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Standard Practice for Determining Cracking Susceptibility of Metals Exposed Under Stress to a Hot Salt Environment[1]

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1. Scope

1.1 This practice covers procedures for testing metals for embrittlement and cracking susceptibility when exposed under stress to a hot salt environment. This practice can be used for testing all metals for which service conditions dictate the need for such information. The test procedures described herein are generally applicable to all metal alloys; required adjustments in environmental variables (temperature, stress) to characterize a given materials system should be made. This practice describes the environmental conditions and degree of control required, and suggests means for obtaining this desired control.

1.2 This practice can be used both for alloy screening for determination of relative susceptibility to embrittlement and cracking, and for the determination of time-temperature-stress threshold levels for onset of embrittlement and cracking. However, certain specimen types are more suitable for each of these two types of characterizations.

NOTE 1—This practice relates solely to the performance of the exposure test. No detailed description concerning preparation and analysis of specimen types is offered. However, the optimum sample design may be one that uses the same type of stress encountered in service loading situations. Standards describing principal types of stress corrosion specimens, their preparation, and analysis, include Practices G 30, G 38, and G 39.

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.* (For more specific safety hazard statements see Section 8.)

2. Referenced Documents

2.1 *ASTM Standards:*

D 1141 Practice for the Preparation of Substitute Ocean Water[2]

D 1193 Specification for Reagent Water[3]

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens[4]

G 30 Practice for Making and Using U-Bend Stress-Corrosion Test Specimens[4]

G 38 Practice for Making and Using C-Ring Stress-Corrosion Test Specimens[4]

G 39 Practice for Preparation and Use of Bent-Beam Stress-Corrosion Test Specimens[4]

G 49 Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens[4]

3. Summary of Practice

3.1 The hot salt test consists of exposing a stressed, salt-coated test specimen to elevated temperature for various predetermined lengths of time, depending on the alloy, stress level, temperature, and selected damage criterion (that is, embrittlement, cracking, or rupture, or a combination thereof). Exposures are normally carried out in laboratory ovens or furnaces with associated loading equipment for stressing of specimens.

3.2 The ovens are provided with facilities to circulate air at various flow rates and ambient pressure. However, for certain specific applications, airflow and pressure may be adjusted to obtain information on material behavior in simulated service environments. Exposure temperatures and stress levels are generally selected on the basis of mechanical property data for a given alloy, or of expected service conditions, or both.

4. Significance and Use

4.1 The hot salt test as applied to metals is utilized as a secondary design consideration indicator, as cracking has been shown to occur in laboratory tests simulating possible service conditions. Although limited evidence exists linking this phenomenon to actual service failures, cracking under stress in a hot salt environment should be recognized as a potential design controlling factor.

4.2 The hot salt test is not to be misconstrued as being related to the stress corrosion cracking of materials in other environments. It is considered solely as a test in an environment that might be encountered in service.

4.3 Because hot salt cracking under stress is considered a secondary design consideration and service failures have not been attributed solely to this phenomenon, manufacturing

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[2] *Annual Book of ASTM Standards*, Vol 11.02.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 03.02.

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processes will be optimized or alloying changes will be made only after consideration is given to primary design factors such as creep resistance of a given high temperature alloy. The usefulness of the test lies rather in limiting maximum operating temperatures and stress levels or categorizing different alloys as to susceptibility, or both, if it is found that hot salt damage may accelerate failure by creep, fatigue, or rupture.

4.4 Finally, the test does not lend itself to the utilization of pre-cracked specimens because cracking reinitiates at any salt-metal-air interface, resulting generally in many small cracks which extend independently. For this reason, specimens that are recommended for utilization in routine testing are of the smooth specimen category.

5. Interferences

5.1 Hot salt cracking under stress is often considered a hydrogen-related phenomenon, and the source of hydrogen is a corrosion reaction involving moisture, available either from the hydrated salt, trapped as fluid inclusions in nonhydrated salt, or from humidity in the test atmosphere if absent in the salt crystals. Because of this fact, considerable variation in test results can be obtained, simply from the method of salt deposition on the test specimen, even when effective controls on other test variables are realized. Efforts should be made to standardize the salt deposition techniques and to control or monitor humidity in order to achieve desired test validity.

5.2 The effects of cycling time at temperature to achieve a given total cumulative exposure have been shown to have a significant effect on test results, with shorter cycle duration and greater cycle frequency generally resulting in less damage for the same cumulative exposure time. For this reason, selection between continuous and cyclic exposure, duration, and frequency of cycling, and heating and cooling rates must be made with the end purpose of the test in mind.

5.3 Variations in heat to heat or product forms, or both, have been shown to have a significant effect on damage thresholds determined from experimental testing. This effect may be more pronounced than is observed in more conventional stress corrosion testing of the aqueous type. For this reason, it is important to obtain and document to the fullest extent possible all certified analyses and tests associated with the material to be tested and associated fabrication and treatment histories. Interstitial concentration levels, chemical contaminants, and thermomechanical processing should be included in the documentation (see Section 12).

5.4 Details regarding general surface preparation and use of bent-beam stress-corrosion specimens are outlined in Practice G 39. Procedures for making and using direct tension stress-corrosion specimens is described in Practice G 49. However, because of the highly localized nature of onset of attack at the surface in hot salt exposure testing, it is desirable to characterize as fully as possible the surface condition of the material. If an as-received surface condition is to be investigated, efforts should be made to ascertain the state of residual stress as regards the material surface. Both magnitude and algebraic sign (tension or compression) of residual stress should be determined and reported if possible. Chemical milling can be employed in final surface preparation in order to avoid extraneous surface effects. However, care should be taken to ensure that proper chemical milling techniques are employed, and that hydrogen uptake does not occur during the surface preparation.

6. Apparatus

6.1 *Apparatus for Salt Coating*—A conventional air brush should be used for spraying the specimens to accomplish the salt-coating procedure. This will generally provide a thin uniform salt deposition of the desired density.

6.2 *Apparatus for Conducting Exposure Test*:

6.2.1 Apparatus required for conducting the exposure test depends on the selection of the specimen type to be used. If a constant-deflection type specimen is utilized for which no external loading requirement exists, conventional laboratory ovens are suitable for conducting the exposure test. Provision for controlling or monitoring inlet air humidity is recommended.

6.2.1.1 *Specimen Holders* suitable for applying stress to constant-deflection type specimens should be made of the same or a similar alloy as the material to be tested in order to avoid galvanic effects. The requirement for the use of a fixture to apply stress can be avoided when testing sheet materials by utilizing a self-stressed specimen design.[5]

6.2.1.2 *Racks* suitable for supporting specimens in the oven and for transferring specimens should be made of the same or a similar alloy as the material to be tested. Opencircuit conditions should be maintained, although galvanic effects are considered to be highly localized on the surface.

6.2.2 If a constant-deflection type specimen is utilized, care must be taken to either avoid or take into account differences in thermal expansion between test specimen and test fixture. Thermal expansion differences can substantially change the stress level applied at ambient temperature when specimens are heated to the test temperature.

6.2.3 If a constant-load type specimen is to be utilized, provision must be made to combine both heating and loading equipment. Vertical-tube resistance-wound furnaces can be utilized with dead-weight loading or conventional creep frame equipment for low and high loading conditions, respectively (Note 2). Direct induction or resistance heating of the specimen itself is not recommended.

NOTE 2—When using vertical-tube furnaces care must be taken to avoid a chimney effect through the furnace, which could result in excessive airflow and uneven temperature distribution along the specimen length. Sealing at both ends will allow control of air flow and improve temperature distribution within the furnace.

7. Reagents and Materials

7.1 Reagent grade salts shall be used when preparing solutions from which the salt coating is derived. Sodium chloride (NaCl) should be used for routine testing. Other salts that may be encountered in service can be used for specialized applications. Synthetic sea water (Note 3), should be used for characterizing alloys for use in marine environments.

NOTE 3—If tests are to be conducted on specimens with salt deposits derived from substitute ocean water, solutions should be prepared in accordance with Specification D 1141.

[5] See "A Stress Corrosion Test for Structural Sheet Materials," *Materials Research and Standards*, Vol 5, No. 1, January 1965, pp. 18–22.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type IV water prepared in accordance with Specification D 1193.

8. Hazards

8.1 Shatterproof glasses with side shields should be worn when handling and examining stressed samples. Generally the required safety equipment is similar to that used for conducting routine mechanical tests.

8.2 Appropriate heat-resistant equipment, for example, gloves, may be required when exposing test samples to high temperatures.

9. Calibration and Standardization

9.1 When conducting elevated temperature exposure tests, determination of the temperature profile within the oven or furnace should be made, including temperature sampling along the width, depth, and height of the hot zone to ensure that temperatures within all locations of specimen exposure are within prescribed limits. Deviation from the desired test temperature should not be more than ±2 % of the absolute temperature.

9.1.1 Temperature control of the exposure test shall be accomplished by determining true specimen temperature. This can be done by means of affixing a thermocouple of appropriate sensitivity for the temperature range to be investigated onto a control specimen either by spotwelding or mechanical fastening. In either instance it must be determined that the technique of thermocouple fastening does not introduce any interference effects.

9.2 The degree of control required on the applied stress of a test specimen depends on the nature and purpose of the test. When determining threshold values of time-temperature-stress for onset of embrittlement or cracking for a given alloy as a secondary design consideration, control should be more stringent than that for indication of trends or determination of relative material susceptibilities.

9.2.1 In determining threshold values for onset of embrittlement or cracking, constant-load type specimens, for which the level of applied stress can be more tightly controlled, are recommended. Deviation from the desired target stress level should not be more than ±2 %.

9.2.2 When utilizing constant-deflection type specimens for the determination of behavior trends or relative material susceptibilities, specimen geometry should be limited such that control of the applied stress level can be maintained within ±10 % of the desired stress level.

9.3 *Humidity Control*—For routine testing, active control of humidity is not considered mandatory. Most testing is accomplished using ambient laboratory air. However, daily monitoring and recording of humidity should be made and humidity considered as a potential cause of data scatter. In tests for ascertaining the effects of humidity on cracking behavior, moisture levels can be adjusted by mixing various ratios of saturated and dry air to oven or furnace air inlet. Sampling of dew point at oven or furnace inlet will allow determination of humidity of the air at ambient conditions.

9.4 *Airflow*—Care must be taken to prevent airflow velocities beyond that achieved in recirculating ovens (30 to 120 m/min (100 to 400 ft/min)). Variations in this factor have been shown to produce differences in test results. If airflow is an experimental variable to be investigated, it should be controlled and monitored.

10. Procedure

10.1 *Cleaning of Specimens*—Before salt coating, thoroughly clean the specimens to remove all identification markings, grease, oil, or other hydrocarbon contaminants. Specimens may be cleaned in a variety of cleaning media, but end the cleaning procedure with a hot and cold water rinse. Do not clean the specimens with chlorinated hydrocarbons such as trichloroethylene because these compounds can chemisorb, and decompose after heating, which will affect exposure test results. Information contained in Practice G 1 on clearing methods may be utilized where appropriate.

10.2 *Salt Coating of Specimens*:

10.2.1 Salt coat the specimens in such a manner as to provide many small separate particles. This is best accomplished by preparing a salt solution for spraying the specimens. The concentration of the salt solution should provide a reasonable salt deposit for each spray-drying cycle. The 3.5 % salt solution has been shown to produce very satisfactory results and, because of its widespread use in other tests, is arbitrarily selected as a baseline for the test described herein.

10.2.2 Spray the specimens with the prepared solution by a means that provides atomization of the solution and uniform coverage of the test specimen. A conventional air brush will provide satisfactory results. Spray specimens horizontally to minimize run-off prior to drying. Specimens are normally sprayed on the surface(s) that is or will be stressed in tension during the exposure. Spray constant-deflection type specimens after stressing so that salt deposits do not spall off during the stressing operation. After spraying, dry the specimens in a humidity- and temperature-controlled atmosphere. Temperature selection for the drying cycle should be based on the salt composition utilized. Generally, higher drying temperatures will result in less water entrapment within the crystal. Desired salt densities can be accomplished by repetitive spray-drying cycles. Salt densities in the range from 0.155 to 155 g/m^2(0.1 to 100 $mg/in.^2$) are reasonable minimum and maximum levels, respectively (Note 4). For routine testing and materials screening, utilize a salt coating of 15.5 g/m^2(10 $mg/in.^2$).

NOTE 4—Cracking initiates at salt-metal-air interfaces on the surface of a test specimen. Because of this fact, the distribution and size of salt particles can play a significant role in the incidence of cracking and the time to stress rupture. Larger salt particles may result in fewer cracks initiating which may, in turn, affect time to stress rupture. If the salt coating is dry and continuous such that the atmosphere cannot reach the salt-metal interface, cracking will not initiate. An example of such a situation would be in the application of a thicker paste type of salt coating which may not produce cracking within the test section at all. It is based on this explanation that the method of application and coating density limits were selected. Efforts should be made to document salt film appearance as well as to record the density in order to fully characterize test conditions.

10.3 *Exposure*:

10.3.1 After salt coating, expose the specimens to the desired test temperature for a predetermined exposure time. The temperature range of investigation is from 230 to 540°C (450 to 1000°F). Most of the testing is accomplished at the expected service temperature. If the specimens are of the constant-deflection type, insert them into the laboratory oven, which should be at test temperature before insertion (Note 5). Remove the specimens from the oven and allow to air cool to ambient temperature before examination or recycling.

10.3.2 If the specimens are of the constant-load type, place them in the test furnace, seal for necessary atmosphere control, and bring to test temperature before loading. Begin the duration of the exposure test following specimen loading. Unload the specimens following the desired exposure time interval (if less than time to rupture is desired) and allow to cool with furnace power off and end seals removed prior to examination or recycling. Keep the time for heat-up and cool-down as short as possible and note as a potential source of data scatter.

NOTE 5—When utilizing constant-deflection type specimens for elevated temperature testing, exercise caution to ensure that excessive stress relaxation of the specimens does not occur. This can be determined by exposure of control specimens (generally not salt coated) along with the test specimens and examination of control specimens after exposure by unloading and observing whether or not permanent deformation of the specimen has occurred. Stress level of control specimens after exposure can also be determined by measurement of stress using X-ray techniques. Both of these methods have been shown to effectively indicate stress relaxation.

10.4 *Exposure Duration*—Determine the exposure duration by the inherent resistance of the alloy tested, the specimen configuration and dimensions, and the desired criterion to be used as the damage indicator. For instance, the time to complete rupture of the specimen will always be longer than the time for initiation of cracking, which in turn will be longer than time for embrittlement of the material to occur. All three factors may be used as criteria for failure. However, embrittlement and cracking are considered more appropriate from a design consideration standpoint. Appropriate exposure periods can be determined by trial and error at a given test temperature by high initial loadings, which will result in shorter exposure times to failure or damage. Subsequent tests can be conducted at lower stress levels for longer exposure times. Exposure times can also be determined from expected service conditions if simulation of service application is desired.

11. Interpretation of Results

11.1 The interpretation of results will vary with the type of specimen utilized. Generally, cracks will not be visible on specimens after exposure if the exposure has been terminated prior to stress rupture. However, if specimens are subsequently tested in tension or bending at room temperature following the exposure test, damage will become evident either by elongation and a general reduction in failure load (compared to control specimens which were not salt coated), or by the decrease in strength and elongation and the appearance of numerous small cracks in the vicinity of the primary fracture of the test specimens. The former indicator is generally observed in slow strain-rate testing prior to time for actual cracking to occur. By stepwise reduction of exposure duration, temperature, or stress, or a combination thereof, thresholds can be determined for the desired range of exposure conditions for a given material.

11.2 When a constant-deflection type specimen is utilized (for example, a bent-beam specimen), the subsequent room temperature test should be a bending test with the bend radius located in the test section of the specimen. When a constant-load type specimen is utilized (for example, a direct tension specimen), the subsequent room-temperature test should be a conventional tension test of the specimen to determine residual mechanical properties of the specimen. However, strain rate should be kept constant at 0.005/min throughout the test.

11.3 Following the subsequent room-temperature testing, specimens should be examined visually or at low-power magnification (10× to 100×), or by both methods, to verify that failure or damage was caused by exposure under stress to hot salt. Cracks may be small and will often emanate from a salt crystal. If cracks are heat tinted, they occurred during the exposure. If they are not, they occurred during subsequent testing but still may be attributed to exposure damage. Salt crystals, however, often spall off during the subsequent room-temperature test in the areas of greatest damage because of high levels of strain in those areas. Fracture surfaces may also be examined using various fractography techniques to ascertain the extent of area on the fracture surface affected by the hot salt. Any unusual failures or test anomalies should be carefully scrutinized to ascertain failure cause and determine whether or not associated data shall or shall not be included in the report.

12. Report

12.1 It is recommended the following information be included in the report of the test results:

12.1.1 Identification and composition of the alloy, hardness and grain size.

12.1.2 The alloy heat, mill product form, and heat treatment of material tested, including the specification to which the parent material was prepared.

12.1.3 Detailed producer-certified chemical and mechanical property analysis of the lot of material tested. This is mandatory for materials characterization, and desirable for qualifying a given batch of material for service.

12.1.4 Location and orientation of specimens tested.

12.1.5 Specimen details; type and dimensions. See Practices G 30, G 38, G 39, and G 49 for details regarding specimen types, fabrication, and analysis.

12.1.6 Specimen cleaning procedures and surface condition of specimen, including special surface treatments.

12.1.7 Type of salt coating, density, and method of application, including documentation of drying-cycle temperature and humidity control or monitoring, or both, and documentation of salt crystal distribution and appearance.

12.1.8 *Details of the Exposure Test*:

12.1.8.1 Exposure temperature, time, and stress level of test specimens.

12.1.8.2 Whether exposure was continuous or cyclic, and details of the type of cycles used.

12.1.8.3 Statement on humidity control and monitoring.

12.1.9 Details of subsequent room-temperature testing.

12.1.10 Notation of any deviations in the test procedure from that set forth as described herein.

12.1.11 The number of specimens tested per condition and the range of scatter in the test results.

12.1.12 Supporting information derived either visually or metallurgically, from post-test examination, which will aid the potential receiver in the understanding of the behavior of the material tested.

13. Keywords

13.1 hot (230-540°C) salt simulated service environment; laboratory test; sodium chloride coated stressed specimens

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