

Automobile - Structural adhesive - Testing methods

1. Scope

This standard specifies testing methods for structural adhesives ⁽¹⁾ used for assembling automobile bodies.

Note ⁽¹⁾: This standard applies to structural adhesives for substrates, mainly press-formed steel or aluminium alloy sheets for automobile roof-rails, pillars, floors etc., using spot-weld bonding.

2. Applicable Standards

This standard contains references to the following standards, which by inclusion, constitute provisions of this standard. Use the latest edition of these standards.

JIS B 7410	Liquid-in-Glass Thermometers for Testing of Petroleum Products
JIS B 7722	Charpy Impact Testing Machines
JIS G 3141	Cold Rolled Carbon Steel Sheets and Strips
JIS H 4000	Aluminium and Aluminium Alloy Sheets and Plates, Strips and Coiled Sheets
JIS K 6859	Testing Method for Creep Rupture of Adhesive Bonds
JIS K 7100	Standard Atmospheres for Conditioning and Testing of Plastics
JIS K 8284	Diammonium Hydrogen Citrate (Reagent)
JIS R 3503	Glass Apparatus for Chemical Analysis
JIS R 3645	Glass Rods
JIS Z 2371	Methods of Salt Spray Testing
JIS Z 8401	Rules for Rounding off of Numerical Values
JASO M 609	Corrosion Test Method for Automotive Materials

3. Definition

The definition of major terms used in this standard shall conform to the following.

- a) Adhesion failure: A failure occurring on a bonded joint at the boundary between the adhesive and the substrate.
- b) Cohesion failure: A failure occurring on a bonded joint within the adhesive or within the substrate.

c) Shear stress (τ): The shearing force divided by the bonded surface area, expressed in megapascals (MPa).

d) Static shear strength (τ_R): The average static shear stress at failure as defined in Article 16 (Shear strength), expressed in megapascals (MPa).

e) Cyclic stress: Stresses varying between specified values at regular intervals. The stresses appear in a sinusoidal form (refer to Attached Drawing 1) with undulating shear. Cyclic stress is the superposition of the alternating stress on the static stress (mean stress).

1) Maximum stress (τ_{max}): A greatest algebraic value appeared in repeated stress cycles. The value is expressed in megapascals (MPa).

2) Minimum stress (τ_{min}): The smallest algebraic value appearing in repeated stress cycles. The value is always a positive figure and is expressed in megapascals (MPa).

3) Mean stress (τ_m): Half of the algebraic sum of the maximum stress and the minimum stress appearing in repeated stress cycles expressed in megapascals (MPa).

4) Stress amplitude (τ_a): Half of the algebraic difference between the maximum stress and the minimum stress appearing in repeated stress cycles expressed in megapascals (MPa).

5) Stress ratio (R_τ): The algebraic ratio of the minimum stress to the maximum stress.

f) Fatigue limit (τ_D): The upper limit of stress amplitude (τ_a) endured in an infinite number of stress cycles for a specified mean stress (τ_m) or stress ratio (R_τ). Determining the limit of endurance is useful for materials that do not attain full stress amplitude in relation to the number of cycles, and decrease the limit value monotonously along with increases in the number of cycles.

g) Limit of endurance ($\tau_D (N_F)$): The upper limit of shear stress endured in a specified number of cycles (N_F). For tests with a constant stress ratio (R_τ), the result is presented in the form of:

$\tau_D (N_F, R_\tau)$ in megapascals unit (MPa)

For tests with a constant mean stress (τ_m), the result is presented in the form of:

τ_D (N_F , τ_m) in megapascals unit (MPa)

- h) Number of cycles (N): The number of repeated stress cycles required to induce fatigue failure. If no fatigue failure occurs, the life of the material tested shall be assumed to be longer than the tested duration, though the number of cycles cannot be determined.
- i) Ratio of number of cycles (n/N): The ratio of the number of stress cycles (n) with the same stress value, given in a fatigue test to the number of stress cycles (N) inducing failure. The ratio is used as an index for evaluating load-bearing performance along with an SN curve (Woehler's diagram).
- j) SN diagram: A diagram showing the resistance properties of materials. The diagram gives an experimentally-derived relation between the number of stress cycles N and the stress amplitude (τ) or (τ_{max}). Generally, the number of cycles N is given on the horizontal axis (logarithmic scale), and the stress amplitude (τ_a) or (τ_{max}) is given on the vertical axis (normal scale or logarithmic scale). (Refer to Attached Drawings 2 and 3.)

4. Test Items

Test items and major test methods for structural adhesives shall conform to Table 1.

Table 1 Test Items and Major Test Conditions

Test item	Evaluation item	Test condition and test method	Unit	Number of determinations	Rounding of numerical values	Applicable Article
Specific gravity (density)	Specific gravity (density)	Underwater replacement method	-, (g/cm ³)	3 times	Round to two decimal places	6.
Non-volatile matter	Non-volatile matter	(170 ±2)°C, 20 minutes	%	2 times or more	Round to one decimal place	7.
Viscosity	Viscosity	Single cylinder rotational viscometer	mPa·s or Pa·s	2 times or more	Two digits of significant figures	8.
Volume change rate	Volume change rate	A: Aluminium plate method B: Ring method	%	2 times	Round to one decimal place	9.
Water absorption ratio	Water absorption ratio	Immersion at (23 ± 2)°C for 24 hours	%	2 times	Round to one decimal place	10.
Working life	Change in viscosity	Single cylinder rotational viscometer	hour, min	2 times or more	Round to integer	11.
	Change in bonding strength	per discussion between the parties concerned	hour, min	5 samples or more	Round to integer	
Storage stability	Appearance, Coatability	-	-	-	-	12.
	Viscosity change rate	Single cylinder rotational viscometer	%	2 times or more	Two digits of significant figures	
	Lap shear strength change rate	Lap shear strength under 5 mm/min of tensile speed	%	5 samples or more	Two digits of significant figures	
Fluidity	Fluidity	Determined at (170 ± 2)°C for 10 and 20 min, respectively	mm	3 times	Round to integer	13.
Fixation to oily metal surface	Fixation	Dropwise adhesive addition method	hour, sec	3 times	Round to integer	14.
Spot weldability	Reduction rate of weld strength	Failure strength of lap shear joint	%	5 times or more	Three digits of significant figures	15.
Lap shear strength	Lap shear strength	Normal state, High temperature (80 ± 2)°C, Low temperature (-30 ±2)°C	MPa	5 samples or more	Two digits of significant figures	16.
Peel strength	T-peel strength	Normal state, High temperature (80± 2)°C, Low temperature (-30±2)°C	N/25 mm	5 samples or more	Three digits of significant figures	17.

Table 1 Test Items and Major Test Conditions (Continuation)

Test item	Evaluation item	Test condition and test method	Unit	Number of determinations	Rounding of numerical values	Applicable Article
Shear impact strength	Shear impact strength	A: Charpy impact strength	J, (kN)	5 samples or more	Two digits of significant figures	18.
		B: High speed tensile test	kN			
Shear fatigue strength	Shear fatigue strength	Frequency: 30 Hz Stress ratio: 0.1 Number of cycles: 10^7	MPa	3 samples or more	Three digits of significant figures	19.
Heat aging test	Lap shear strength	$(80 \pm 2)^\circ\text{C}$ for 30 days	MPa	each 5 samples or more	Two digits of significant figures	20.
	T-peel strength		N/25 mm		Three digits of significant figures	
Humidity resistance test	Lap shear strength	$(50 \pm 2)^\circ\text{C}$; 95%RH or more; 30 days	MPa	each 5 samples or more	Two digits of significant figures	21.
	T-peel strength		N/25 mm		Three digits of significant figures	
Heat cycle test	Lap shear strength	A: $(-40 \pm 2)^\circ\text{C}$ for 4 hrs. $\rightarrow (80 \pm 2)^\circ\text{C}$ for 4 hrs. B: $(-30 \pm 2)^\circ\text{C}$ for 4 hrs. $\rightarrow (80 \pm 2)^\circ\text{C}$ for 20 hrs. $\rightarrow (-30 \pm 2)^\circ\text{C}$ for 4 hrs. $\rightarrow (50 \pm 2)^\circ\text{C}$, 95%RH or more, 20 hrs.	MPa	each 5 samples or more	Two digits of significant figures	22.
	T-peel strength		N/25 mm		Three digits of significant figures	
Corrosion resistance test	Corrosion depth	Salt spray at $(35 \pm 2)^\circ\text{C}$ for 2 hrs.	mm	each 5 samples or more	Two digits of significant figures	23.
	Lap shear strength	$\rightarrow$ Drying at $(60 \pm 2)^\circ\text{C}$ for 4 hrs.	MPa		Two digits of significant figures	
	T-peel strength	$\rightarrow$ Wetting at $(50 \pm 2)^\circ\text{C}$, 95%RH or more, for 2 hrs.	N/25 mm		Three digits of significant figures	
Creep test	Failure time under tension loading	Loading range: 0.5 to 2.0 kN Temperature range: 50 to 80°C	kN hour	3 samples or more under every condition	Round to integer	24.

5. General Test Conditions

5.1 Standard State of Test

Humidity and temperature at the test site shall conform to, in principle, **JIS K 7100** Article 2.2 (Grading standard temperature states) standard temperature state Grade 2 (23 ± 2)°C and Article 3.2 (Grading standard humidity states) standard humidity state Grade 2 (50 ± 5)%RH. Under special conditions, however, **JIS K 7100** standard temperature state Grade 3 (23 ± 5)°C and standard humidity state Grade 3 (50^{+20}_{-10})%RH may be applied.

5.2 Method for Sampling

Samples shall be collected from specified lots of comparable quality. Samples shall be sealed in a container to prevent evaporation. Before testing, agitate the samples for uniformity.

5.3 Preparation of Samples and Specimens

Samples and specimens shall be maintained as specified in Article 5.1, and adjusted for testing.

5.4 Types and Surface Treatment of Specimens

The types and the surface treatment of specimens shall conform to **Table 2**, or by agreement between the parties concerned when separate specifications are given.

Table 2 Types and Surface Treatment of Specimens

Types of specimens	Material	Surface treatment
Steel sheets	Cold-rolled steel sheets SPCC-SD having a standard thickness ⁽²⁾ of 0.8 mm or 1.6 mm, defined by JIS G 3141.	Before testing, test piece materials shall be degreased with a solvent ⁽³⁾ , dried, immersed in a specified rust-preventing oil or lubricant oil ⁽⁴⁾ , then allowed to stand as described in Article 4.1 for 24 hours to remove oil.
Surface-treated steel sheets	Per discussion between the parties concerned	
Aluminium alloy sheets	Aluminium alloy sheets of 5000 Series or 6000 Series having a standard thickness ⁽²⁾ of 0.8 mm or 1.6 mm, defined by JIS H 4000.	

Notes ⁽²⁾: Sheet thickness shall be determined by the parties concerned.

⁽³⁾: Select appropriate solvent such as; acetone, methylethylketone, 2-butanone, or a mixture of allowable hydrocarbons.

⁽⁴⁾: Lubricant oil shall be determined by discussion the parties concerned.

5.5 Preparation of Test Joints

Specifications given in Article 16.2 (Preparation of specimens) and Article 17.2 (Preparation of specimens).

5.6 Names and Symbols of Failure Modes

The name and symbol of each failure mode shall conform to **Table 3**. A single failure mode shall be indicated solely

by the corresponding symbol. For combined failure modes, percentage (%) of each shall be indicated in accordance with Table 3. (Refer to **Fig. 1**.)

For failures in which the coating layer is torn off from the substrate, enter DF after the coating layer type.

Note: Types of coating layers include plating, primer, paint, phosphatization. When vibration failure occurs under two kinds of modes, enter OSC after the description of the failure mode. (Refer to **Fig.2**)

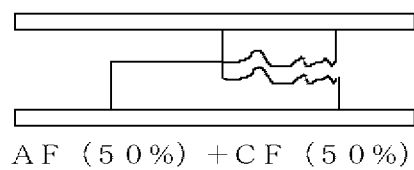


Fig. 1 Example of Mixed Failure

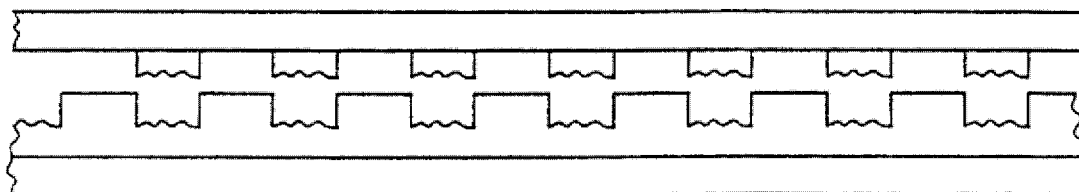
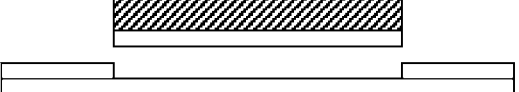
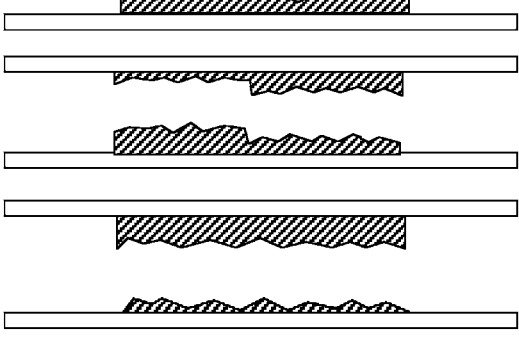
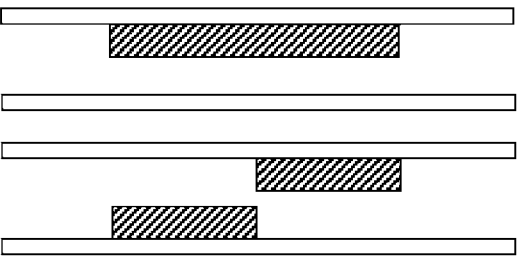
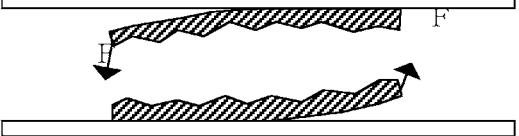


Fig. 2 Example of Oscillation Failure

Table 3 Names and Symbols of Failure Modes

	Name of failure mode	Designation
Substrate	Substrate failure (Failure of one or both adherents) 	SF
	Cohesive substrate failure (Failure of an adherent) 	CSF
	Delamination failure (Failure through delamination) 	DF
Adhesive	Cohesion failure (Failure of adhesive layer) 	CF
	Special cohesion failure (Failure leaving uneven adhesive layer thickness on each side of substrates) 	SCF
	Thin cohesion failure (Failure leaving thin adhesive layer on one side of substrates) 	TCF
	Adhesion failure (Failure occurred from bonded surface) 	AF
	Adhesion and cohesion failure with peel 	ACFP

5.7 Test Condition, Number of Specimens, and Rounding of Numerical Values

Conditions for individual test items and the number of specimens used in a single test shall conform to Table 1. The test result shall be rounded to digits given in Table 1 in accordance with JIS Z 8401.

6. Specific Gravity and Density

6.1 Purpose

The purpose is to determine the specific gravity and density of structural adhesives.

6.2 Apparatus

Use the following test apparatus:

- a) Chemical balance: A weighing capacity of 100 g or more, and a sensitivity of 1 mg.
- b) Hanging string: Sufficient strength to hang a sample, with negligible error in volume change when immersed.
- c) Holder: If a sample cannot be directly hung, a holder shall be used to immerse the sample.
- d) Beaker: To be filled with immersing liquid, shall be large enough to prevent contact between the sample and the beaker wall.
- e) Fixing support: capable of fixing the sample, hanging string, and holder.
- f) Immersing liquid: Fresh or boiled distilled water with 0.1% or less of a wetting agent such as ethanol to eliminate bubbles.

6.3 Procedure

- a) Tie a 0.5~5 g sample to a hanging string, or place in a holder. Weigh the sample with the hanging string or the holder to within 1 mg.
- b) Pour immersing liquid into a beaker at $(23 \pm 2)^\circ\text{C}$. Place the beaker on the fixing support. Avoiding contact with the moving part of the balance.
- c) Immerse the sample in the liquid. Attach the other end of the string to the balance.
- d) Completely remove bubbles from the surface of the sample using a fine hanging string or by other means. Weigh the mass of the sample to within 1 mg.

6.4 Calculation

Compute the specific gravity as follows.

- a) With hanging string only:

$$SG = \frac{a}{a - b}$$

where,

SG: the specific gravity

a: the mass of sample in air (g)

b: the mass of sample in immersing liquid (g)

- b) With holder

$$SG = \frac{a}{a + b' - c}$$

where,

SG: the specific gravity

a: the mass of sample in air (g)

b': the mass of holder with hanging string in the immersing liquid (g)

c: the mass of holder with sample and hanging string in the immersing liquid (g)

6.5 Density

Density shall be computed as follows.

$$D = d \times SG$$

where,

D: the density (g/cm^3)

d: the density of water at a measured temperature (g/cm^3) (0.998 at 23°C)

SG: the specific gravity

6.6 Expression of the Result

Obtain data from three samples, determine the average values and compute to obtain the data required for Table 1.

7. Non-volatile Matter

7.1 Purpose

The purpose is to determine the non-volatile matter in structural adhesives.

7.2 Test Apparatus

Use the following test apparatus.

- a) Weighing container: 50 ml, flat weighing bottle with a ground stopper, as described in JIS R 3503, or an aluminium foil dish formed along the curvature of the inner wall surface of the flask.
- b) Chemical balance: Weighing capacity of 100 g or 200 g, with a sensitivity of 1 mg.

- c) Constant temperature bath: Hot air circulation bath capable of maintaining a temperature of $(170 \pm 2)^\circ\text{C}$.
- d) Desiccator: As described in JIS R 3503, with a bottom diameter of 150 mm or more, using silica gel as the desiccant.

7.3 Procedure

- a) Place 3.0 g of the sample into the weighing container.
- b) Dry the sample in a bath at $(170 \pm 2)^\circ\text{C}$ for 20 minutes, then allow the sample to cool in a desiccator. Weigh the mass of the cooled sample.

7.4 Calculation

Compute the percentage of non-volatile matter N (%) as follows:

$$N = \frac{M_o}{M_s} \times 100$$

where,

M_s : the weight of sample before drying (g)

M_o : the weight of sample after dried (g)

7.5 Expression of the Result

Obtain data from two or more samples, determine the average values and compute to obtain the data required for Table 1.

8. Viscosity

8.1 Purpose

The purpose is to determine the viscosity of structural adhesives.

8.2 Test Apparatus

The test apparatus shall be those listed in the following.

- a) Constant speed rotation, with a spring balance to measure the torque generated by the viscosity resistance. Fig. 1 shows an example of the viscometer.
- b) Container: Approximately 85 mm inner diameter and 120 mm depth, with a lid, or an outer cylinder attached to the viscometer.
- c) Glass rod: As described in JIS R 3645, with a diameter of about 8 mm.
- d) Stopwatch: Accuracy of 0.2 seconds.
- e) Thermometer: The thermometer shall conform to JIS B 7410 for tag-seal low flash point use (TAG-50).

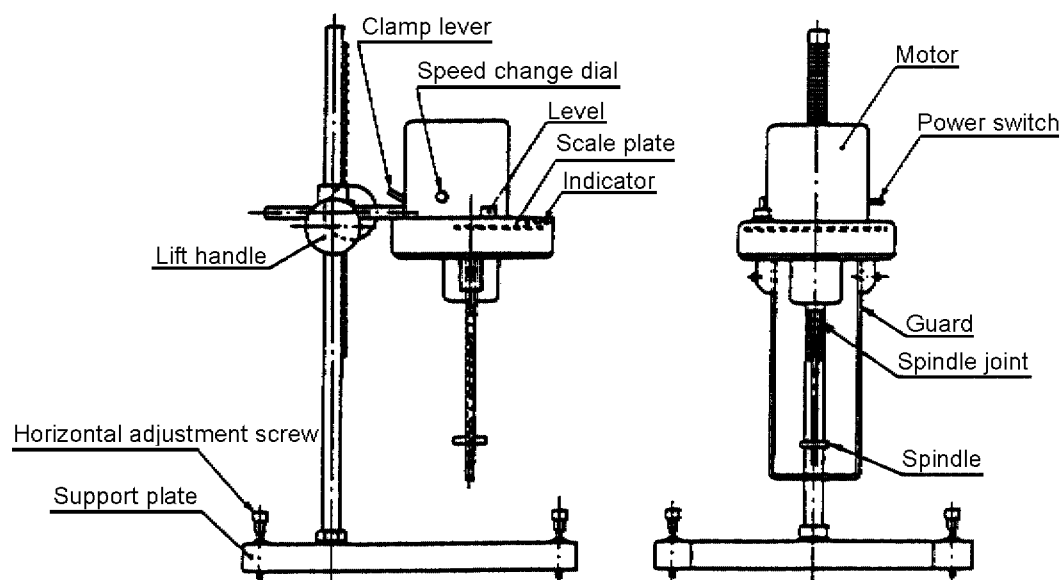


Fig. 3 Example of Viscometer

8.3 Procedure

Place 500 ml of the sample into the container. Slowly agitate with the glass rod for about 1 minute, avoiding bubbles. Adjust to the specified temperature. Dip the rotor of the viscometer in the sample to the immersion mark. Avoid leaving bubbles on the rotor surface. Agitate for about 2 minutes, then begin the measurement. Select the rotor No. and the rotation speed in a range of from 15 to 85% of the full capacity. Rotate for 1 minute(s). Record the data shown on the indicator. Multiply the specified conversion factor by the indicated value to obtain the viscosity. When using a viscometer that shows viscosity value, record the data as indicated.

Note ⁽⁵⁾: For a high viscosity systems, rotate for two minutes.

8.4 Recording Results

Obtain data from two or more tests on the same sample, determined the average values and compute to obtain the data required in Table 1. Record the type of viscometer, the applied rotor No. and rpm, and the test temperature.

9. Volume Change Rate

9.1 Purpose

The purpose is to determine the volume change of structural adhesives after heating to cure.

9.2 Test Method A

9.2.1 Test Apparatus

Use the following test apparatus.

- Aluminium sheet: 50 × 50 mm with an adequate thickness.
- Chemical balance: Sensitivity of 1 mg.
- Constant temperature bath: Hot air circulation type capable of maintaining a temperature level of (170±2)°C.

9.2.2 Procedure

- Place 2 g of the sample onto an aluminium sheet (50 × 50 mm) in the shape of a bead about 10 mm in diameter. Avoid forming bubbles. Weigh the sample with the aluminium sheet. Weigh again in pure water ((23±2)°C) to determine the apparent mass.
- Put the test piece into a constant temperature bath held to (170±2)°C, and heat the test piece for 20 minutes. Allow the test piece to stand as specified in Article 5.1

(Standard state of test) for 60 minutes. Weigh the test piece with the applied sample in the open atmosphere, and the apparent mass of the test piece with the applied sample in pure water ((23±2)°C).

9.2.3 Calculation

Compute the volume change rate V (%) as follows:

$$V = \frac{(m_3 - m_1) - (m_4 - m_2) - (m_5 - m_1) + (m_6 - m_2)}{(m_3 - m_1) - (m_4 - m_2)} \times 100$$

where,

- m_1 : mass of aluminium sheet in air (g)
- m_2 : apparent mass of aluminium sheet in pure water (g)
- m_3 : mass of aluminium sheet plus sample in air (g)
- m_4 : mass of aluminium sheet plus sample in pure water (g)
- m_5 : mass of aluminium sheet plus sample after heating in air (g)
- m_6 : mass of aluminium sheet plus sample after heating in pure water (g)

9.3 Test Method B

9.3.1 Test Apparatus

Using the following test apparatus:

- Ring: Made of an anti-corrosive metal, 2 mm thick, 12 mm high, and about 65 mm inner diameter, with flat-finished top and bottom faces. Provide flush contact with the glass plate. Fig. 4 shows an example of the ring.
- Smoothing spatula: Made of an anti-corrosive metal. Fig. 5 shows an example of the spatula.
- Burette: Capacity of 50 ml or less for use in chemical analysis