(3) **Test method** After applying compression to the test piece, compression equipment is put into a cryostat within 30 min, the test is carried out for the prescribed time at the test temperature. At least 1 h before completion of the test time, the thickness gauge is put into the cryostat.

(4) **Measurement of thickness after test** After completion of the test time, the compression plate is immediately removed, and the thickness at one position of the central part of the test piece is measured 30 min later. Since those operations are always carried out in the cryostat, care shall be taken so that temperature in the cryostat is kept within a tolerable range.

18.5 Calculation Calculate low temperature compression permanent set according to the following formula:

$$C = \frac{d_0 - d_2}{d_0 - d_1} \times 100$$

where, C : low temperature compression permanent set (%)

d_0 : thickness of test piece before test (mm)

d_1 : thickness of spacer (mm)

d_2 : thickness of test piece after test (mm)

18.6 Arrangement of test results Test results shall be, as a rule, expressed by the mean value of values obtained on three test pieces.

18.7 Record The following items shall be stated on the test results.

(1) Test results

(2) Shape and dimensions of test piece

(3) Method for sampling and preparation of test piece

(4) Test temperature

(5) Test time

(6) Other necessary items

19 Air oven aging test

19.1 Purpose The test is carried out for examining the aging property of elastomer by measuring various properties(24) i.e. tensile strength, elongation at break, tensile stress, hardness, etc., after accelerated aging of elastomer by heating for the specified time at the specified temperature, and obtaining variation of those values from those before heating treatment.

Note (24) Physical properties used for evaluation of aging properties shall be determined according to the practical use. Unless otherwise especially specified, physical properties of tensile strength, elongation at break, tensile stress, hardness, etc., shall be taken as objects for evaluation.

19.2 Tester For the tester, the Geer oven aging tester equipped with an automatic temperature regulator, or an apparatus equivalent in performance thereto shall be used. The size of a test chamber shall be such that the test pieces put therein are separated at least 10 mm from each other, and can be suspended at positions at least 50 mm apart from the wall in the test chamber.

The test chamber should be so constructed that the air in the test chamber is replaced at a rate of 3 turns to 10 turns per hour, the temperature of each part in the chamber is such that the temperature of the test piece put in the chamber can be kept within an aging temperature range specified in 19.4.1 (4), and the average wind velocity in the chamber is preferably (0.5 ± 0.1) m per second.

Remarks: When the tester is inspected, it should be done according to the Annex to JIS K 6257.

19.3 Test piece Test pieces shall be as follows:

19.3.1 Shape and dimensions of test piece The shape and dimensions of test pieces shall be as described in 5.3.1.

19.3.2 Sampling and preparation of test piece The sampling and preparation of test pieces shall be, as a rule, as described in 4.3.

19.3.3 Number of test pieces The number of test pieces shall be at least three.

19.3.4 Measurement of thickness and width of test piece The thickness and width of test pieces shall be measured before aging as described in 5.3.5.

19.3.5 Bench marks for measurement of elongation of test piece When bench marks for measurement of elongation are marked on a dumbbell test piece, they are marked after aging, and their marking shall be as described in 5.3.6.

19.3.6 Selection of test piece Test pieces shall be selected as described in 5.3.7.

19.4 Test method The test method shall be as follows:

19.4.1 Test conditions Test conditions shall be as follows:

(1) The standard conditions of the test room shall be as described in 4.1.

(2) The samples and test pieces shall be storaged as described in 4.2 (1).

(3) The standard conditions of test pieces shall be as described in 4.2 (2).

(4) Accelerated aging temperature shall be, as a rule, selected from among those stated below.

(70 ± 1) °C, (85 ± 1) °C, (100 ± 1) °C, (120 ± 2)°C

(5) Accelerated aging time shall be, as a rule, selected from among those stated below.

$24^{0}_{-0.5}$ h, 48^{0}_{-1} h, 72^{0}_{-2} h, 96^{0}_{-2} h, 168^{0}_{-2} h, and a multiple of seven days

(6) The test temperature at the measurement of tensile strength, elongation at break, tensile stress, and hardness, shall be, as a rule, (23 ± 2) °C.

(7) The volume of the test pieces put in the test chamber shall not exceed 10 % of the inside volume of the test chamber.

19.4.2 Operating method Test pieces shall be subjected to accelerated aging by heating in the test chamber set at the prescribed temperature while being suspended. At that time, the test pieces in the test chamber shall be separated at least 10 mm from each other, and further apart at least 50 mm from the wall of the test chamber.

Different kinds of elastomer test pieces likely to act on each other shall not be simultaneously put in the chamber.

After accelerated aging of the test pieces for the prescribed time, they are taken out from the test chamber, left to stand to cool to room temperature, and tensile strength, elongation at break, and tensile stress are measured according to 5.4, and hardness is measured according to 7.5 after 16 h or longer but within six days.

19.5 Calculation The calculation shall be as follows:

19.5.1 Calculate change rate and remaining factor of tensile strength, elongation at break, and tensile stress according to the following formulas (1) and (2).

$$A_C = \frac{X_1 - X_0}{X_0} \times 100 \quad \text{..... (1)}$$

$$A_R = \frac{X_1}{X_0} \times 100 \quad \text{..... (2)}$$

where, A_C : change rate of tensile strength, elongation at break, and tensile stress after accelerated aging to those before accelerated aging (%)

A_R : remaining factor of tensile strength, elongation at break, and tensile stress after accelerated aging to those before accelerated aging (%)

X_0 : tensile strength, elongation at break, and tensile stress before accelerated aging

X_1 : tensile strength, elongation at break, and tensile stress after accelerated aging

19.5.2 Calculate the change of hardness according to the following formula (3).

$$A_H = H_1 - H_0 \quad \text{..... (3)}$$

where, A_H : change of hardness

H_0 : hardness before accelerated aging

H_1 : hardness after accelerated aging

19.6 Arrangement of test results The test results for tensile strength, elongation at break, and tensile stress are expressed in integral places by rounding off the change rate and remaining factor according to JIS Z 8401 obtained from the calculation formulas in 19.5.1 from the measured values obtained by using at least three test pieces for both before and after accelerated aging.

For hardness, the change of hardness obtained according to the calculation formula of 19.5.2 is expressed in integral places.

19.7 Record The following items shall be stated on the test results.

(1) The change rate and remaining factor of tensile strength, elongation and tensile stress, and the change of hardness

(2) The shape and dimensions of test piece (when laminated, the number of sheets, and its thickness)

(3) Methods for sampling and preparation of test piece

(4) Number of test pieces

(5) Accelerated aging temperature and its time

(6) Other necessary items

20 Resistance to ozone cracking test

20.1 Purpose The test is carried out for measurement of deterioration by ozone of elastomer by exposing the test piece to which static or dynamic tensile strain is applied in the air containing the ozone of low concentration generated artificially.

20.2 Classification of tests The ozone cracking test is classified into the following two types:

(1) Static ozone cracking test(25)

(2) Dynamic ozone cracking test(26)

Notes (25) The static ozone cracking test means that the test piece is exposed to the air containing determined ozone in the chamber kept at a constant temperature under such condition that a static tensile strain is applied thereto.

(26) The dynamic ozone cracking test means that the test piece is exposed to the air containing determined ozone in the chamber kept at a constant temperature under such condition that a dynamic tensile strain is applied thereto. The dynamic ozone cracking test shall be classified into two types of methods A (tensile method) and method B (belt rotation method).

20.3 Static ozone cracking test The static ozone cracking test shall be as follows:

20.3.1 Tester The tester shall be composed of a test chamber, an ozonizer, an ozone concentration controller, and an extension jig.

(1) **Test chamber** The test chamber shall have a volume of at least 0.1 m^3. The inside of the chamber is composed of lining of mateial difficult to decompose ozone and the light from outside is intercepted as far as possible. The chamber shall be so composed that the prescribed relative humidity can be maintained, the temperature can be controlled within ±2 °C, and the air in the test chamber is discharged by about $\frac{3}{4}$ of the volume per minute and circulated.

It is desirable that the chamber is equipped with a window and an illuminating lamp by which the test piece in the chamber can be observed as far as possible.

(2) **Ozonizer**(27) The ozonizer shall be selected from among two types stated below.

(a) **Ozone generating ultraviolet lamp** When an ozone generating ultraviolet lamp is used, the generated amount of ozone shall be able to be controlled by varying the load voltage of the lighting trasformer.

(b) **Silent discharge type** When a silent discharge type is used, the generated amount of zone shall be able to be controlled by varying the feed voltage and flow rate of air to the generator.

Note (27) For the air introduced from outside the chamber, contaminating materials which may influence the concentration of ozone or crack shall be removed by activated carbon or other suitable filter medium.

(3) **Ozone concentration regulator** The ozone concentration regulator is that which controls the ozone concentration manually or automatically, and an automatical controller is preferable.

When test pieces are in the test chamber, the controller shall keep the prescribed concentration of ozone.

Further, when the door of the test chamber is opened and closed for exchanging or inspecting the test piece, the concentration of ozone shall be able to be restored to the prescribed concentration within 30 min.

(4) **Extension jig** The extension jig shall be the jig capable of extending the test piece to a set extension rate.

20.3.2 Measuring method and verification of ozone concentration The method for measurement of ozone concentration shall be manual or automatic.

The verification of ozone concentration is different according to the ozone concentration controller. The concentration is measured according to Annex to JIS K 6259 for a certain period(28), and the verification and confirmation shall be carried out in order to keep the prescribed ozone concentration.

(1) **Manual ozone concentration measuring method** The manual ozone concentration measuring method shall be as follows:

(a) An ozone concentration measuring method using a constant current electrolysis method

(b) An ozone concentration measuring method using a counter current type absorbing device

(2) **Automatic ozone concentration measuring method** The automatic ozone concentration measuring method shall be as follows:

(a) An ozone concentration measuring method by a coulometric method

(b) An ozone concentration measuring method by an ultraviolet absorbing method

Note (28) The verification period differs according to the ozone concentration control method.

Ozone concentration shall be verified and confirmed at least once per week for a manual type and at least once per month for an automatic type.

20.3.3 Test piece Test pieces shall be as follows.

(1) **Shape and dimensions of test piece** The shape and dimensions of test pieces shall be any one of those stated below.

(a) The strip test piece of about 60 mm length, about 10 mm width, and (2 ± 0.2) mm thickness, having smooth surfaces

(b) The strip test piece of about 100 mm length, about 25 mm width, and (2 ± 0.2) mm thickness, having smooth surfaces

(c) The dumbbell No. 1 shape test piece

Remarks : In general, it is desirable to use the above (a) as the test piece.

(2) **Sampling and preparation of test piece** The sampling and preparation of test pieces shall be, as a rule, as described in 4.3.

(3) **Number of test pieces** The number of test pieces shall be, as a rule, three.

(4) **Measurement of thickness and width of test piece** The thickness and width of test pieces shall be measured as described in 4.4.

(5) **Bench marks for measurement of tensile strain** Bench marks for measurement of tensile strain shall be marked on the test piece according to the following methods.

(a) The gauge length shall be 20 mm for a strip one and 40 mm for a dumbbell one.

(b) The bench marks shall be accurately marked with the central part of the test piece being the center.

20.3.4 Test method The test method shall be as follows:

(1) **Test conditions** The test conditions shall be as follows:

(a) **Ozone concentration** The ozone concentration shall be, as a rule, (50 ± 5) pphm.

However, suitable other ozone concentrations may be adopted according to the ozone resistance and service conditions of the test piece.

Remarks 1 Since fluctuation of atmospheric pressure influences the concentration of ozone even if test pieces are exposed to a constant ozone concentration (pphm), the concentration of ozone may be expressed by its partial pressure, i.e. in the millipascal (mPa) unit in order to carry out the test under the constant ozone partial pressure.

2 Under the condition of standard atmospheric pressure and temperature (101 kPa, 0 °C), the concentration of 1 pphm corresponds to the partial pressure of 1.01 mPa.

(b) **Tensile strain** The tensile strain shall be given in such a way that the bench marks of 20 mm or 40 mm interval are marked on the test piece and the piece is elongated by means of the extension jig in 20.3.1. Though the tensile strain is (20 ± 2) % as the standard, it may be selected from among those stated below subject to agreement between the supplier and purchaser.

(5 ± 1) %, (10 ± 1) %, (30 ± 2) %, (50 ± 2) %

Remarks : Since the vicinities of the fringe part of a test piece and the gripper part of a device giving tensile strain are apt to be influenced especially from ozone, it is recommended to form a film by applying toluene solution of chlorosulfonated polyethylene, ethylene propylene rubber, etc., or to put the test piece between suitable sponge rubber, and mount it to the extension jig.

(c) **Test temperature** The test temperature shall be, as a rule, (40 ± 2) °C. The test may be carried out at a suitable other test temperatures according to the service condition of the product.

(d) **Test time** The test time shall be, as a rule, selected from among those stated below.

2 h, 4 h, 8 h, 24 h, 72 h, 96 h

(2) **Operating method** The operating method shall be as follows:

(a) Adjust the test chamber to the prescribed ozone concentration and test temperature.

(b) Carry out the conditioning of test pieces maintaining the tensile strain given by the extension jig, at the room temperature for 20 h to 96 h, in a closed dark box.

(c) Mount the test pieces in such a manner that they come in contact with ozonized air with the longitudinal direction of test pieces being in parallel with the gas flow and they do not come in contact with each other. The amount of the test pieces put in the test chamber is regulated by the total sum of the surface area of the test pieces and shall be limited to at most 60 % of the horizontal sectional area of the test chamber.

(d) After exposing the test piece continuously for the prescribed time, take it out from the test chamber. Observe and record the condition of cracking based on Table 11 and Fig. 19. It is allowed to take out the test piece from the test chamber at intervals of a certain period and to observe and record the state of cracking.

Remarks 1 When the test piece is observed, neigther touch the observed surface by the hands nor hit it by an article.

2 Take care for the toxicity of ozone during operation. Take an appropriate method so that tester's touching ozone is minimized.

20.3.5 Arrangement of test results In general, the states of cracking of three test pieces shall be observed based on Table 11 and Fig. 19, and the observed state shall be written together with the test time.

20.3.6 Record The following items shall be stated on the test results.

(1) Test results

(2) Type of test

(3) Method for sampling and preparation of test piece

(4) Ozone concentration measuring system and ozone concentration (pphm)

(5) Test temperature (°C)

(6) Tensile strain (%)

(7) Other necessary items

20.4 Dynamic ozone cracking test Method A (tensile method)

20.4.1 Tester The tester shall be composed of a test chamber, an ozonizer, an ozone concentration controller, and a pulling device.

(1) **Test chamber** The test chamber shall be as described in 20.3.1 (1).

(2) **Ozonizer** The ozonizer shall be as described in 20.3.1 (2).

(3) **Ozone concentration controller** The ozone concentration controller shall be as desribed in 20.3.1 (3).

(4) **Pulling device** The pulling device shall be composed of a fixed part to which the gripper holding one end of a test piece is attached, and a dynamic tester cosisting of the same structure which holds the other end of test piece and reciprocates. The motion shall be able to give zero strain when the distance between the grippers is shortest, and highest strain when it is longest. The reciprocating part shall be in parallel to a straight line and the planes of upper and lower grippers are parallel to each other in operation. The reciprocating motion shall be carried out at a constant speed of (30 ± 1.5) cycles per minute.

20.4.2 Measurement and verification of ozone concentration The measuring method and verification of ozone concentration shall be as described in 20.3.2.

20.4.3 Test piece Test pieces shall be as follows:

(1) **Shape and dimensions of test piece** The shape and dimensions of test pieces shall be as described in 20.3.3 (1).

(2) **Sampling and preparation of test piece** The sampling and preparation of test pieces shall be, as a rule, as described in 4.3.

(3) **Number of test pieces** The number of test pieces shall be, as a rule, three.

(4) **Measurement of thickness and width of test piece** The thickness and width of test pieces shall be measured as described in 4.4.

(5) **Bench marks for measurement of tensile strain** Bench marks for measurement of elongation shall be marked on a test piece according to the following methods.

(a) The gauge length shall be 20 mm or 40 mm.

(b) The bench marks shall be accurately marked with the central part of the test piece being the center.

20.4.4 Test method The test method shall be as follows:

(1) **Test conditions** The test conditions shall be as follows:

(a) **Ozone concentration** The ozone concentration shall be as described in 20.3.4 (1) (a).

(b) **Tensile strain** The tensile strain shall be applied in such a way that the bench marks of 20 mm or 40 mm interval are marked on the test piece, and the test is carried out so that the highest value of dynamic tensile strain becomes any one of those stated below. However, the standard shall be (10 ± 1) %.

elongation of (5 ± 1) %, (10 ± 1) %, (15 ± 2) %, (20 ± 2) %, (25 ± 2) %, and (30 ± 2) %

(c) **Test temperature** The test temperature shall be as specified in 20.3.4 (1) (c).

(d) Test speed The test speed shall be (30 ± 1.5) cycles per minute.

(e) Test time The test time shall be as specified in 20.3.4 (1) (d).

(2) **Operating method** The operating methods shall be as follows:

(a) Adjust the test chamber to the prescribed ozone concentration and test temperature.

(b) Expose both faces of each test piece to ozonized air, and hold them so that the lengthwise direction becomes the direction of air flow.

(c) Mount the test peice under zero strain state to the dynamic tester, let the tester to reciprocate, and adjust so that the prescribed tensile strain is applied thereto.

(d) After exposing continuously the test piece for the prescribed time, take it out from the test chamber, and observe and record the state of cracking based on Table 11 and Fig. 19. Or it is allowed to take out the test piece from the test chamber at intervals of a certain period, and to observe and record the state of cracking.

Remarks 1 When the test piece is observed, neither touch the observed surface by the hands, nor hit it by an article.

2 Take care for the toxicity of ozone in operation, and adopt an appropriate method so that tester's touch of ozone is minimized.

20.4.5 Arrangement of test results In general, the states of cracking of three test pieces shall be observed based on Table 11 and Fig. 19, and the observed state shall be written together with the test time.

20.4.6 Record The following items shall be stated on the test results.

(1) Test results

(2) Type of test (record use of tensile method)

(3) Methods for sampling and preparation of test piece

(4) Used ozone concentration measuring system and ozone concentration (pphm)

(5) Test temperature (°C)

(6) Test speed

(7) Tensile strain (%)

(8) Other necessary items

20.5 Dynamic ozone cracking test Method B (belt rotary method)

20.5.1 Tester The tester shall be as follows:

(1) **Test chamber** The test chamber shall be as described in 20.3.1 (1).

(2) **Ozonizer** The ozonizer shall be as described in 20.3.1 (2).

(3) **Ozone concentration controller** The ozone concentration controller shall be as described in 20.3.1 (3).

(4) **Belt-turning gear** The belt-turning gear is composed of the belt and pulley set which are specified as below. The pulley set shall have 63.5 mm diameter and consists of upper and lower ones. The upper pulley rotates the test belt at a speed of (40 ± 2) revolutions per minute. The lower pulley moves vertically. A weight of about 18 kg is suspended so that the test belt is sufficiently tensed to make the test piece and belt adapt to the upper and lower pulleys. The test belt is made of cotton canvas of 1.1 kg/m^2, which has 100 mm width and (2300 ± 25) mm circumferential length.

20.5.2 Measurement and verification of ozone concentration The measurement and verification of ozone concentration shall be as described in 20.3.2.

20.5.3 Test piece Test pieces shall be as follows:

(1) **Shape and dimensions of test piece** The shape and dimensions of test pieces shall be a strip one having a smooth surface of about 100 mm length, about 25 mm width, and (3 ± 0.2) mm thickness.

(2) **Sampling and preparation of test piece** The sampling and preparation of test pieces shall be, as a rule, as described in 4.3.

(3) **Number of test pieces** The number of test pieces shall be, as a rule, three.

20.5.4 Test method The test method shall be as follows:

(1) **Test conditions** The test conditions shall be as follows:

(a) **Ozone concentration** The ozone concentration shall be as described in 20.3.4 (1) (a).

(b) **Test temperature** The test temperature shall be as described in 20.3.4 (1) (c).

(c) **Test speed** The test speed shall be (40 ± 2) revolutions per minute.

(d) **Test time** The test time shall be, as a rule, selected from among those stated below.

1 h, 2 h, 3 h, 4 h, 5 h, 6 h, 24 h, 30 h, and 48 h

(2) **Operating method** The operating method shall be as follows:

(a) Adjust the test chamber to the prescribed ozone concentration and test temperature.