Designation: ~~D352 – 97 (Reapproved 2016)~~ D352 – 23

Standard Test Methods for Pasted Mica Used in Electrical Insulation[1]

This standard is issued under the fixed designation D352; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the testing of bonded mica splittings and bonded mica paper to be used for commutator insulation, hot molding, heater plates, and other similar insulating purposes.

1.2 These test methods appear in the following sections:

Test	Sections
Compressive Creep	4 – 10
Dielectric Strength	38 – 41
Mica or Binder Content	19
Molding Test	31 – 36
Organic Binder	20 – 24
Resistivity	42 – 46
Silicone Binder	25 – 30
Stability Under Heat and Pressure	11 – 18

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety and health~~safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* See 40.1 and 45.1 for specific hazard statements.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies

D257 Test Methods for DC Resistance or Conductance of Insulating Materials

D1711 Terminology Relating to Electrical Insulation

[1] These test methods are under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and are the direct responsibility of Subcommittee D09.01 on Electrical Insulating Products.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms relating to electrical insulation, refer to Terminology D1711.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *binder content, n,* (of pasted mica)—the percent by weight of binder relative to the original weight of a specimen as determined by procedures specified herein.

3.2.1.1 *Discussion—*

Binder content includes any residual solvent. Pasted mica materials not fully cured (such as molding and flexible plates) ~~may~~possibly contain appreciable quantities of solvent in the binder. This solvent is usually later removed when the material is cured in the manufacture of electrical equipment. In such cases, the binder content after cure is less (by the amount of solvent removed) than would be determined by this method. To determine the binder content after cure of materials that are not fully cured, but subsequently will be, it is necessary, before initially weighing the specimen, to heat the specimen for a time and at a temperature that depends upon the material from which the specimen is prepared.

3.2.2 *compressive creep, n*—the change in thickness of a bonded micaceous material resulting from exposure to elevated temperature for a specified time while a specimen is under a specified compressive load.

3.2.3 *mica content, n,* (of pasted mica)—the percent by weight of mica relative to the original weight equal to 100 % minus the binder content as determined by procedures specified herein.

COMPRESSIVE CREEP

4. Significance and Use

4.1 This test determines the compressive creep under laboratory conditions or under conditions that ~~may be~~are possibly encountered during manufacture of electrical equipment. It has special significance if the material to be tested is applied as commutator segment insulation. It serves as a measure under specified conditions of the ability of the material to resist deformation while under compressive load, during exposure to elevated temperature for a specified time. This test is suitable for acceptance tests and for manufacturing control.

5. Apparatus

5.1 *Hydraulic Press*—A hydraulic press having temperature controlled, electrically heated platens, or a press with other provisions for heating the specimen and controlling the temperature. The platens shall be at least 4 by 4 in. (102 by ~~102 mm)~~ 102 mm) in size. The press shall be capable of exerting a force of at least 4000 lb (18 kN). The apparatus shall be capable of maintaining a specimen temperature of at least ~~200 ± 5°C.~~200 °C ± 5 °C. It is preferable that the apparatus have platens with water ducts or other provisions for cooling the specimen. (See Note 1 in 7.3.)

5.2 *Pressure ~~Gage—~~Gauge—*A pressure ~~gage~~gauge capable of determining the pressure on the specimen with an accuracy of ±5 %.

5.3 *Thickness ~~Gage—~~Gauge—*A thickness ~~gage~~gauge capable of measuring the thickness of the specimen to the nearest 0.001 in. (0.025 mm).

5.4 *Potentiometer*—Temperature measuring instrument and a No. 30 AWG or smaller thermocouple with overall accuracy of ~~± 2°C~~± 2 °C for measurement of specimen temperature.

5.5 *Steel Plates*—Two 4 by 4-in. (102 by ~~102-mm)~~102 mm) or larger polished steel plates of at least 1⁄16-in. ~~(1.6-mm)~~(1.6 mm) thickness, surface ground so that the top and bottom surfaces of each piece are parallel, one plate each for the top and bottom of the test specimen.

6. Test Specimen

6.1 The test specimen shall consist of a sufficient number of pieces of bonded micaceous plate, 2 by 2 in. (51 by ~~51 mm)~~ 51 mm)

in size, to form a stack approximately but not greater than ~~1.000 in.~~ 1.000 in. (25.40 mm) in thickness. The pieces shall be selected so as to be representative of the entire sheet. At least three specimens shall be tested for each lot of material.

7. Procedure

7.1 Center the stacked specimen between the 4 by 4-in. (102 by ~~102-mm)~~102 mm) steel plates and then center this assembly in the press. Place the thermocouple between pieces near the middle of the stack. Carefully align the stack to form a right parallelepiped. Apply a pressure of 1000 psi (7 MPa) to the specimen surfaces, and carefully determine the average thickness of the stack by means of the ~~gage.~~gauge. Where inside ~~gages~~gauges are used, measure the thickness at each of the four corners as close to the specimen as possible. Measurements shall be made within ~~5 min.~~5 min.

7.2 Pack approximately 2 in. (51 mm) of thermal insulation material around the specimen without disturbing it. Heat the specimen to ~~160 ± 5°C or 200 ± 5°C~~160 °C ± 5 °C or 200 °C ± 5 °C as specified. The time required to reach the specified temperature ~~should~~shall be not less than 30 min nor more than 75 min. The platen temperature shall not exceed the specified temperature by more than the specified tolerance. If the specimen is heated by other means, the surrounding medium shall not exceed the specified temperature by more than the specified tolerance. Allow the specimen to remain at the specified temperature for 2 h after reaching that temperature, and at the same time maintain the 1000-psi pressure.

7.3 Remove the thermal insulation and, while maintaining the pressure, allow the specimen to cool until the temperature is ~~5°C~~5 °C above the temperature (room ambient) at which the original thickness was measured. Control the rate of cooling such that it does not exceed the rate at which the temperature was raised. Then determine the thickness of the stack while under 1000-psi compressive load.

NOTE 1—Experience has shown that in order to cool the specimen to the specified temperature within a reasonable time, forced-cooling means must be employed. It is suggested that a fan be initially utilized to force air across the specimen for the first 5 min, after which cooling ~~water may be allowed~~ it is allowable to circulate in ducts provided in the platens. The rate of water flow, if used, ~~should~~shall be adjusted to give a cooling rate no greater than the rate at which the specimen was initially heated.

8. Calculation

8.1 Calculate the percentage compressive creep, C, as follows:

$$C, \% = [(T - T')/T] \times 100 \quad (1)$$

where:

T = thickness of the stack at 1000 psi (7 MPa) before heating, and
T' = thickness of the stack at 1000 psi after heating.

9. Report

9.1 Report the following information:

9.1.1 The identity of the material,

9.1.2 The nominal thickness of the pasted mica,

9.1.3 The observed values of T and T',

9.1.4 The percentage compressive creep, and

9.1.5 The specimen temperature.

10. Precision and Bias

10.1 This method has been in use for many years but no statement for precision has been made and no activity is planned to develop such a statement.

10.2 A statement of bias is not possible due to a lack of a standard reference material.

STABILITY UNDER HEAT AND PRESSURE

~~**11. Scope**~~

~~11.1 The test for stability under heat and pressure determines mica or binder displacement, or both, under the specified conditions of test.~~

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11.1 The test for stability under heat and pressure determines mica or binder displacement, or both, under the specified conditions of test.

12. Significance and Use

12.1 This test serves as a measure of the ability of bonded micaceous materials to maintain their physical integrity under exposure to heat and pressure. It has special significance where the material to be tested is employed as commutator segment insulation. This test is suitable for acceptance tests and for manufacturing control.

13. Nature of Test

13.1 This test method utilizes the application of a shearing force as well as a compressive force, which is accomplished by placing the specimens between specified wedges, thereby causing the applied force to resolve into compression and shear components. This test is particularly useful for material used in commutator assemblies where shearing as well as compressive forces are encountered. Test results are expressed quantitatively as units of linear deflection.

14. Apparatus

14.1 *Hydraulic Press, Pressure ~~Gage,~~Gauge, and Thermocouple* as described in Section 5, except that the hydraulic press shall be capable of producing a force of 26 400 lb (118 kN) on the specimen,

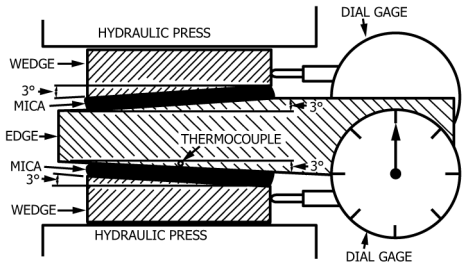
14.2 *Steel Wedges*—Two steel wedges of the same size as the specimen by approximately ¾ in. (19 mm) thick, with one face tapered at an angle of 3° with the horizontal and a center wedge as shown in Fig. 1. They shall be hardened and surface ground top and bottom.

14.3 *Dial ~~Gages~~—Gauges—*Two dial ~~gages~~gauges having 0.001-in. ~~(0.02-mm)~~(0.02 mm) graduations and a range of at least ½-in. ~~(13-mm),~~(13 mm), designed to be accurate at the specified test temperature, and suitably mounted on the steel wedges described in 14.2.

NOTE 2—Where the dial ~~gages~~gauges are mounted through nonmetallic bushings, or if some other suitable method is used to interrupt the metallic thermal path, it shall not be necessary to utilize ~~gages~~gauges designed to be accurate at the test temperature.

15. Test Specimen

15.1 The specimen shall consist of two rectangular pieces of bonded micaceous plate between 4 and 6 in.^{2} (2580 and ~~3870 mm~~3870 mm^2) in area, the shorter side being not less than 1½ ~~in. (38 mm).~~ in. (38 mm).



Minimum size of sheet: *A* = 3 in.; *B* = 18 in.

FIG. 1 Apparatus for Stability Test Under Heat and Pressure, Angular Method

16. Procedure

16.1 Insert the specimen between the wedges, as shown in Fig. 1. Center the assembly in the press and carefully align, using just enough pressure to hold the assembly together. Insert the thermocouple and fit it tightly in the hole provided in the center wedge. Cement the thermocouple into the hole. Apply a pressure of 100 ± 10 psi (690 ± 70 kPa) on the top and bottom assembly surfaces.

16.2 Pack approximately 2 in. (51 mm) of thermal insulating material, such as glass or other inorganic fiber mat, around the specimen without disturbing either the specimen or dial ~~gages.~~gauges. Heat the specimen to ~~160 ± 5°C or 200 ± 5°C,~~160 °C ± 5 °C or 200 °C ± 5 °C, as specified, and allow to remain at the specified temperature for 5 +1, −0 min. Do not allow the platen temperature to exceed the specimen temperature by more than ~~10°C.~~10 °C.

16.3 Adjust both ~~gages~~gauges to read zero. Apply and hold a pressure of 4400 psi (30 MPa) within 5 s on the top and bottom assembly surfaces and maintain for 15 min at the specified temperature. Record the deflection as determined by the top and bottom dial ~~gages~~gauges after 15 s, 30 s, 1, 2, 5, 10, and 15 min beginning with the instant that the 4400 psi pressure is obtained.

17. Report

17.1 Report the following information:

17.1.1 The identity of the material,

17.1.2 The dimensions of the specimen used,

17.1.3 The temperature used, and

17.1.4 The average deflection at each of the time intervals in accordance with 16.3.

18. Precision and Bias

18.1 See 10.1.

18.2 See 10.2.

MICA OR BINDER CONTENT

19. Significance and Use

19.1 Physical (such as the ability to hot mold, flexibility) and electrical (such as dielectric strength, resistivity) properties of bonded micaceous materials are affected, among other things, by the proportional contents of the binder and mica. The methods for mica or binder content are suitable for acceptance tests and manufacturing control.

ORGANIC BINDER

20. Apparatus

20.1 *Burner*—Bunsen burner or muffle furnace.

20.2 *Dishes*—Platinum or porcelain dishes or crucibles.

21. Test Specimen

21.1 *Specimens from Plates*—From a plate, cut a sufficient number of individual pieces in accordance with Fig. 2 to obtain a composite specimen weighing ~~5~~5 g to 10 g.

21.2 *Specimens From Fabricated Parts*—From a lot, take a representative test specimen weighing ~~5~~5 g to 10 g.

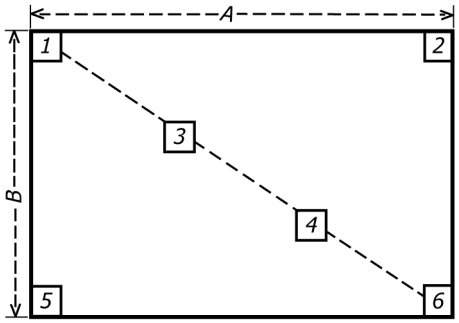


FIG. 2 Pattern for Location of Test Pieces for Determination of Mica of Binder Content

22. Procedure

22.1 **Warning—**This test method involves the use of heat to remove organic material which in a gaseous state is possibly hazardous. Conduct this test under a hood equipped with adequate ventilation. Alternatively, a muffle furnace with an adequate exhaust system is allowed to be used to burn off the mica until it is carbon free.~~Warning—This test method involves the use of heat to remove organic material which in a gaseous state may be hazardous. Conduct this test under a hood equipped with adequate ventilation. Alternatively, a muffle furnace with an adequate exhaust system may be used to burn off the mica until it is carbon free.~~

22.2 Weigh each specimen to the nearest ~~0.001 g~~ 0.001 g in a tared dish or crucible.

22.3 Place the dish with the specimen over a bunsen burner or in a muffle furnace and heat at a low red heat (to avoid the dehydration of mica) until all the organic material and carbon are burned off. After cooling in a desiccator, determine the weight of the residue.

23. Report

23.1 Report the following information:

23.1.1 The identity of the material,

23.1.2 Percentage loss in weight of each specimen indicated as binder, and

23.1.3 Percentage of residue in the crucible indicated as mica.

24. Precision and Bias

24.1 See 10.1.

24.2 See 10.2.

SILICONE BINDER

25. Apparatus

25.1 *Gooch Crucible,* containing a prewashed, dried, and weighed glass fiber mat (see Fig. 3).

25.2 *Beaker,* ~~500-mL,~~500 mL, alkali-resistant.

25.3 *Condenser,* for condensing reagent vapors.

25.4 *Flask*—Suction flask, ~~500-mL,~~500 mL, alkali-resistant, fitted with Gooch crucible adaptor.

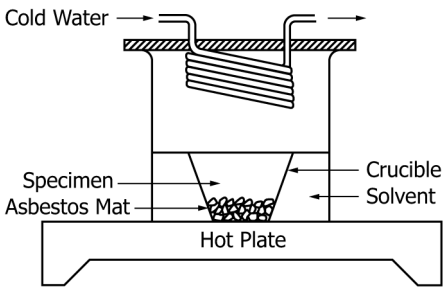


FIG. 3 Apparatus for Determination of Mica or Binder Content

25.5 *Hot Plate,* for boiling solvent mixture.

NOTE 3—It is not necessary to use the glass fiber mat of 25.1 if a test specimen contains bonded mica splittings.

26. Reagents

26.1 *Butyl Alcohol,* normal, cp grade.

26.2 *Toluol,* cp grade.

26.3 *Alkaline Solvent*—Dissolve about 5 g of potassium hydroxide (KOH) (ACS grade) in ~~100-mL~~100 mL normal butyl alcohol and add 400 mL of toluol.

26.4 *Ethyl Alcohol,* cp grade.

27. Test Specimen

27.1 Refer to Fig. 2. Cut sufficient material into pieces approximately ¼ in. (6 mm) size to obtain a specimen of ~~1~~1 g to 1.5 g.

28. Procedure

28.1 **Warning—**This test method involves the use of chemicals which are potentially hazardous. Conduct this test under a hood equipped with adequate ventilation. Keep flammable solvents away from open flames.~~Warning—This test method involves the use of chemicals which may be hazardous. Conduct this test under a hood equipped with adequate ventilation. Keep flammable solvents away from open flames.~~

28.2 Weigh the specimen in the previously weighed and dried gooch crucible packed with a suitable mat so as to prevent loss of fine mica flakes through the bottom (see Note 3). Place the crucible with specimen on or near the bottom of the alkali-resistant ~~500-mL~~500 mL beaker. Add sufficient alkaline solvent to the beaker to completely cover the mica sample so that the level of the solvent will be flush with the top of the crucible. Place a cover with the condenser over the beaker and boil vigorously for 4 h, taking care not to boil mica flakes out of the crucible.

28.3 Remove the crucible, taking care not to lose any of the fine mica flakes, and place it in the suction flask fitted with a suitable crucible adaptor. Wash with the contents of the beaker using a glass rod, if necessary, to return any fine mica flakes from the beaker to the crucible.

28.4 Clean the beaker and replace the crucible as before. Fill the beaker to the previous level with normal butyl alcohol, place the condenser on top of the beaker and boil for ½ h to remove the last traces of KOH.

28.5 Repeat the procedures described in 28.3 and 28.4, but boil with toluol instead of butyl alcohol.