



Standard Test Method for Evaluating Hygrothermal Corrosion Resistance of Permanent Magnet Alloys¹

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1. Scope

1.1 This test method covers the equipment, procedures, and measurement of the resistance of permanent magnet alloys against corrosion in high temperature, high pressure water vapor environments. This test is also known as the Bulk Corrosion Test (BCT).

1.2 The values and equations stated in customary (cgs-emu and inch-pound) units or SI units are to be regarded separately as standard. Within this standard the SI units, when different from customary units, are shown in brackets, the values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with this standard.

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.*

2. Referenced Documents

2.1 ASTM Standards:²

D3330/D3330M Test Method for Peel Adhesion of Pressure-Sensitive Tape

2.2 JEDEC Standards:³

JESD22-A102-C Accelerated Moisture Resistance – Unbiased Autoclave

3. Terminology

3.1 Definitions:

¹ This test method is under the jurisdiction of ASTM Committee A06 on Magnetic Properties and is the direct responsibility of Subcommittee A06.01 on Test Methods.

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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 Available from JEDEC Solid State Technology Association, 2500 Wilson Boulevard, Arlington, VA 22201-3834. www.jedec.org

3.1.1 *bulk corrosion test (BCT) grade, n*—grade given to ranges of specific weight loss to indicate the relative corrosion resistance of a material. (The grades are listed in 15.2.)

3.1.2 *specific weight loss, n*—weight loss of the specimen per unit of exposed surface area, in units of milligrams per square centimetre, due to corrosion and disintegration.

4. Summary of Test Method

4.1 Permanent magnet samples are exposed to pressurized steam to determine the resistance of these materials, especially rare earth-type permanent magnets, to degradation by the combined action of heat and water vapor. The resulting weight loss (including removed loosely-attached material) is used to rate the hygrothermal corrosion resistance of the magnet.

5. Significance and Use

5.1 This test method provides a controlled corrosive environment which can be utilized to produce relative corrosion resistance information for sintered permanent magnets.

5.2 Although prediction of performance in natural environments has seldom been statistically correlated with corrosion test results, sufficient empirical results are available to support the usefulness of this test in quantifying relative resistance to corrosion from moisture and heat.

5.3 The reproducibility of results in this test method is dependent on the type, size, and shape of specimens tested, and the control of the operating variables. In any testing program, sufficient replicates should be included to establish confidence limits. Replicates may be run simultaneously, in subsequent test runs, or in duplicate test chambers.

5.4 When multiple test chambers and operators are utilized, efforts shall be made to perform a suitable repeatability and reproducibility study for the equipment, operators, and test method.

6. Interferences

6.1 This test method will not yield meaningful results if nonhomogeneous specimens such as assemblies or partially coated magnets are tested because of the potential aggravating effect of galvanic corrosion between dissimilar materials,

chemical reactivity with included materials, incompletely exposed surfaces, and difficulty in determining the amount of weight loss attributable to the magnet and coating.

6.2 Many cleansers contain bleach or chlorides which will increase the corrosion rate of the samples being tested. Do not use bleach- or chloride-containing cleansers to clean the interior of the test chamber or sample racks. Nonmetallic scrubbing pads and water rinsing have been found to be suitable for routine cleaning of chambers and racks.

7. Apparatus

7.1 *Autoclave*—A heated pressure chamber capable of maintaining pressurized steam at controlled temperature. The temperature shall be maintained within $\pm 2^{\circ}\text{C}$ of the selected temperature for the duration of the exposure. The atmosphere during test shall be saturated steam (100% RH). This can be achieved by venting the chamber as it reaches operating temperature and pressure and resealing the chamber after pressure has been reduced to 5 psi [34 kPa] through venting. The escaping steam purges the chamber of air, leaving only steam within the chamber. While commercial autoclaves are available that have self-contained heaters and timers, a conventional pressure cooker with external heater may also be used, as long as temperature and pressure controls capable of maintaining suitable control of these variables are present.

7.2 *Balance*—With suitable holders capable of measuring the mass of magnetic material to within $\pm 0.001\text{ g}$ or $\pm 0.1\%$ of the magnet's nominal weight (whichever is greater).

7.3 *Specimen Holders or Racks*—Suitable containers or racks which can keep specimens from touching each other or metallic parts of the autoclave chamber. They must be constructed of material that will not affect the corrosion rate of the sample. Suitable materials of construction have been found to be aluminum (see [Note 1](#)), borosilicate or laboratory glass, or halogen-free polymers.

NOTE 1—Aluminum readily develops an insulating film when exposed to air or steam. Care should be exercised when using freshly prepared or refinished aluminum surfaces as the oxide film may be thin enough to allow galvanic corrosion with the magnet under test. Generally, after exposure to steam, the oxide film is sufficiently thick to prevent galvanic corrosion.

8. Reagents and Materials

8.1 *Distilled Water*.

8.2 *Masking Tape*—Paper tape with a pressure-sensitive adhesive with strength of at least 1.57 lb/in. [28 kg/m] when tested in accordance with Procedure A of Test Method [D3330/D3330M](#).

8.3 *Brush*—Bristle brush made with synthetic polymer or natural fiber.

9. Known Hazards

9.1 This test method utilizes equipment operating at elevated pressure. Suitable means should be employed to ensure the equipment meets local safety regulations and is in good operating condition, particularly regarding the security of the chamber door and ports. Suitable means (typically a pressure

relief valve) shall be employed to ensure that the pressure within the equipment does not exceed its design limit.

9.2 This test method utilizes pressurized steam which has the ability to cause severe burns. Suitable means should be employed to ensure that personnel are protected from vented steam during operation, and in particular when the chamber is opened at the conclusion of the test.

9.3 This test method utilizes elevated temperature. Suitable means should be employed to prevent burns caused by the equipment, vented steam, and the specimens.

10. Sampling, Test Specimens, and Test Units

10.1 A minimum of three replicates should be tested.

10.2 Whenever possible the samples should be unmagnetized. Magnetized samples may be demagnetized electromagnetically or thermally, provided the thermal process does not damage the sample or significantly change its metallurgical state. Magnetized samples may be used in the magnetized state provided they are secured on the rack so that they do not contact other samples. Care shall be taken to prevent magnetic particles from being attracted to and sticking to the test samples, causing erroneous readings. Obtaining accurate weights of magnetic material is difficult on many balances. For magnetized samples, equipment and techniques that yield accurate weights shall be used, including spacing the magnet away from the mechanism of the balance.

11. Preparation of Apparatus

11.1 All equipment and specimen holders shall be inspected and cleaned as necessary prior to use to ensure that the test chamber, support racks, and other components are free of debris and visible contamination.

12. Calibration and Standardization

12.1 Verify calibration of balance and autoclave (temperature controller). Verify that autoclave test temperature is operating within specified range (see [7.1](#)).

13. Sample Preparation

13.1 Test specimens shall not come into direct contact with skin at any point during the procedure due to the potential for skin oils to influence the corrosion rate. Handle the specimens with gloved hands or clean tools.

13.2 Coatings (if present) or surface debris shall be removed using nonmetallic scrubbing pads or virgin nonmetallic abrasive grit blasting. Metallic scrapers or used abrasive grit shall not be used.

13.3 If the specimens must be cleaned with a liquid prior to test, use ACS reagent grade, nonpolar organic solvents only; do not use cleansers or bleaches which may influence the corrosion rate.

13.4 Immediately prior to the beginning of the test, the specimens shall be brushed (see [8.3](#)) to remove loosely adhered material. Once sufficiently brushed, the specimens shall be “taped” (see [8.2](#)) by repeatedly pressing fresh tape onto the surface of the specimen until there is no further evidence of transfer of material from the specimen to fresh tape.

13.5 Specimen identification shall be accomplished by a means that does not alter the surface of the specimen, such as mapping the specimen location in the specimen holders, or by features on the specimen holders. Marking the surface of the specimen with ink, labels, or scribed marks is not permitted due to potential influence on the corrosion rate. Even when the effects of marking do not influence the test, if corrosion is substantial, surface marks are likely to be removed by the testing.

13.6 Immediately prior to referee testing, the specimens shall be dry baked at temperatures between 100 to 122°C until a constant weight is achieved after “taping.”

14. Procedure

14.1 Determine the surface area of each specimen to the nearest 0.05 cm² or with a minimum resolution of 0.1% of the nominal surface area, whichever is greater.

14.2 Weigh the specimens (prepared as detailed in Section 13) to the nearest 0.001 g or 0.1% of the sample weight, whichever is greater.

14.3 Place the specimen in the autoclave such that there is ample exposure to the test atmosphere and so that contact with condensed water is minimized.

14.4 Expose the specimen to a saturated steam environment (100% RH) at the required temperature and time duration. Time duration is the cumulative time when the temperature and relative humidity are at required values. Once the test begins, the total intentional or unintentional interruptions of the test shall not exceed 1% of the total time duration. Standard conditions are 120 ± 2°C temperature for 96 h. A temperature of 120°C produces a nominal steam pressure of 29 psia [200 kPa]. See [Appendix X1](#) for additional temperature-pressure information. Other conditions (see [Note 2](#)) may be used if agreed upon by all affected parties.

NOTE 2—JEDEC JESD22-A102-C lists several time durations that are commonly used for autoclave testing of electronic components.

14.5 At the end of the test, the specimens shall be promptly removed from the test chamber, washed or scrubbed in warm tap water, pat dried and allowed to cool to room temperature. Prior to weighing, the specimens shall be brushed to remove loose corrosion products. Once sufficiently brushed, the specimens shall be “taped” in the same manner as during sample preparation.

14.6 The specimen shall be weighed to the nearest 0.001 g or 0.1% of the starting magnet weight, whichever is greater.

15. Calculation or Interpretation of Results

15.1 Calculate the specific weight loss, SWL, in milligrams per square centimetre of exposed surface, by the formula:

$$SWL = \frac{1000(W_i - W_f)}{A} \quad (1)$$

where:

W_i = the initial weight of specimen, in grams,

W_f = the final weight of specimen, in grams, and

A = the total surface area of the specimen, in square centimetres.

15.2 When specimens were exposed to the standard test conditions (in accordance with [14.4](#)), the BCT grade is defined by the following table based on the specific weight loss rounded as follows. For weight loss less than 10 mg/cm², round to the nearest 0.1; for weight loss equal to or higher than 10, round to the nearest whole number:

BCT Grade	Specific Weight Loss (mg/cm ²)
A	≤ 1.0
B	1.1 to 3.9
C	4.0 to 10
D	11 to 20
E	21 to 35
F	36 or more

16. Report

16.1 Report the following data for each test performed:

16.1.1 Specimen identification, including identity of the magnet material and any lot-specific notes.

16.1.2 Condition of the specimens, including whether the samples were magnetized, nonmagnetized, or demagnetized (including method of demagnetization); and whether the samples were previously coated (including method of coating removal).

16.1.3 Temperature, pressure, and duration of test.

16.1.4 The BCT grade (if the standard test conditions were used), or the average weight loss in mg/cm² of exposure surface area, or both.

16.1.5 Report results for each specimen.

17. Precision and Bias

17.1 Because the repeatability and reproducibility of the test method is significantly influenced by the specific characteristics of the test specimens, it is not considered possible to state meaningful values for repeatability and reproducibility that are universally applicable.

17.2 Since there is no accepted reference material for determining the bias for the procedure in this test method, bias has not been determined.

18. Keywords

18.1 autoclave; BCT; bulk corrosion test; corrosion resistance; hygrothermal; permanent magnet

APPENDIX

(Nonmandatory Information)

X1. APPENDIX

X1.1 Autoclave Test—An autoclave is a sealed vessel capable of being raised to greater than atmospheric pressure through the application of a gas or vapor. For the purposes of this test, the atmosphere is steam obtained by heating water in a closed vessel. Under these conditions, the relationship between temperature and pressure has been well established and the information, called “steam tables,” is published in numerous places. The chart in **Fig. X1.1** shows the relationship between the temperature within the vessel and the pressure measured using an absolute pressure gauge calibrated in kilopascals or a standard relative pressure gauge calibrated in pounds per square inch. Since the pressure is achieved by adjusting the temperature, accurate control of the temperature is necessary for proper testing. Even simple on-off type controls are capable of maintaining the temperature and pressure settings over the 96 plus hour test period, and variations are fully averaged out.

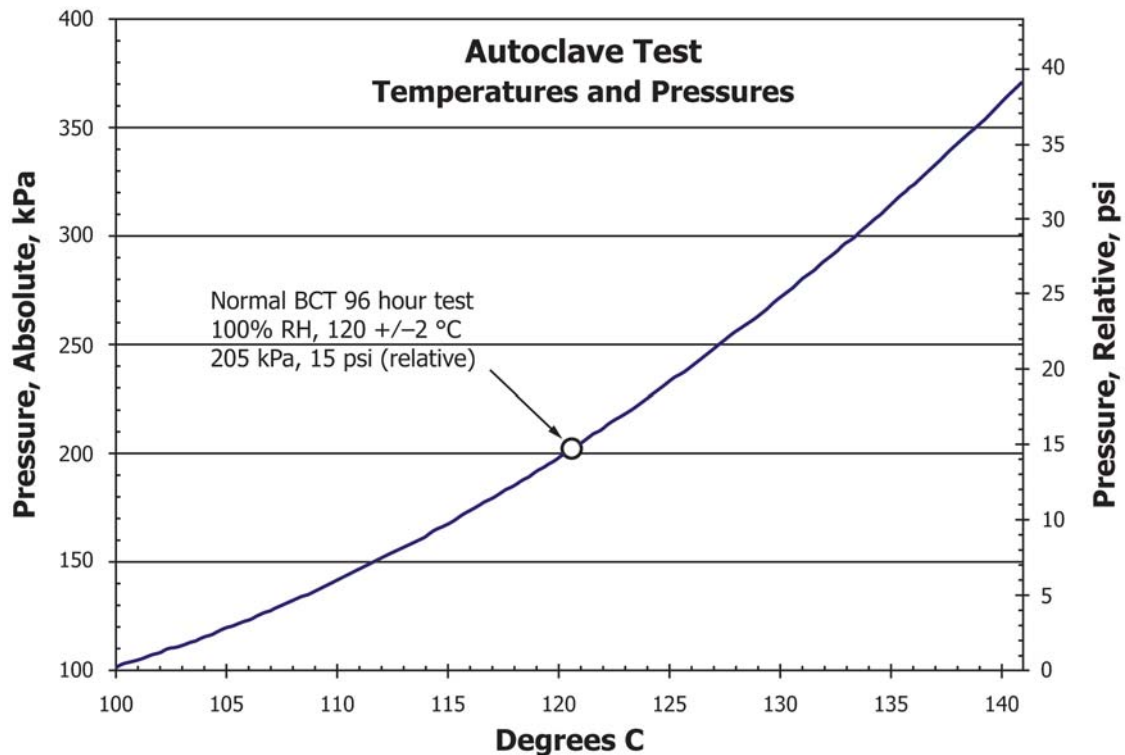


FIG. X1.1 Autoclave Test



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