}{S_{13}} + \frac{S_{24}}{S_{14}} \right) \times 100$$

$$A_C(T_B) = A_R(T_B) - 100$$

- (b) In the case of three test pieces

$$A_R(T_B) = \frac{1}{3} \left(\frac{S_{21}}{S_{11}} + \frac{S_{22}}{S_{12}} + \frac{S_{23}}{S_{13}} \right) \times 100$$

$$A_C(T_B) = A_R(T_B) - 100$$

- (c) In the case of two test pieces

$$A_R(T_B) = \frac{1}{2} \left(\frac{S_{21}}{S_{11}} + \frac{S_{22}}{S_{12}} \right) \times 100$$

$$A_C(T_B) = A_R(T_B) - 100$$

where, $A_R(T_B)$: respective residual rate (%) for tensile strength, elongation and tensile stress of the sample after heating against those of the sample before heating.

$A_C(T_B)$: respective change rate (%) for tensile strength, elongation and tensile stress of the sample after heating against those of the sample before heating.

$S_{11} \cdot S_{12} \cdot S_{13} \cdot S_{14}$: each measured value for tensile strength (MPa {kgf/cm²}), elongation (%) and tensile stress (MPa {kgf/cm²}) of the test pieces before heating which is arranged in the order of measurement.

$S_{21} \cdot S_{22} \cdot S_{23} \cdot S_{24}$: each measured value for tensile strength (MPa {kgf/cm²}), elongation (%) and tensile stress (MPa {kgf/cm²}) of the test pieces after heating which is arranged in the order of measurement.

- (2) Change in hardness shall be calculated by the following formula:

$$A_H = H_2 - H_1$$

where, A_H : change in hardness
 H_1 : hardness before heating
 H_2 : hardness after heating

6.8 Arrangement of the results of test Results of the test shall, as a rule, be obtained from the measured values of four test pieces. However, when 4 test pieces can not be taken, 3 or 2 test pieces shall be taken. In this case, the number of test pieces should be recorded.

6.9 Record Following items shall be recorded in the results of the test.

- (1) Change rate for tensile strength, elongation and tensile stress, and change in hardness
- (2) Kind of ageing tester
- (3) Temperature and time of ageing test
- (4) Other necessary matters

7. Peeling test

7.1 Purpose This test is performed for the measurement of the strength of adhesion by peeling off between the cloth layers or between the rubber layer and cloth layer, bonded closely with rubber at a constant rate.

This method shall be applicable to the layer whose surface is akin to the cylindrical surface such as tire case, belt, insertion sheet, hose and others and when the surface of layer to be tested is sharply curved, angled or irregularly shaped, other special method should be employed respectively.

7.2 Test piece

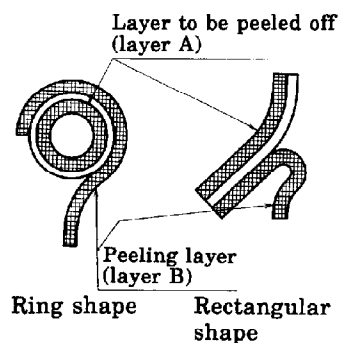
7.2.1 Shape and dimensions of test piece Shape of the test piece shall be rectangular (belt, tire case, sheet and others) and a ring shape (hoses).

Dimensions of the rectangular shape shall, as a rule, be 25.0 ± 0.5 mm in width and 100 mm or over in length.

Dimensions of the ring shape shall be 25.0 ± 0.5 mm in length and its cut end should be at right angles to the direction of axis.

In both cases, the thickness of the layer to be peeled off (referred to as "layer A". Refer to Fig. 9) shall be within 6 mm.

Fig. 9



7.2.2 Sampling and preparation of test piece

- (1) Rectangular shaped test piece Rectangular shaped test piece shall be cut into 25.0 ± 0.5 mm width for all parts and its section should be cut precisely. Section should be parallel to the warp in the lengthwise direction and parallel to the woof in the sideways direction.

If there is the possibility that the section is impaired due to the irregularity of the cord layer, the test piece shall be taken somewhat wider and only the peeling layer (hereafter referred to as "layer B". Refer to Fig. 9) shall be prepared in the width of 25 mm.

- (2) Ring-shaped test piece For the preparation of the ring-shaped test piece, a wooden column and others shall be put in the sample and then the sample shall be cut uniformly. Ring-shaped test piece of which internal diameter is 100 mm or more shall, as a rule, be cut open to prepare the rectangular shaped test piece.
- (3) Thickness of test piece When the thickness of the layer to be peeled off exceeds 6 mm, it should be sliced off with a knife or finished to within 6 mm with a grinding machine. Layer to be peeled off shall not be thinner than the peeling layer.

7.3 Testing apparatus Testing apparatus of the following performance shall be used.

7.3.1 Tester Tester shall be equipped with a clamp which automatically tightens the test piece and an automatic recorder for tensile load and peeling distance. It shall be used in a state where a pointer moves freely with the change in load.

7.3.2 Capacity of tester Tester with a capacity 15 % to 85 % of which corresponds to the tensile load at the time of test shall be used.

7.3.3 Tension speed Rate of movement of the clamp for the test piece is required to be changed to 25.0 ± 1.5 mm per minute or 50.0 ± 2.5 mm per minute.

7.4 Testing method

7.4.1 Attachment of test piece Let one end of the test piece peel off with hand and attach it to the clamp of the tester. At this time, care shall be taken to uniformly apply the force required for peeling off.

In case of the rectangular shaped test piece, it shall be attached so that layers A and B make an angle of about 180° each other.

In case of the ring-shaped test piece, it shall be attached to a column made of wood and others so that the layer B makes an angle of 90° with the tangent to the layer A at a spot where the layer B peels off and the reaction of peeling off makes an angle of 90° to the axis of rotation.

7.4.2 Testing operation Rate of the movement of the clamp for the test piece shall be 50.0 ± 2.5 mm per minute in the case of the rectangular shaped test piece and 25.0 ± 1.5 mm per minute in the case of the ring-shaped test piece and let it peel off to the length which is considered that the adhesion strength is fully measured.