Designation: D 352 – 97 (Reapproved 2003)

An American National Standard

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

This standard is issued under the fixed designation D 352; 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 *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 practices and determine the applicability of regulatory limitations prior to use.* See 40.1 and 45.1 for specific hazard statements.

1.4 The values stated in inch-pound units are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies[2]

D 257 Test Methods for DC Resistance or Conductance of Insulating Materials[2]

D 1711 Terminology Relating to Electrical Insulation[2]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms relating to electrical insulation, refer to Terminology D 1711.

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 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 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) in size. The press shall be capable of exerting a force of at least 4000 lb (18 kN). The apparatus shall be capable of

[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 D09.19 on Dielectric Sheet and Roll Products.

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[2] *Annual Book of ASTM Standards,* Vol 10.01.

maintaining a specimen temperature of at least 200 ± 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*—A pressure gage capable of determining the pressure on the specimen with an accuracy of ±5 %.

5.3 *Thickness Gage*—A thickness gage 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 for measurement of specimen temperature.

5.5 *Steel Plates*—Two 4 by 4-in. (102 by 102-mm) or larger polished steel plates of at least 1⁄16-in. (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) in size, to form a stack approximately but not greater than 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) 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. Where inside gages 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.

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 as specified. The time required to reach the specified temperature should 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 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 to circulate in ducts provided in the platens. The rate of water flow, if used, should 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.

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, 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*—Two dial gages having 0.001-in. (0.02-mm) graduations and a range of at least ½-in. (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 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 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^2) in area, the shorter side being not less than 1½ in. (38 mm).

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. Heat the specimen to 160 ± 5°C or 200 ± 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.

16.3 Adjust both gages 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 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 to 10 g.

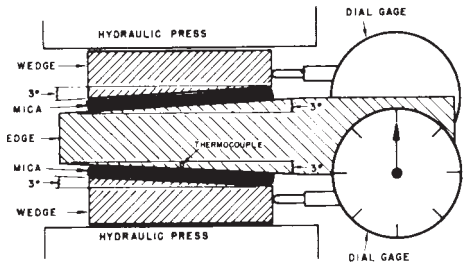
21.2 *Specimens From Fabricated Parts*— From a lot, take a representative test specimen weighing 5 to 10 g.

22. Procedure

22.1 (**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 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.



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

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

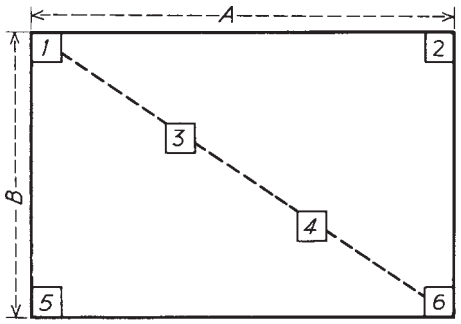


FIG. 2 Pattern for Location of Test Pieces for Determination of Mica of Binder Content

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, alkali-resistant.

25.3 *Condenser*, for condensing reagent vapors.

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

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 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 ¼in. (6 mm) size to obtain a specimen of 1 to 1.5 g.

28. Procedure

28.1 (**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 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.

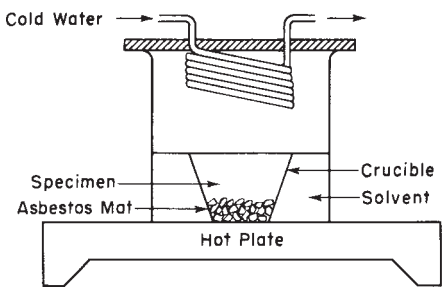


FIG. 3 Apparatus for Determination of Mica or Binder Content

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.

28.6 Repeat the procedure described in 28.3. Wash with ethyl alcohol. Then wash with approximately 250 mL of hot distilled or deionized water.

28.7 Dry the crucible and contents for 1 h at 110°C, cool in a desiccator to room temperature, and weigh.

29. Calculation

29.1 Compute the percent silicone binder from the ratio:

$$100\left[\frac{\text{Loss in weight of specimen}}{\text{Original weight of specimen}}\right] \quad (2)$$

30. Report

30.1 Report the following information:

30.1.1 The identity of the material, and

30.1.2 The percentage silicone binder for each specimen.

MOLDING TEST

31. Precision and Bias

31.1 See 10.1.

31.2 See 10.2.

32. Significance and Use

32.1 Under the prescribed heating conditions, the binder used in molding plate mica softens sufficiently to allow the plate to become flexible and to be formed by bending and with slight movement of the component mica splittings to take a definite shape. After cooling, the degree of form retention, flaking, and buckling indicates the suitability of the pasted mica plate for the purpose intended. This test is of considerable value for quality control and acceptance tests.

33. Apparatus

33.1 For specimens having a thickness of 1/16 in. (1.6 mm) and less, a mandrel having a diameter of 1½ in. (38 mm) shall be used.

33.2 For specimens over 1/16 in. (1.6 mm) up to and including 3/32 in. (2.4 mm) in thickness a mandrel having a diameter of 2 in. (51 mm) shall be used.

34. Test Specimen

34.1 The test specimen shall be 2 in. (51 mm) in width and of sufficient length to form a butt joint on the mandrel specified in 33.1 or 33.2.

35. Procedure

35.1 Heat the test specimen on a hot plate or a steam table at a specified temperature between the range from 90 and 125°C (194 and 257°F) and form around a mandrel as specified in 33.1 or 33.2. Then roll the specimen on a cold surface plate until it is cool and remove the mandrel.

36. Report

36.1 Report the following information:

36.1.1 The identity of the material,

36.1.2 The nominal thickness of the test specimen prior to forming on the mandrel,

36.1.3 The specified temperature of the hot surface,

36.1.4 A statement as to whether or not the mandrel buckles,

36.1.5 A statement as to the presence of flakes on the specimen surface, and

36.1.6 A statement as to whether or not the specimen retains its shape.

37. Purpose

37.1 The purpose of the molding test is to measure the ability of the sheet to hold its shape when molded.

38. Precision and Bias

38.1 No statement about precision or bias is warranted since this test method is qualitative.

DIELECTRIC STRENGTH

39. Significance and Use

39.1 In the applications for pasted mica for hot molding, commutator insulation, heating plates, and similar purposes pasted mica is subjected to electrical stresses in service which are a small fraction of the breakdown stresses determined by the short-time or step-by-step tests. The short-time and step-by-step tests are, however, well adapted for specification acceptance and factory control purposes in that they will detect spots, impurities, voids, and other defects which may render the material unsuitable in service.

40. Procedure

40.1 (**Warning—**Lethal voltages may be present during this test. It is essential that the test apparatus and all associated equipment that may be electrically connected to it be properly designed and installed for safe operation. Solidly ground all metal parts that any person might come into contact with during the test. Thoroughly instruct all operators in the proper way to conduct the tests safely. When making tests at high voltage, particularly in compressed gas or oil, the energy released at breakdown may be sufficient to result in fire, explosion, or rupture of the test chamber. Design of test equipment, test chambers, and test specimens should be such as to minimize the possibility of such occurrences, and to eliminate the possibility of personal injury.)

40.2 Determine the dielectric strength in accordance with Test Method D 149 except as follows:

40.2.1 Select either Method A of D 149 (the short time test) or Method B of D 149 (step-by-step test).

40.2.2 Use Type 1 electrodes from Table 1 of D 149.

40.2.3 If Method A is selected, use a rate of voltage application of 0.5 kV/s.

40.2.4 If Method B is selected, use a rate of voltage application in accordance with Table 1 of this standard using a 1 min dwell interval at each step. Make a change from each step to the next higher as rapidly as possible. Include the time to change in the succeeding test interval.

40.2.5 Make all tests in air at room temperature unless otherwise specified.

40.3 From a lot obtain a representative sample sufficient to prepare specimens which are large enough to provide at least five dielectric breakdown tests. Unless otherwise specified, test one specimen. The dielectric strength for any specimen is the average of the five tests.

41. Report

41.1 Unless otherwise specified, report the following information:

41.1.1 The identity of the material tested,

41.1.2 The average thickness of each specimen,

41.1.3 The breakdown voltage at each puncture,

41.1.4 The average, minimum, and maximum breakdown voltage for each specimen,

41.1.5 The ambient temperature,

41.1.6 The ambient relative humidity in percent,

41.1.7 The dielectric strength for each specimen,

41.1.8 Whether Method A or B of Test Method D 149 was used,

41.1.9 The short-time dielectric strength,

41.1.10 The deviations, if any, from any of the conditions specified in Section 39 of this test method, and

41.1.11 Any preconditioning of the specimens that was used.

42. Precision and Bias

42.1 See 10.1.

42.2 See 10.2.

RESISTIVITY

43. Terminology

43.1 See Test Methods D 257 for definitions relating to resistivity. The results are expressed in ohm-centimeter units and ohms, respectively.

TABLE 1 Rate of Voltage Application

Breakdown Voltage by Method A	Start Voltage for Method B	Step Increments for Method B
25 kV or less	1.0 kV	1.0 kV
over 25 to 50 kV	2.0 kV	2.0 kV
over 50 to 100 kV	5.0 kV	5.0 kV
over 100 kV	10.0 kV	10.0 kV

44. Significance and Use

44.1 The resistivity of pasted mica is a measure of its effectiveness as an electrical insulator under the application of constant unidirectional voltage. It serves primarily to indicate the presence of impurities and moisture, and to give information as to uniformity in processing. At this time this test is not recommended as a basis for acceptance or rejection.

45. Procedure

45.1 (**Warning—**Lethal voltages may be present during this test. It is essential that the test apparatus and all associated equipment that may be electrically connected to it be properly designed and installed for safe operation. Solidly ground all metal parts that any person might come into contact with during the test. Thoroughly instruct all operators in the proper way to conduct the tests safely.)

45.2 Determine the resistivity in accordance with Test Methods D 257 using Foil or Painted conductive electrodes under ambient conditions.

46. Report

46.1 Report the following information:

46.1.1 Description and identification of the material (name, grade, color, manufacturer, etc.),

46.1.2 Shape and dimensions of the test specimen,

46.1.3 Type and dimensions of electrodes,

46.1.4 Conditioning of the specimen (cleaning, predrying, hours at humidity and temperature, etc.),

46.1.5 Test conditions (specimen temperature, relative humidity, etc., at time of measurement),

46.1.6 Method of measurement,

46.1.7 Applied voltage,

46.1.8 Time of electrification of measurement,

46.1.9 Measured values of the appropriate resistances in ohms or conductances in mhos,

46.1.10 Computed values when required, of volume resistivity in ohm-cm units, volume conductance in mho-percentimetre units, surface resistivity in ohms, or surface conductivity in mhos, and

46.1.11 Statement as to whether the reported values are “apparent” or “steady-state.”

47. Precision and Bias

47.1 See 10.1.

47.2 See 10.2.

48. Keywords

48.1 binder content; bonded mica paper; bonded mica splittings; commutator segment insulation; compressive creep; heater plates; pasted mica