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INTERNATIONAL

Designation: ~~G139 – 05 (Reapproved 2011)~~ G139 – 05 (Reapproved 2015)

Standard Test Method for Determining Stress-Corrosion Cracking Resistance of Heat-Treatable Aluminum Alloy Products Using Breaking Load Method[1]

This standard is issued under the fixed designation G139; 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 procedures for evaluation of stress corrosion cracking (SCC) resistance by the breaking load test method, a concept which uses residual strength as the measure of damage evolution (in this case environmentally assisted cracking).

1.2 This test method covers specimen type and replication, test environment, stress levels, exposure periods, final strength determination, and statistical analysis of the raw residual strength data.

1.3 The test method was developed for use with heat-treatable aluminum alloys, that is, 2XXX alloys and 7XXX with 1.2 to 3.0 % Cu, and test specimens oriented in the short-transverse direction relative to grain structure (**1, 2**).[2] However, the residual strength measurements and the statistics used to analyze the data are not specific to heat-treatable aluminum alloys and can be used for other specimen orientations and different types of materials.

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

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2. Referenced Documents

2.1 *ASTM Standards:*[3]

E8 Test Methods for Tension Testing of Metallic Materials
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
G44 Practice for Exposure of Metals and Alloys by Alternate Immersion in Neutral 3.5 % Sodium Chloride Solution
G47 Test Method for Determining Susceptibility to Stress-Corrosion Cracking of 2XXX and 7XXX Aluminum Alloy Products
G49 Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens
G64 Classification of Resistance to Stress-Corrosion Cracking of Heat-Treatable Aluminum Alloys

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3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *censor*—a statistical term indicating that the value from an individual observation may fall outside of the range that can be measured because of test procedures or conditions.

3.1.2 *sample*—the nominally uniform, bulk material from which individual stress-corrosion cracking specimens are obtained.

4. Summary of Test Method

4.1 This test method describes a procedure for using residual strength after exposure to a corrosive environment to evaluate stress corrosion cracking susceptibility in heat treatable aluminum alloy product forms such as sheet, plate, extrusions, forgings, and bar. These products generally are most susceptible to SCC in the long transverse direction of sheet, the short transverse

[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.

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[2] The boldface numbers in parentheses refer to the list of references at the end of the standard.

[3] 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.

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direction of plate, extrusions and forgings, and the transverse direction of rod and bar stock. In this test, tensile bars or direct tension sheet specimens, prepared according to Practice G49, are exposed to 3.5 weight % aqueous sodium chloride solution (Practice G44), are removed before they fail and are tension tested to determine the amount of corrosion damage that has occurred. The average retained strength is then calculated and the Box-Cox Transformation can be used for statistical analysis of the results.

4.2 The procedure calls for exposure of unstressed specimens which are used to factor out the effects of pitting, intergranular, and general corrosion. These phenomena degrade residual strength but do not require applied stress for their occurrence.

5. Significance and Use

5.1 The test method was developed for use with high strength aluminum alloys (2XXX and Cu containing 7XXX) that are normally tested in 3.5 weight % NaCl by alternate immersion. However, the concept which uses residual strength as a measure of damage evolution (in this case environmentally-assisted cracking) can, in principle, be applied to any alloy and environmental system.

5.2 This test method has been developed for research studies of alloys and tempers with improved resistance to SCC. The test results permit different material variants to be compared with a high degree of confidence and with much more precision than the results of pass/fail tests. Thus, it is particularly useful for comparing materials with similar levels of resistance to stress-corrosion cracking. The procedure could be modified for use as a quality assurance tool but this has not been a primary purpose during its development.

5.3 The exposure periods and conditions that are described in this test method apply specifically to high strength aluminum alloys, but the statistical techniques should be valid for other alloy systems with different exposure conditions.

5.4 Although this particular procedure was primarily intended for testing products in the short-transverse stressing direction, it is useful for other stressing directions, particularly the long-transverse direction in sheet and thin plate products.

5.5 Determination of the actual serviceability of a material requires stress-corrosion testing performed in the intended service environment, under conditions relating to the end use, including protective measures such as coatings and inhibitors and is outside the scope of this test method.

5.5.1 There is no good way to compare test environments to actual service because most service environments have large inherent variability with respect to a single structure that may experience many different environments or with respect to two identical structures that serve in different locations. Unless a sample can be tested in the actual service environment for the expected life of the component, no conclusive determination can be made about the suitability of a particular material for a particular application. Designers must therefore make judgments on the suitability of particular materials for applications based on knowledge of the material and of the service environment. To avoid service failures, the environment used for preliminary evaluations is often chosen based on a worst case scenario leading to intentional overestimations of corrosion damage.

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6. Interferences

6.1 The breaking load test factors out pitting corrosion that occurs in environments such as the 3.5 % NaCl solution used in alternate immersion testing per Practice G44. The primary concern in using the breaking load test is choice of appropriate exposure stress. If the exposure stress is too low no damage will accumulate. On the other hand, if the applied stress is too high many of the specimens will fail before the end of their scheduled exposure periods. The statistical procedures included in this test method can accommodate small numbers of failed specimens but not large numbers.

6.2 The breaking load test is applicable to specimens that have been exposed in natural and service environments. However, conditions in these environments may not be constant so consideration must be given to the period and timing of exposure to avoid biasing results. For example, environmental conditions that vary seasonally such as temperature, moisture, and pollutant concentration may affect the corrosivity of outdoor exposure stations. Direct material comparisons should be made using identical environmental conditions.

6.3 Some care is required when comparison samples have different original (uncorroded) tensile strength and fracture toughness values. Large variations in initial properties can either reduce or increase the apparent differences in SCC performance of the samples. To avoid bias due to tensile properties, the statistical procedures incorporated in this test method are based on percentages of original strength. However, to examine the effect of fracture toughness, which affects residual strength, a flaw size calculation must be done using fracture mechanics techniques (**3**).

7. Test Specimens

7.1 The breaking load procedure may be conducted using any specimen that can be axially stressed in a fixture that will sustain an applied displacement. However, results obtained using different specimen geometries or stressing methods can not be directly compared. While the relative susceptibilities of the samples will not be changed, the absolute numbers can be quite different.

7.2 Whenever the geometry of the metal sample permits, the test should be conducted using smooth, round tension specimens prepared in accordance with Practice G49. In the case of sheet and other products that may be too thin to yield tensile bars, sheet tensile specimens may be used. The test sensitivity increases with the ratio of surface area to volume in the specimen gage section; however tests made using round tensile specimens have shown that the same relative rankings can be achieved with different size specimens (**1**).

8. Exposure Procedure

8.1 *Stressing Procedure and Exposure Conditions*—The specimens shall be stressed by axially loading in constant deflection-type fixtures as in Figure 1 of Practice G49 and exposed to the 3.5 % NaCl alternate immersion test per Practice G44. The number of specimens for each stress level/exposure time combination should be a minimum of three; five or more are preferable.

8.2 *Stress Level*—The minimum number of stress levels is two, one of which is a complete set of specimens exposed with no applied stress. For samples with unknown SCC resistance it is preferable to start with two or three stress levels in addition to the unstressed specimens. The unstressed specimens allow the damage caused by general, pitting and intergranular corrosion to be calculated and separated from damage caused by the applied stress. The other stress level(s) must be chosen for each individual sample by considering the expected performance of the sample. The more SCC resistant the sample, the higher the stresses should be. The ideal maximum stress would be one that leads to significant damage by way of cracking but does not cause more than a few specimens to actually break into two pieces before the end of the scheduled exposure period (**2**). One stress level can be used but the statistical calculations only evaluate the performance of the sample at that stress level. In other words, there is no good way to extrapolate and estimate performance at higher or lower stress levels without actually conducting the test.

8.3 *Exposure Time*—This parameter must be adjusted for the sample to be tested and the size and orientation of the test specimens. In general, two to four time periods (plus zero days with no stress) should be used with the maximum time being approximately ten days for short transverse tests on 2XXX and 7XXX alloys. In general, long-transverse specimens and more resistant alloy systems (such as 6XXX alloys) should be exposed for longer periods. Classification G64 gives time periods for these situations which can be used to estimate a reasonable maximum exposure time.

NOTE 1—For material variants with unknown SCC performance in the test environment, it is advisable to test a limited number of pass/fail specimens according to the procedures in Test Method G47. This will provide guidance for choosing appropriate stress levels and exposure times for the sample. This can prevent the expenditure of large amounts of time and money for specimens that do not provide information with significant value.

8.4 *Determination of Residual Strength*—Upon completion of each exposure period, a set of specimens should be removed from test, rinsed, unstressed, and tension tested in accordance with Test Method E8. It is recommended that tensile testing be completed on the day the specimens are removed from exposure. If a time delay between completion of exposure and tensile testing is unavoidable, the specimens must be thoroughly rinsed with deionized water, stored in a desiccated environment, and the delay period should be recorded. The breaking strength must be calculated and recorded for each test specimen.

8.5 The residual strength data can be used to show trends between samples by simply calculating average residual strength for each stress/time combination as shown in Fig. 1. However, statistical procedures must be used to evaluate whether the trends are

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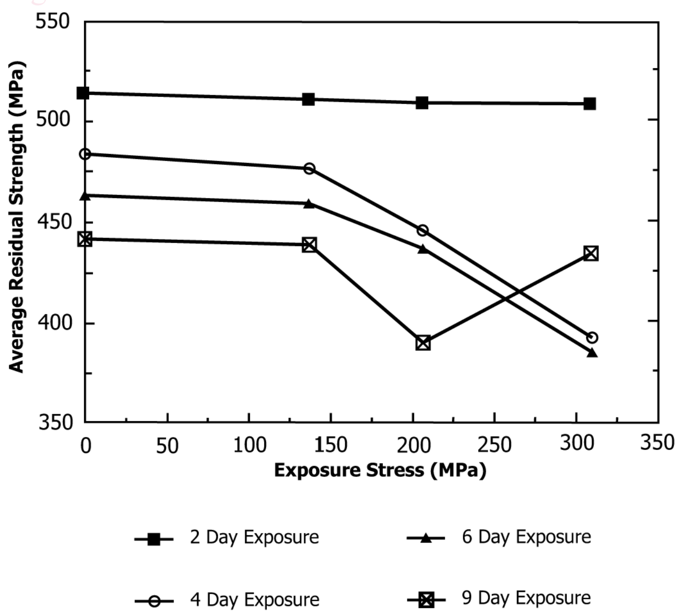

NOTE 1—Some specimens in this set did fail before the end of their scheduled exposure periods, but these failed specimens have not been included in the averages. The averages represent only specimens that survived to be tensile tested. The upturn in the nine-day data at 310 MPa is caused by not including failed specimens.

FIG. 1 Plot of Average Residual Strength Values for a Representative Data Set (one laboratory)

real or merely data scatter.

8.5.1 During the development of the breaking load test method, the variance of data within individual cells (a single sample/stress/time combination) has been shown to increase as resistance to SCC decreases. This tendency for variance to increase with decreasing residual strength means that the ability of the breaking load test to resolve differences between cells can be much greater for the better performing cells than the poorer performing cells. Therefore, plots of average residual strength can be very misleading.

9. Statistical Analysis—Box-Cox Transformation

9.1 Breaking load data can be statistically analyzed by following the steps outlined here. There are undoubtedly other procedures that will work but the Box-Cox transformation has demonstrated its usefulness for situations in which variance is not constant throughout the data set (**4, 5**). In the case of stress corrosion cracking data, as residual strength decreases, variance generally increases. The following procedure assumes that a fixed number of specimens have been tested for each material variant, exposure period, and exposure stress. Some of these values will be left-censored, that is, some specimens will fail before they complete their scheduled exposure period. For such specimens the breaking load value is known to be less than or equal to the exposure stress but this procedure includes a statistical method for estimating the values of those data points.

NOTE 2—Appendix X1 contains a sample Box-Cox calculation that follows the procedure described in this section of the test method.

9.2 Transform the original values, X, by means of the preliminary transformation

$$X_{tr} = \left(\frac{X}{X_O}\right) 100 \tag{1}$$

where X_O is the average breaking load for no exposure for the given material variant. This transformation expresses the percent retention of original strength for each specimen, and thereby normalizes the residual strength of different materials.

9.3 The Box-Cox parameters are determined using all data that have been generated simultaneously for relatively similar samples. For example, when testing several samples from one alloy that have been produced using various fabricating routes or are in different tempers, all data should be considered in determining the following parameters. This would also apply to alloys from the same system. On the other hand, alloys that react differently to the test environment should be considered separately. This would be the case for comparisons of 6XXX versus 2XXX alloys, for example.

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9.3.1 For all data cells with more than one observed value (that is, noncensored value), calculate the average, m, and the standard deviation, s. Plot ln(s) versus ln(m), and determine the slope, α, of the best fit straight line. The parameter λ in the Box-Cox transformation:

$$Y = C\,X_{tr}^{\lambda} - 1 \tag{2}$$

is 1 − α.

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9.3.2 The constant C can be chosen in any way that gives numbers of convenient size. One convenient choice is:

$$C = \frac{100}{X_{tr}^{\lambda}{,}max} \tag{3}$$

where $X_{tr,max}$ is the maximum value for X_{tr} among the noncensored values in the data set. This gives numbers in the range from 0 to 100, which is the same range as the values of X_{tr}.

9.4 Generate statistically plausible values for the censored observations, representing the failed specimens, by uniform random number generation over the interval (O, Y_c), where Y_c is the transformation of the censoring value (that is, the exposure stress).

9.5 Analyze the complete, transformed data set using standard statistical techniques. A simple way of analyzing a set of data transformed to the Box-Cox metric is to find the averages and standard deviations of all cells in the data table. Since each cell has the same number of observations, the pooled estimate of the standard deviation for r cells is

$$s_p = \sqrt{\frac{(s_1^2 + s_2^2 + \ldots + s_r^2)(N - r)}{r(N - r - c)}} \tag{4}$$

In this equation, N is the total number of observations, r the number of cells, and c the number of censored values.

9.5.1 Then the smallest difference in the averages of two cells that is statistically significant, the so-called least significant difference or LSD, is

$$\text{LSD} = t_{\nu}^{*} s_p \sqrt{\left(\frac{2}{n}\right)} \tag{5}$$

This value can be used to compare two cells statistically to determine whether or not the data in the cells really comes from two populations with different means.

9.5.1.1 In this expression n is the number of observations per cell; the t-test coefficient, t_{ν}, depends on the significance level chosen, and the degrees of freedom, ν, are given by

$$\nu = N - r - c \tag{6}$$

For 95 % significance and $\nu \approx 100$, $t_\nu \approx 2$. As ν becomes small, the value of $t\nu$ increases; this increases the value of the smallest difference which will be considered significant. For exact values for t_ν, tables of student's t-distribution must be consulted; the correct value will represent a two-tailed t-test.

NOTE 3—The transformed LSD value(s) which has just been calculated applies to the entire data set over which the Box-Cox Transformation parameters were determined.

9.5.1.2 When comparing data sets which have been considered separately, one should first pool the estimated variances from the two sets. For example, if the data sets are called 1 and 2, with variance estimates s_1^2 and s_2^2 and degrees of freedom ν_1 and ν_2 respectively, the pooled standard deviation will, in general, be

$$s_p = \sqrt{\frac{\nu_1 s_1^2 + \nu_2 s_2^2}{\nu_1 + \nu_2}} \tag{7}$$

If both variance estimates are associated with the same number of degrees of freedom, the equation becomes

$$s_p = \sqrt{\frac{s_1^2 + s_2^2}{2}} \tag{8}$$

To compare two averages which are not associated with the same number of observations, n, the above expression for LSD is used, with $\nu = \nu_1 + \nu_2$ and s_p equal to the above expression for the pooled standard deviation.

9.5.1.3 A more elaborate statistical analysis of the data in this study can be based on the analysis of variance procedure.

9.5.2 A lower confidence limit for the mean value for any data cell can be calculated from the expression

$$\text{LCL} = m_{B-C} - \frac{t_\nu s_p}{\sqrt{n}}\ , \tag{9}$$

where m_{B-C} is the average Box-Cox transformed value and the t_ν value represents a single-tailed t-test and is not the same as the t_ν value used for the LSD above. For example, when a 99 % LCL is required and $\nu \approx 100$, the value of t_ν is approximately 2.36.

9.6 If desired, transform the LCL values back to either the X_{tr} or the original X metrics.

9.7 The results of the Box-Cox calculations can be used to present the data graphically as in Figs. 2 and 3.

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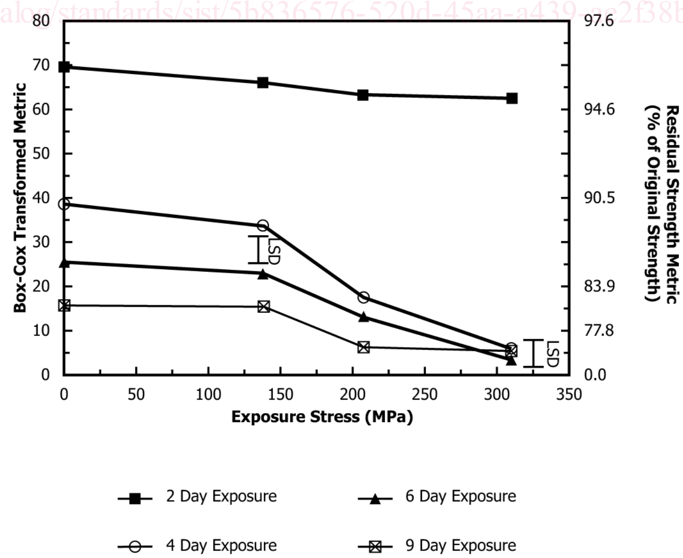
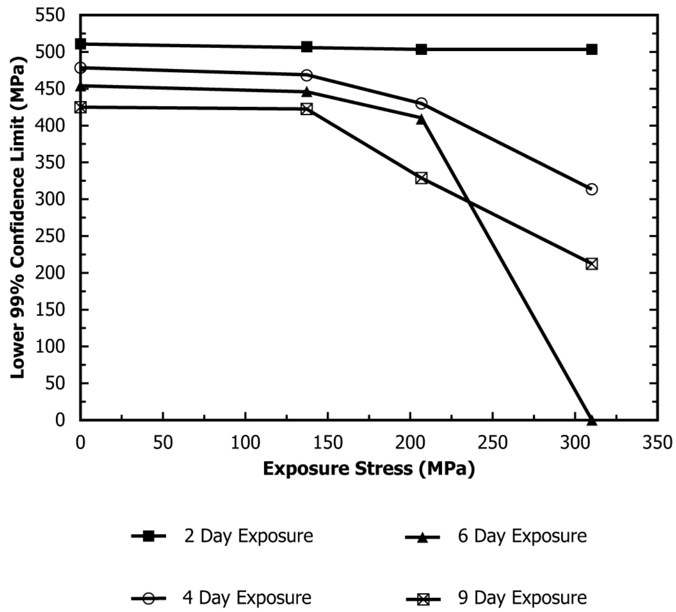


NOTE 1—In this case random values have been imputed for the failed specimens. Note the non-linear nature of the Box-Cox Metric (left Y-axis) as compared to the original metric (right Y-axis).

NOTE 2—The Box-Cox transformation makes variance approximately constant throughout the entire plot. The least significant difference (LSD) can be used to compare any two values to determine whether or not they are different with a given degree of confidence. Examples of this are shown on the graph; the four and six day 138 MPa values are indeed different while the four, six, and nine day 310 MPa values are all similar. Contrast this with Fig. 1 where the differences appear to be larger at the higher stress level.

FIG. 2 Plot of Averages in Box-Cox Transformed Metric (same data set as Fig. 1)



NOTE 1—This representation shows the stress/time combinations that cause significant SCC damage. From the LCLs the sample can be seen to perform very well at all stress levels during the two day exposure and at 138 MPa for the entire nine day period. However, stresses above 138 MPa and times longer than two days cause the residual strength of the material to decline more rapidly under an applied stress than under no applied stress. Determinations of the statistical significance of these results requires analysis of the LSD as shown in Fig. 2.

FIG. 3 Plot Showing Lower Confidence Limit (LCL) Values for Each Cell (from data plotted in Figs. 1 and 2)

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10. Interpretation of Results

10.1 Stress corrosion cracking test results are generally quite reproducible when the applied stress is either high enough to cause rapid failures of all specimens or so low that no damage is induced in the specimen. However, at intermediate stresses there is considerable variability in specimen performance. This variability becomes evident in pass/fail testing when some but not all specimens from a group fail. Using the breaking load procedure, the variance can manifest itself either as specimen failures or as large variance in measured residual strength. A large portion of this variability results from inhomogenities in the microstructure of heat-treatable aluminum alloys and is independent of test procedure.

10.2 Statistical results, such as the lower confidence limit and least significant difference, are intended to rank the stress corrosion cracking performance of different material variants for given environments, exposure periods, and applied stresses.

10.2.1 Because the statistical results are relative indicators of performance in a given environment, different laboratories may not obtain the same absolute values for similar samples. This is discussed in detail in the Statement on Precision starting in 12.1 of this test method.

10.2.2 These statistical results cannot be used to predict performance in other situations (especially other environments) unless a correlation has already been developed. For example, SCC performance of low-Cu and Cu free 7XXX aluminum alloys in natural environments cannot be predicted based on breaking load tests conducted in 3.5 weight % NaCl by alternate immersion (Practice G44) with any more accuracy than with traditional pass/fail approaches (Test Method G47). The reason is that the breaking load procedure does not compensate when the test environment correlates poorly with service environments.

11. Report

11.1 The following information shall be reported:

11.1.1 Identification of all samples, including alloy, temper, product form, thickness, and specimen location and orientation.

11.1.2 All raw data including original tensile strength, exposure time, stress level, and raw breaking strength of each corroded specimen. This is best done in tabular form using cells for each stress/time combination. The table shall note any specimens that failed before removal from test along with the day that the failure was detected. Whenever possible, it is advisable to report fracture toughness in the same orientation as the SCC cracks would propagate. For example, for rolled plate that has been tested using short transverse SCC specimens the most appropriate value would be S-L plane-strain fracture toughness (K_{IC}).

11.1.3 All calculated statistical quantities. The minimum would be average breaking strength and standard deviation for each data cell.

11.1.4 All deviations from the above procedure.