

JIS

JAPANESE INDUSTRIAL STANDARD

**Testing Methods for Prepreg,
Carbon Fiber and Epoxy Resins**

JIS K 7071—1988

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In the event of any doubt arising,
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Testing Methods for Prepreg,
Carbon Fibre and Epoxy Resins

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1. Scope

This Japanese Industrial Standard specifies testing methods for carbon fibre-epoxy resin prepreg.

- Remarks 1. The measurement of prepreg properties such as mass per unit area, volatile content, resin flow, gel time and water content are useful to determine the optimum manufacture conditions, to examine the change of quality in storage and to set the optimum conditions for cure.
2. The units and numerical values given in { } in this Standard are based on the traditional unit system and are appended for informative reference.

Applicable Standards:

JIS B 7514-Steel Straightedges

JIS C 1602-Thermocouples

JIS C 6402-Fixed Carbon Film Resistors

JIS K 0068-Testing Methods for Water Content of Chemical Products

JIS K 1107-High Purity Nitrogen

JIS K 6900-Glossary of Terms Used in Plastic Industry

JIS K 7100-Standard Atmospheres for Conditioning and Testing of Plastics

JIS K 8891-Methanol

JIS R 3414-Glass Fabrics

JIS R 3503-Glass Apparatus for Chemical Analysis

JIS Z 8401-Rules for Rounding off of Numerical Values

Reference Standard:

JIS Z 8203-SI Units and the Use of their Multiples and of Certain
other Units

2. Definitions

The main definitions in this Standard are in accordance with those specified in JIS K 6900, and the following definitions are applicable for others.

- (1) volatile content The rate of the volatiles for initial mass when it is exposed to a prescribed cure temperature.
- (2) resin flow The rate at which resin flows out for initial mass when it is cured at a prescribed temperature and pressure. The resin flow varies with the volatile content and resin content of the prepreg, and with the degree of cure of the resin.
- (3) gel time The fact that the epoxy resin increases suddenly their viscosity to attain a gel state during curing is defined as gelation. The time to reach gelation is called gel time at a prescribed cure temperature. The gel time is an index for the degree of cure of the resin.
- (4) water content The content of water vapourized at 150°C.
- (5) prepreg One of the intermediates to fabricate fibre reinforced plastics, having the shape of flat sheet, in which the carbon fibre reinforcement is impregnated with epoxy resin mixture which would also contain some colorant, filler, etc. in an appropriate amount.
- (6) release film A film to make easy the release of the cured products from the molding tool after curing. Film made out of heat resistant polymers, such as copolymer of tetrafluoroethylene and hexafluoropropylene (FEP), polytetrafluoroethylene (PTFE) and polyethyleneterephthalate (PETP) are applicable.
- (7) porous release film A release film with appropriate pores through which the resin can flow out. To manufacture the fibre reinforced plastics products, the porous release film is used for testing the resin flow as well as for the bleed curing of the prepreg.
- (8) bleeder cloth A woven cloth used for absorbing excessive resin which flows out of the prepreg at cure. Usually, glass satin cloth, for example, ES 22 B as specified in JIS R 3414, can be used.
- (9) hot press A device capable of heating and pressing the prepreg specimen of controllable temperatures and pressures.
- (10) bleed cure molding In the case of curing the prepreg to carbon fibre reinforced plastics, hereinafter represented as CFRP, by either vacuum bag molding or pressure bag molding, a part of resin in the prepreg is removed by the absorption of the woven cloth and/or mat at cure in order to control the fibre content and thickness of product of CFRP and to eliminate the voids of the CFRP. This procedure is called "bleed cure molding". The term "bleed" means to squeeze a part of resin out of the prepreg.

3. Test Items

The test items shall be as follows:

- (1) Mass per Unit Area of Prepreg, Mass per Unit Area of Carbon Fibre, Resin Content and Fibre Content.
- (2) Volatile content
- (3) Resin flow
- (4) Gel time
- (5) Water content

4. General Conditions for Test

4.1 Preparation of Sample The carbon fibre-epoxy resin prepreg is usually stored at low temperatures, such as -18°C . Allow the sealed packages of the prepreg to warm up to ambient temperature before opening the seal, then taking the sample to ensure that the prepreg does not absorb moisture from the atmosphere.

4.2 Sampling of Test Specimen Sampling of test specimens shall be as follows:

4.2.1 Three specimens shall be taken for each test.

4.2.2 The specimens shall be drawn in width direction of the prepreg. In the case where the prescribed amount of the specimens can not be taken in width direction, for example in the case of narrow width tape, it is allowed to take the specimens in longitudinal direction. Edges or selvages of the prepreg shall be excluded to take the specimens.

4.3 Standard Atmospheric Conditions of Testing Room Standard atmospheric conditions of the testing room shall be grade 3 : the temperature ($23 \pm 5^{\circ}\text{C}$) and relative humidity of $50 \pm 10\%$.

4.4 Cure Conditions The cure conditions shall be determined with agreement between the user and supplier of the prepreg.

4.5 Expression of Test Results The test results shall be taken to the tenth place of the specified value, and shall be rounded off in accordance with JIS Z 8401.

5. Testing Methods

5.1 Mass per Unit Area of Prepreg, Mass per Unit Area of Carbon Fibre, Resin Content and Fibre Content

5.1.1 Summary of Testing Method Specimens of prepreg are weighed, the resin is then dissolved in a solvent by immersing at ambient temperature or by boiling. Filtrate them, wash the remaining fibre with solvent and reweigh after drying. The mass per unit area of prepreg and carbon fibre, resin content and fibre content are obtained by calculation.

5.1.2 Apparatus

- (1) Cutting Template Metals or ceramics, 100 ± 1 mm by 100 ± 1 mm and about 5 mm thick. Hard metals, such as steel, are recommended.
- (2) Cutting Knife Single edge.
- (3) Analytical Balance Capable of measurement to 0.001 g.
- (4) Watch Capable of measuring 60 minutes or more.
- (5) Draft Chamber Suitable to operate with the filtrating solvent.
- (6) Glass Beaker 300 ml in volume.
- (7) Erlenmeyer Flask 500 ml in volume with ground joint.
- (8) Condenser Tube Fitting the Erlenmeyer Flask with Ground Joint 300 mm to 400 mm in length.
- (9) Desiccator Specified in JIS R 3505, containing a desiccating agent, such as silica gel, calcium chloride, phosphorous pentoxide, or concentrated sulfuric acid.
- (10) Water Bath Capable of heating up to 95°C and maintaining constant temperature.
- (11) Glass Filter Crucible Type 2G2 specified in JIS R 3503.
- (12) Suction Bottle For filtration specified in JIS R 3503.
- (13) Vacuum Source Capable of reducing the pressure to about 13 kPa {100 mmHg}, for example a glass aspirator to connect with water works.
- (14) Graduated Cylinder 500 ml in volume.
- (15) Circulating Air Oven Capable of raising the temperatures up to 200°C , hereinafter referred to as "oven".

5.1.3 Reagents

- (1) Methylethyleketone Industrial grade, hereinafter referred to as MEK.
- (2) Tetrahydrofuran Industrial grade, hereinafter referred to as THF.

Remark: Either reagents are able to cause injury in health and are highly inflammable, it is therefore, necessary to ventilate properly, to install fireproof construction and to adopt prevention of spraying on skin.

5.1.4 Test Specimen The specimen size shall be 100 mm by 100 mm by the thickness of the sample.

5.1.5 Procedure

- (1) Weigh a test specimen to the nearest 0.001 g and record the mass as W_1 .
- (2) Dissolution of Resin Resin shall be dissolved by the following method A or method B.

(2.1) Method A

- (a) Put the specimen into a beaker, pour about 200 ml of MEK or THF and place the beaker at ambient temperature for 20 minutes by stirring the content several times.
- (b) Filtrate supernatant liquid with a glass filter (mass W_2).
- (c) Put about 200 ml of MEK or THF into the beaker in which carbon fibre remains, and repeat the procedure of (a) and (b) by three times. Then filtrate the content with the glass filter by the aid of suction.

(2.2) Method B

- (a) Put a test specimen into an Erlenmeyer flask, pour about 300 ml of MEK or THF, and attach a condenser for reflux. Place the flask into a water bath at 85 to 90°C for MEK or at 75 to 80°C for THF, and keep boiling for 40 minutes.
- (b) Remove the flask from the water bath, then leave it cool for about 5 minutes. The following procedure should be carried out in a draft chamber. Filtrate the content using a glass filter (mass W_2), and wash the carbon fibre collected on the glass filter with about 300 ml of MEK or THF at ambient temperature with aid of suction.
- (3) After filtration expose the carbon fibre with the filter at 105^{+10}_0 °C for about 90 minutes in an oven, then cool in a desiccator for 45 minute or longer.
- (4) Weigh the carbon fibre with the glass filter to the nearest 0.001 g and record the mass as W_3 .
- (5) Repeat the procedure of (1) to (4) for each of the three specimens.

5.1.6 Calculation

- (1) Calculate the mass per unit area of prepreg, mass per unit area of carbon fibre, resin content and fibre content according to the following equations (1) to (4).

$$W_F = W_1 \times 100 \dots\dots\dots (1)$$

$$W_o = (W_3 - W_2) \times 100 \dots\dots\dots (2)$$

$$W_{RP} = \frac{W_1 - (W_3 - W_2)}{W_1} \times 100 \quad \dots\dots\dots (3)$$

$$W_{FP} = \frac{W_3 - W_2}{W_1} \times 100 \quad \dots\dots\dots (4)$$

where W_P : mass per unit area of prepreg (g/m²)
 W_C : mass per unit area of carbon fibre (g/m²)
 W_{RP} : resin content by mass (%)
 W_{FP} : fibre content by mass (%)
 W_1 : mass of prepreg test specimen (g)
 W_2 : mass of glass filter (g)
 W_3 : mass of glass filter with collected fibre (g)

- (2) The resin content and fibre content for volatile free prepreg may be obtained by calculation using equations (5) and (6) for the equations (3) and (4) respectively.

$$W_{RP} = \frac{W_1 \left(1 - \frac{VC}{100}\right) - (W_3 - W_2)}{W_1 \left(1 - \frac{VC}{100}\right)} \times 100 \quad \dots\dots\dots (5)$$

$$W_{FP} = \frac{W_3 - W_2}{W_1 \left(1 - \frac{VC}{100}\right)} \times 100 \quad \dots\dots\dots (6)$$

where VC : volatile content (%)

The results calculated by equations (1) to (6) shall be expressed by integer place.

5.1.7 Report The report shall include the following:

- (1) Identifications of the samples
- (2) Test results for each of the three test specimens and average value. The test method, reagent, temperature and time which were used.
- (3) In the case of considering the volatile content, test results for each test specimens, average value and the temperature to measure the volatile content.

5.2 Volatile Content

5.2.1 Summary of Testing Method The test specimens taken from the width direction are dried in an oven, and the rate of the volatiles by mass is obtained. The volatile content may provide information of fluidity of resin at cure and of generation of void in the cured products.