



Standard Test Method for Determining Whether Gas-Leak-Detector Fluid Solutions Can Cause Stress Corrosion Cracking of Brass Alloys¹

This standard is issued under the fixed designation G186; 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 This test method covers an accelerated test method for evaluating the tendency of gas leak detection fluids (LDFs) to cause stress corrosion cracking (SCC) of brass components in compressed gas service.

1.2 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.

1.3 *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 to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

B135 Specification for Seamless Brass Tube

B135M Specification for Seamless Brass Tube [Metric]

D1193 Specification for Reagent Water

G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens

G15 Terminology Relating to Corrosion and Corrosion Testing³

G37 Practice for Use of Mattsson's Solution of pH 7.2 to Evaluate the Stress-Corrosion Cracking Susceptibility of Copper-Zinc Alloys

G38 Practice for Making and Using C-Ring Stress-Corrosion Test Specimens

¹ This test method is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.06 on Environmentally Assisted Cracking.

Current edition approved March 1, 2011. Published April 2011. Originally approved in 2005. Last previous edition approved in 2005 as G186–05. DOI: 10.1520/G0186-05R11.

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³ Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 **Gas Leak Detector Solutions**—Also known as leak detection fluids, leak detector solutions, bubble solutions, and soap solutions, designated in this standard as LDFs, are fluids used to detect leaks in pressurized gas systems by the formation of bubbles at the leak site.

3.1.2 The terminology used herein, if not specifically defined otherwise, shall be in accordance with Terminology **G15**.

4. Summary of Test Method

4.1 This test method consists of three steps: The first step consists of running a sample of the test specimens to verify that they are susceptible to stress corrosion cracking using Mattsson's Solution (see Practice **G37**). The second step is to expose the specimens to a solution that does not cause SCC to verify that the test environment does not contain components that can cause SCC to brass. The third step is to test the LDF to determine if it causes SCC of the brass specimens within 15 wetting and evaporation cycles.

4.2 The specimen used in this test is a C-ring stressed to create at least 0.65% strain in the outer fibers of the specimen.

4.3 Macroscopic examination of the specimens is carried out after every second wetting cycle and if cracking is suspected the specimen is examined at higher magnifications for confirmation. Metallographic sectioning through the stressed area is used to verify minor cracking at the end of the fifteen cycles.

4.4 LDFs that cause SCC in any specimens within 15 wetting cycles are considered to have failed this test and not suitable for use in pressurized gas systems with brass components.

5. Significance and Use

5.1 Brass components are routinely used in compressed gas service for valves, pressure regulators, connectors and many other components. Although soft brass is not susceptible to ammonia SCC, work-hardened brass is susceptible if its hardness exceeds about 54 HR 30T (55HRB) (Rockwell scale). Normal assembly of brass components should not induce sufficient work hardening to cause susceptibility to ammonia

SCC. However, it has been observed that over-tightening of the components will render them susceptible to SCC, and the problem becomes more severe in older components that have been tightened many times. In this test, the specimens are obtained in the hardened condition and are strained beyond the elastic limit to accelerate the tendency towards SCC.

5.2 It is normal practice to use LDFs to check pressurized systems to assure that leaking is not occurring. LDFs are usually aqueous solutions containing surfactants that will form bubbles at the site of a leak. If the LDF contains ammonia or other agent that can cause SCC in brass, serious damage can occur to the system that will compromise its safety and integrity.

5.3 It is important to test LDFs to assure that they do not cause SCC of brass and to assure that the use of these products does not compromise the integrity of the pressure containing system.

5.4 It has been found that corrosion of brass is necessary before SCC can occur. The reason for this is that the corrosion process results in cupric and cuprous ions accumulating in the electrolyte. Therefore, adding copper metal and cuprous oxide (Cu_2O) to the aqueous solution accelerates the SCC process if agents that cause SCC are present. However, adding these components to a solution that does not cause SCC will not make stressed brass crack.

5.5 Repeated application of the solution to the specimen followed by a drying period causes the components in the solution to concentrate thereby further increasing the rate of cracking. This also simulates service where a system may be tested many times during its life. These features of the test method accelerate the test and allow an answer to be obtained more rapidly.

5.6 This test method applies only to brasses. Successful passage of this test does not assure that the LDF will be acceptable for use on other alloy systems such as stainless steels or aluminum alloys.

6. Interferences

6.1 When conducting this test, it is very important that the air not be contaminated with ammonia vapors. Reagent bottles with ammonium hydroxide or other tests that involve ammonia or its compounds including amines must not be in the vicinity of these tests. This also includes Mattsson's test solution.

6.2 Cross contamination may result in false stress corrosion cracking results therefore concurrent exposure tests with different leak detector solutions should be conducted in such a way that any set of samples does not influence the results of any of the other samples.

6.3 In this test, the susceptibility of the C-ring specimens to crack in a particular test solution can be affected by the temper of the brass alloy; therefore, it is crucial that the C-rings be fabricated from hard drawn temper brass tubing that meets the minimum specified hardness requirements.

7. Reagents and Materials

7.1 Reagent grade cuprous oxide (Cu_2O) and USP/FCC grade glycerin ($\text{C}_3\text{H}_8\text{O}_3$) conforming to specifications of the

committee on Analytical Reagents of the American Chemical Society, where such specifications are applicable,⁴ shall be used.

7.2 Fine pure copper powder with particle size $<68\ \mu\text{m}$ shall be used.

7.3 Solutions using water shall be prepared using distilled or deionized water conforming to the purity requirements of Specification **D1193**, Type IV reagent water.

7.4 Leak detector solutions shall meet manufacturer's specifications.

7.5 Mattsson's Solution shall be freshly prepared according to Practice **G37**.

8. Hazards

8.1 Consult Material Safety Data Sheets (MSDS) for all chemicals both reagent and commercial before testing to gain a full understanding of any potential hazards.

8.2 The test solutions present no undue safety hazard. It is recommended, however, that appropriate personnel protection equipment such as resistant gloves and shatterproof eyewear with side shields be worn when the chemicals or specimens are handled.

8.3 The solutions contain copper and are thus considered poisonous so they should not be ingested.

8.4 Ammonium sulfate, $(\text{NH}_4)_2\text{SO}_4$, in the Mattsson's solution has been reported to be allergenic. Repeated short-time skin contact with the solution over extended periods of time should be avoided.

8.5 The fumes given off by the Mattsson's test solution contain ammonia. The least detectable ammonia odor corresponds to a concentration of 50 ppm; 100 ppm can be tolerated for several hours without serious disturbance; 700 ppm causes immediate eye irritation; and greater than 5000 ppm can be lethal. The mixing and the actual testing with Mattsson's solution should therefore be run in a well-ventilated area.

9. Test Solutions

9.1 *Control Solution (benign water solution)*—Add 2.5 g Cu powder and 2.5 g Cu_2O to 1000 mL of H_2O . Then add 10 mL of glycerin to solution. Shake solution vigorously to thoroughly mix contents.

9.2 *Leak Detector Solutions*—Add 2.5 g Cu powder and 2.5 g Cu_2O to 1000 mL of each manufacturer's solution to be tested. Shake solutions vigorously to thoroughly mix contents.

NOTE 1—Some of the solids will settle out of the solutions in between cycles, therefore, it is very important to shake the solutions vigorously prior to their use in the wetting cycle.

10. Test Specimen

10.1 *Type and Size*—An unnotched C-ring in accordance with Practice **G38** shall be used (**Fig. 1**). C-rings shall have an outer diameter (OD) between 1.0 to 2.0 in. (25.4 to 50.8 mm)

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

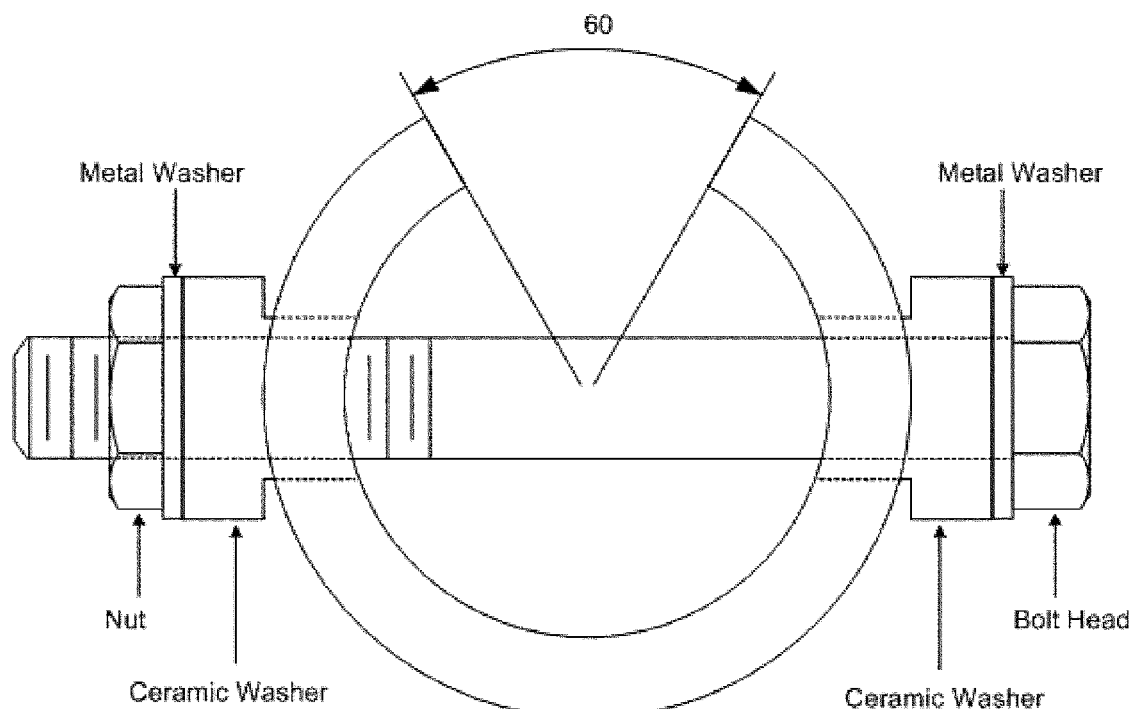


FIG. 1 C-ring Specimen

and thickness between 0.065 to 0.25 in. (1.65 to 6.4 mm). Widths shall be fixed at 0.75 in. (19.0 mm).

10.2 *Alloy Composition and Temper*—Test specimens shall be fabricated from copper alloy (UNS C27200) seamless tube with a H80 hard-drawn temper. The hard drawn temper shall have a minimum hardness of 70 on the 30T Rockwell scale which corresponds to minimum tensile strength of 66 ksi (455 MPa). Refer to Specifications B135 and B135M for tube information.

10.3 *Surface Finish*—Specimens shall have a 120-grit finish. Grind marks shall be in the circumferential direction.

10.4 *Restraining Hardware*—Nuts, bolts, and flat washers shall be made from a metal resistant to the chemicals used in this test (for example, Type 316 stainless steel, UNS S31600). Insulation washers shall be made from a hard resistant material to prevent relaxation of the stressed C-ring specimen (for example, alumina). Refer to Fig. 1.

10.5 *Pre-test Condition*—C-rings will be unstressed prior to testing.

10.6 *Stressing Method*—Constant-strain stressing method shall be used in stressing the C-rings (refer to Practice G38). Tensile stress will be introduced on the exterior of the ring by tightening a bolt centered on the diameter of the ring.

10.7 *Surface Preparation*—The specimen surface should be free of oil, grease, and dirt. This usually entails cleaning with organic solvent such as acetone followed by an alcohol rinse.

NOTE 2—Every precaution shall be taken to maintain the integrity of the surface after the final preparation. Avoid rough handling that could mar the surface and handle with gloves to prevent fingerprinting and the transfer of contaminants.

11. Test Setup and Apparatus

11.1 *C-ring Test Assembly*—Individual C-ring specimens shall be placed apex down in a glass-fiber-wick covered dish (Fig. 2).

11.2 *Exposure Dish*—The individual dishes shall be made out of a material that will not react with the chemicals being used for the exposure (for example, glass, polystyrene, polycarbonate). The required dish dimensions are 2.36- to 3.94-in. (60- to 100-mm) diameter and 0.59- to 0.79-in. (15- to 20-mm) height.

11.3 *Wick Material*—Borosilicate glass wick material with a fiber diameter of about 0.3 mil (8 μ m) shall be used.

11.4 *Number of Specimens*—Five individual C-ring specimen test assemblies will be used for each leak detector or control solution to be tested.

11.5 *Test Assembly Arrangement*—Individual exposure dishes shall be laid out such that there is at least a 1.0 ft (305 mm) separation between groups of specimens (Fig. 3). The control solution specimens shall always be positioned in the center of the test group.

12. Calibration and Standardization

12.1 When a new batch of specimens is to be used, it is necessary to first test a representative number of C-rings from the batch with Mattsson's solution.

12.2 Mattsson's Solution test must produce cracking of the test specimens before any further testing is carried out.

12.3 If cracking is not produced during the prescribed Mattsson's solution test the following variables need to be verified before rejecting the batch of C-rings.

12.3.1 Specimen material hardness shall exceed 70 HR 30T (Rockwell scale).

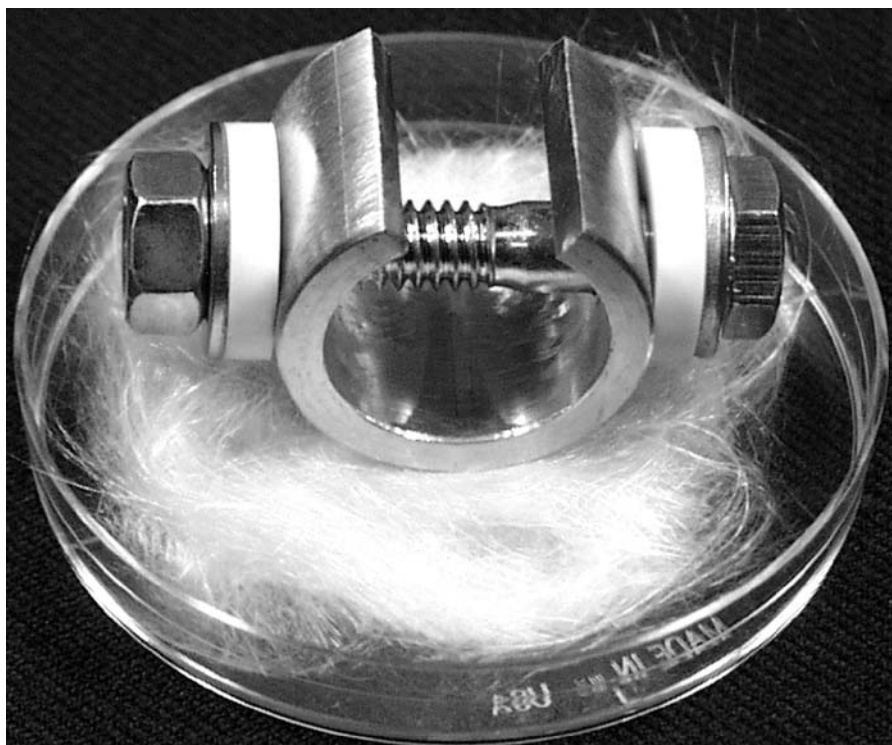


FIG. 2 C-ring Specimen Ready for Exposure to Test Solution

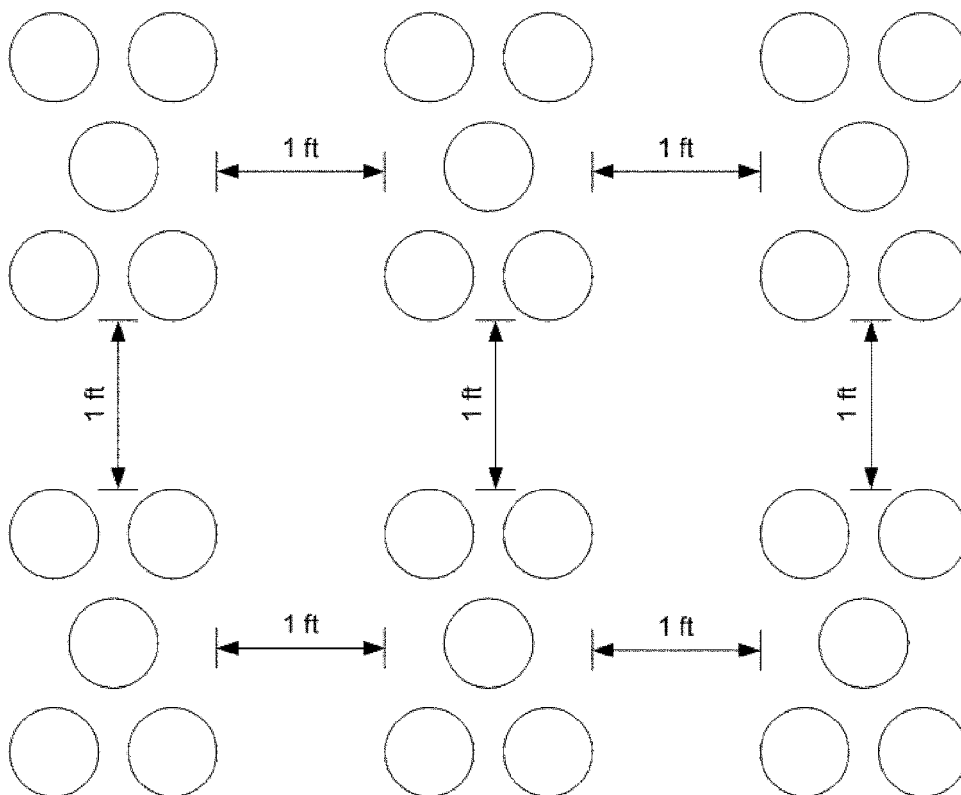


FIG. 3 Arrangement and Spacing of Specimens for Multiple Solution Testing

12.3.2 Strain should be checked with a strain gauge if hardness is sufficient.

12.3.3 Mattsson's solution should be checked to make sure it conforms to Practice G37.

12.4 Follow steps in **Annex A1** to test the C-rings in Mattsson's Solution.

13. Air Conditions

13.1 *Temperature*—Air temperature shall be maintained in a range of 70 to 80°F (21 to 27°C) throughout the entire test duration.

13.2 *Relative Humidity*—Percent relative humidity of the air shall be maintained in a range of 15 to 60 % throughout the entire test cycle.

13.3 Air Circulation:

13.3.1 Air circulation is considered to be very important to testing since it affects the rate at which the solutions lose water by evaporation. Optimum conditions for air circulation have not been established, but recommendations described in **13.3.2** shall be considered.

13.3.2 The most important consideration is to achieve moderate evaporation of the test solutions in a 2-3 day time period.

13.3.3 Stagnant air conditions should be avoided.

13.3.4 Testing in a ventilated enclosure may be required to achieve adequate circulation.

NOTE 3—Care should be taken to avoid overnight and weekend changes in operation of the laboratory heating, ventilation, and air conditioning equipment which could result in systematic excursions outside the desired temperature, humidity, and air circulation ranges.

14. Procedure

14.1 Leak detector fluid and control solutions are tested concurrently. No Mattsson's solution tests shall be conducted while control or test solutions are in progress.

14.2 Stressing of the C-rings:

14.2.1 The specimens shall be stressed the same way for exposures in the leak detector fluid and control solutions.

14.2.2 Individual specimens should be assembled as shown in **Fig. 1**.

14.2.3 Constant strain stressing method shall be used in stressing the C-rings. Tensile stress will be introduced on the exterior of the ring by tightening the bolt centered on the diameter of the ring.

14.2.4 Tighten the restraining bolt and nut until the deflection required to produce a total strain of 0.65% on the outer fiber of the C-ring specimen is achieved.

14.2.5 The required deflection can be determined from **Table 1**, which is based on the outer diameter and wall thickness of the original unstressed C-ring or the formula given in **Appendix X1**.

14.2.6 Measure the deflection of the C-ring across the faces perpendicular to the bolt as seen in **Fig. 4**.

14.3 Assembly of Individual Exposure Dishes:

14.3.1 Make sure individual dishes are clean of any foreign matter.

14.3.2 Cut swatches of glass fiber wick about 1/8 in. (3 mm) thick to fit the bottom of the dishes.

14.3.3 Place the layer of glass fiber wick across the bottom of the individual dishes.

14.3.4 Place the C-rings inverted into the individual dishes. The apex of the C-ring should be resting on the wick material at the bottom of the dish (**Fig. 2**).

TABLE 1 Deflection for Stressing C-rings

O.D. (inch)	Wall Thickness (in.)				
	0.065	0.08	0.091	0.125	0.25
1	0.073	0.059	0.051	0.036	0.015
1 1/16	0.083	0.067	0.058	0.041	0.018
1 1/8	0.094	0.075	0.065	0.046	0.020
1 3/16	0.105	0.089	0.073	0.052	0.023
1 1/4	0.116	0.083	0.081	0.057	0.026
1 5/16	0.129	0.099	0.090	0.064	0.028
1 3/8	0.142	0.114	0.099	0.070	0.032
1 1/2	0.169	0.136	0.119	0.084	0.038
1 5/8	0.184	0.148	0.129	0.092	0.042
1 3/4	0.199	0.160	0.140	0.100	0.046
1 7/8	0.232	0.187	0.163	0.116	0.054
1 7/8	0.267	0.215	0.188	0.134	0.062
2	0.304	0.245	0.214	0.153	0.072

14.3.5 The exposure dishes shall be laid out in such a pattern that there is at least 1 ft (305 mm) separation between groups of specimens (**Fig. 3**). The control solution specimens shall be located at the center of the exposure dish cluster.

14.4 Exposing the Specimens:

14.4.1 Start exposure by temporarily removing the individual C-ring specimens and add 10 mL of leak detector fluid and control solution to corresponding exposure dish.

14.4.2 Replenish LDF solutions (wetting cycle) in each of the corresponding exposure dish by adding an additional 10 mL every 2 to 3 days (evaporation cycle) (**Fig. 5**).

14.4.3 Continue to add the leak detector and control solutions for a total of 15 cycles (including initial addition) or until specimen cracks.

14.4.4 At the end of the 15 cycles examine the individual C-rings for cracks. If no cracks are detected continue exposure of the C-ring for an additional 7 days with no further test solution additions.

14.5 Inspection of Specimens (during exposure):

14.5.1 Specimens shall be visually inspected at least every 2 cycles prior to the next solution addition.

14.5.2 C-rings should be removed from their test assembly for visual inspection. A low magnification up to 50× may be required to identify fine cracks.

14.5.3 Do not remove dried solutions from surface of C-rings. If surface cannot be seen through dried solution films, continue testing until an obvious crack is present or test duration is reached.

14.5.4 If cracks are detected or the test duration is reached, testing is finished for that particular C-ring specimen.

14.6 Cleaning of Specimens (after testing):

14.6.1 Upon discovery of crack or at completion of testing, rinse the specimens thoroughly with distilled or deionized water.

14.6.2 Disassemble rinsed specimens.

14.6.3 Remove any remaining dried leak detector fluid solution with a soft bristle brush and water rinse.

14.6.4 Dry specimen after cleaning/rinsing.

14.6.5 Remove corrosion products if needed with acid cleaning as outlined in Practice **G1**. Use acid mixtures according to designations C.2.3 and C.2.4.



FIG. 4 Caliper Measurement of C-ring's Deflection

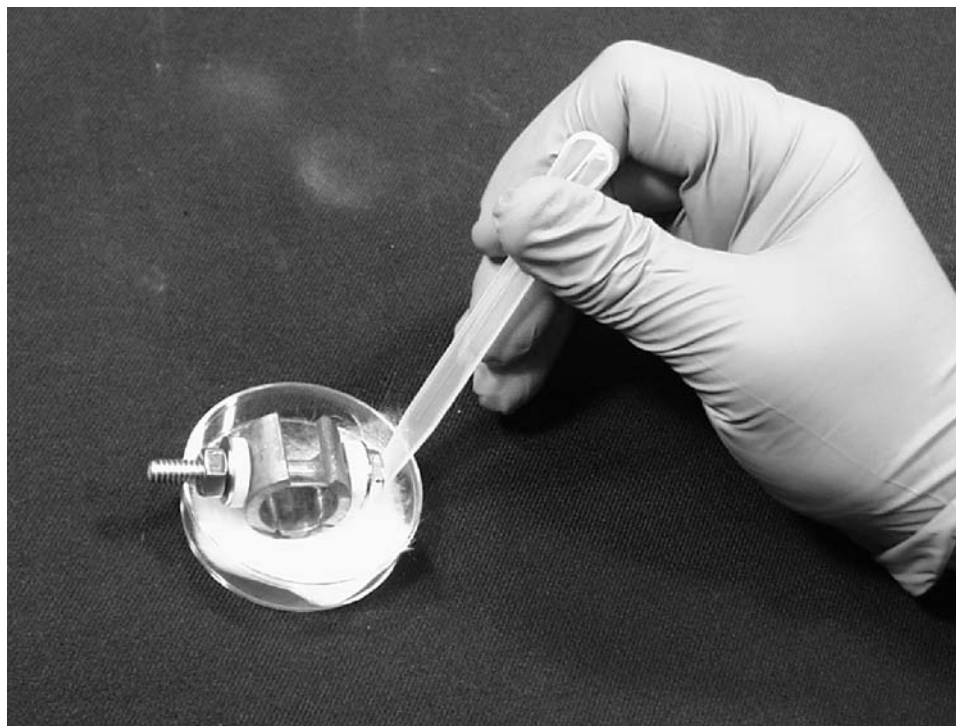


FIG. 5 Replenishment of Test Solution to Individual C-ring

15. Final Examination of Specimens

15.1 Macroscopic Examination:

15.1.1 Perform examination at a magnification of at least 25 $\times$ on all specimens after cleaning. Examine the entire bend section of the specimen. Stress corrosion cracking does not always occur at the apex.

15.1.2 The cleaned C-rings may need to be re-stressed in order to clearly detect any cracks.

15.2 Microstructural Examination:

15.2.1 Perform a microscopic examination on any specimen, if the presence of SCC is questionable.

15.2.2 Samples shall be prepared as outlined in Annex section [Annex A2](#).

15.2.3 Examine the entire outer circumference of the cross section for cracking. Perform examination at magnifications up to 500 $\times$.

16. Interpretation of Results

16.1 Criteria of Failure:

16.1.1 No cracks shall be detected on the C-ring specimens exposed to the control solution (water) after the full exposure period. If there are cracks observed within the test period, it indicates that there has been contamination by either the other test solutions or from something in the surrounding air. The test is invalid in this case, and a retest is required.

16.1.2 When the test is valid according to section 16.1.1, a C-ring specimen that exhibits cracking in the leak detector fluid solution shall be considered as a stress corrosion failure unless proved otherwise by the provisions of section 16.2 or 16.3.

16.1.3 An individual leak detector fluid solution shall be considered to have failed the test if one or more of the corresponding C-ring specimens fail due to stress corrosion cracking.

16.2 Macroscopic Examination:

16.2.1 Cracking should be clearly identifiable at magnifications less than 50X. Examples of such an occurrence are shown in Fig. 6.

16.2.2 If the presence of stress corrosion cracking is questionable, metallographic examination shall be performed on the cross sections to determine whether or not SCC has occurred.

16.3 Microscopic Examination:

16.3.1 A specimen that reveals cracking greater/deeper than two grains shall be considered as a stress corrosion failure. Examples of microstructure with SCC are seen in Fig. 7.

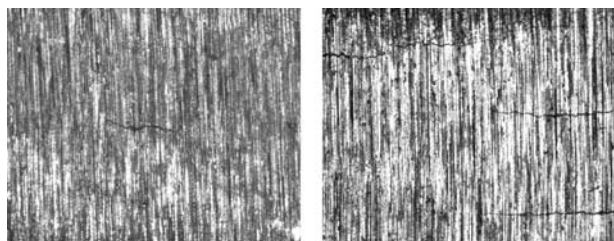


FIG. 6 C-ring Specimens Showing Clear Surface Cracks (Magnification 42X)

16.3.2 A specimen that reveals intergranular cracking, even when accompanied by transgranular cracking shall be considered as a stress corrosion cracking failure.

17. Report

17.1 Report the following:

17.1.1 Identity of laboratory where testing was performed.

17.1.2 Information that identifies the individual leak detector fluid test solutions.

17.1.3 Average, minimum, and maximum temperature and relative humidity of air conditions prior to each test solution addition (wetting cycle).

17.1.4 C-ring specimen dimensions and deflection used for testing.

17.1.5 Results of all tests, including individual crack times, number of specimens found cracked in each solution, examination method by which cracking was detected.

17.1.6 Crack time will be defined by the following equation:

$$CT = ETPI + (TBI/2) \quad (1)$$

ETPI = Total elapsed time to inspection prior to cracking.

TBI = Time between inspections when crack was detected and no crack observed.

CT = Estimated time to cracking.

17.1.7 Clear conclusion on whether solution passes or fails this test.

18. Precision and Bias

18.1 No information about either precision or bias of Test Method G186 is presented because the test result is not quantitative.

19. Keywords

19.1 brass alloys; bubble solutions; C-ring specimens; CDA272; gas-leak-detector solutions; leak detector solutions; soap solutions; stress corrosion cracking

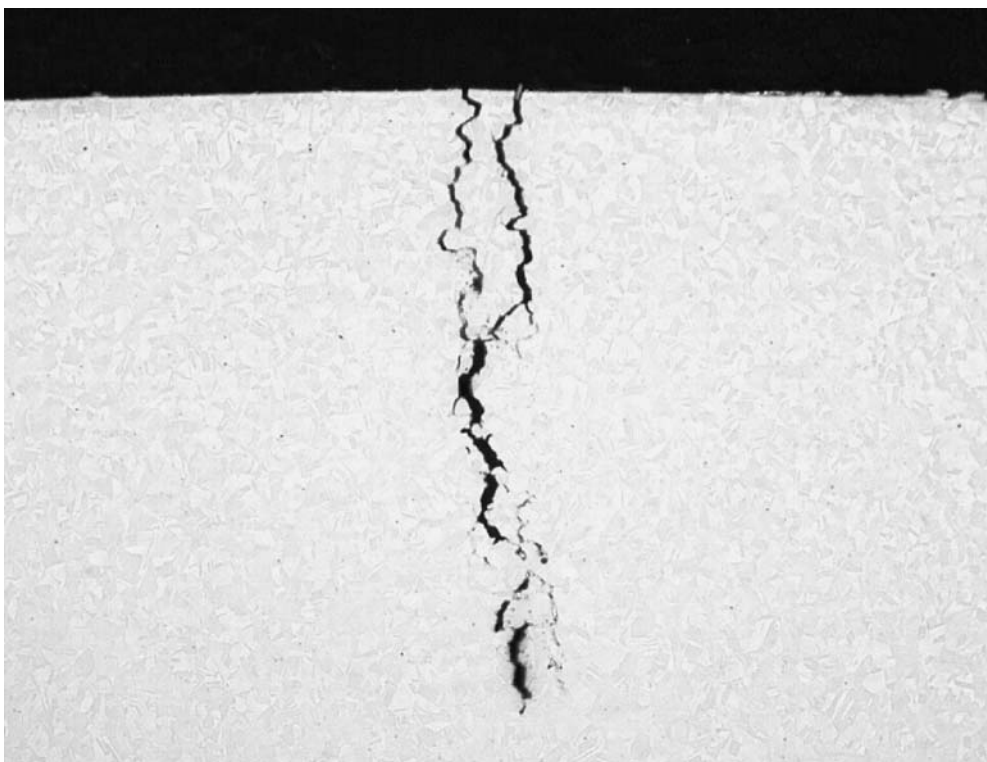


FIG. 7 Micrograph Showing Example of Intergranular SCC (Magnification 100×)

ANNEXES

(Mandatory Information)

A1. PROCEDURE FOR TESTING C-RINGS IN MATTSSON'S SOLUTION

A1.1 Prepare the C-rings and individual exposure dishes as described in sections 14.2 and 14.3.

A1.1.1 Randomly select C-rings from the batch of specimens.

A1.2 Exposing the Specimens with Mattsson's Solution

A1.2.1 The test using the Mattsson's solution should be performed separately and isolated from any of the gas-leak-detector or control solution No. 2.

A1.2.2 Start exposure by temporarily removing the individual C-ring specimens and add 10 mL of Mattsson's solution to each exposure dish.

A1.2.3 Replenish Control Solution No. 1 (wetting cycle) in each exposure dish by adding an additional 10 mL every 2-3 days (evaporation cycle).

A1.2.4 Continue to add the Control Solution No. 1 for a total four cycles (including initial addition) or until specimen cracks.

A1.2.5 Examine the specimens prior to each solution replenishment.

A1.2.6 All C-ring specimens should crack in Mattsson's solution before the end of the sixth cycle (Fig. A1.1).

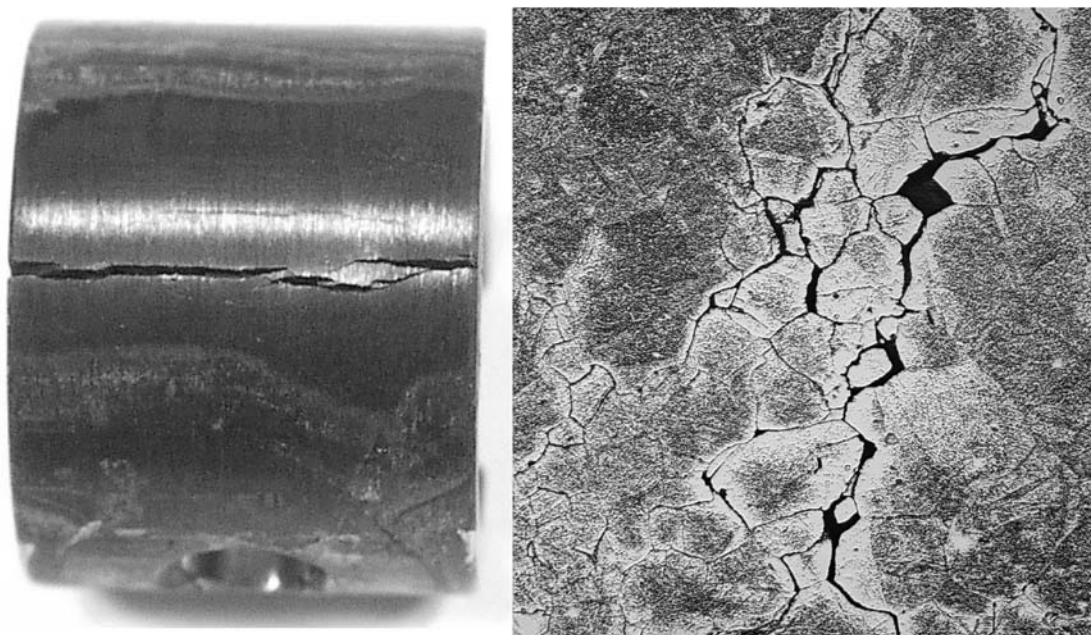


FIG. A1.1 Example of C-ring Exposed to Mattsson's Solution, A) Macro, B) Micro

A2. SECTIONING, MOUNTING, AND POLISHING OF SAMPLES

A2.1 The following steps are suggested for the preparation of metallographic cross section samples of the C-ring specimens.

A2.1.1 Cut off pieces of the C-ring where the bolt holes are located.

A2.1.2 Section the C-ring parallel to the circumference so as to make a perpendicular cut to any suspected cracks.

A2.1.3 Mount the circumferential cross section with the cut face down.

A2.1.4 Grind and polish the mounted cross-sections to at least a 4 $\mu\text{in.}$ (0.1 μm) finish.

A2.1.5 Thoroughly clean the polished surface so as to remove any residual polishing liquid or lubricant.

A2.1.6 Metallographic samples may be etched to detect path of cracking.

APPENDIX

(Nonmandatory Information)

X1. EQUATION FOR STRESSING C-RING SPECIMENS

X1.1 Calculate the final deflection, D_f , required to give the desired target total strain (0.65%) on the outer fiber of the C-ring specimen using the following equation:

$$D_f = 0.0065\pi d(d-t)/4t \quad (X1.1)$$

D_f = final deflection of the C-ring across the bolt holes, in.
 d = original outside diameter of C-ring before stressing, in.
 t = wall thickness of C-ring, in.

where:

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