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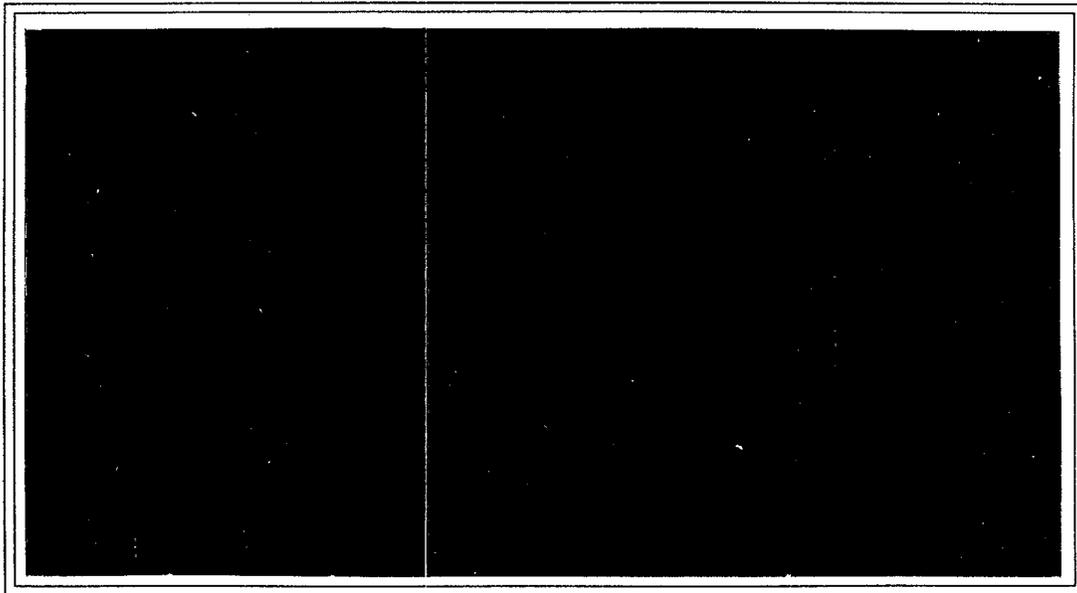
CLASSIFICATION UNCLASSIFIED	SYSTEM NUMBER 148400 
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TITLE
THE EFFECTS OF HARD ANODIZING ON THE STRESS CORROSION CRACKING OF ALUMINUM ALLOYS

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Technical Memorandum 94-126

**THE EFFECTS OF HARD ANODIZING ON THE STRESS
CORROSION CRACKING OF ALUMINUM ALLOYS**

by

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November 1994



Approved By:


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ABSTRACT

The breaking load method was used to determine the effects of alternate immersion in sea water on uncoated, sulphuric acid anodized and hard anodized specimens of AA 7075-T651 in order to evaluate the benefits of anodizing the aluminum pressure hull of an autonomous underwater vehicle to reduce the risk of stress corrosion cracking. The original test method showed that anodizing delayed the onset of stress corrosion cracking of specimens subjected to an externally-applied stress of 69 MPa, with hard anodizing providing a greater benefit than sulphuric acid anodizing. Anodizing was also shown to delay the onset of intergranular cracking in the absence of an external stress. The value of the threshold stress determined for the uncoated specimens was within the range reported by an interlaboratory test program.

The extreme value distribution was found to provide a good fit to the data, but its use did give rise to several problems previously noted in the interlaboratory program. A new statistical procedure involving Box-Cox transformations did alleviate these problems and served to confirm the conclusion reached using the original method, even though the statistical calculations may not have been strictly valid. The new procedure also showed that hard anodizing continued to offer improved resistance to stress corrosion cracking at higher stress levels. Finally, analysis of variance of the transformed data confirmed the conclusion ^{was} that anodizing provided a significant improvement in resistance to stress corrosion cracking, with hard anodizing providing the greater benefit.

1. INTRODUCTION

An autonomous underwater vehicle being designed and built for Canada's Department of National Defence will be subjected to repeated exposure to sea water during its testing and deployment. Its pressure hull will be constructed using a number of cylinders made from Aluminum Alloy (AA) 7075-T7351, an alloy that was originally developed for high strength applications in aircraft and which normally requires protection in severely corrosive environments such as sea water¹. The T7351 temper was developed to improve the resistance of this alloy to stress corrosion cracking (SCC) but cracking has still been observed in the laboratory environment². The original design did not call for any corrosion protection for the pressure hull but concerns about SCC led to the specification of a hard anodized coating for the aluminum alloy.

Some early research suggested that anodizing accelerated the SCC of aluminum alloys³⁻⁵ but Skoulikidis and co-workers⁶⁻⁹ demonstrated that a properly applied, sealed coating had a protective effect by increasing the time to failure of specimens loaded in tension. A new procedure for determining the susceptibility of aluminum alloys to SCC that does not depend on traditional time to failure methods has been developed by Alcoa Laboratories for the National Aeronautics and Space Administration. This procedure, known as the breaking load method¹⁰, appeared to have the ability to separate the effects of SCC from the damage caused by pitting and general corrosion. However, a recent interlaboratory test program aimed at assessing the reproducibility of the breaking load method revealed several shortcomings in the statistical procedure that was used¹¹. An alternate approach, based on Box-Cox transformations¹², was proposed and tested.

In order to evaluate the effectiveness of anodizing the pressure hull to reduce the risk of SCC, the Defence Research Establishment Pacific (DREP) has used both the original breaking load method and the proposed modification incorporating the Box-Cox transformation to determine the effects of alternate immersion in sea water on uncoated, sulphuric acid anodized and hard anodized specimens made from AA 7075-T651. The susceptibility of this temper to SCC has been extensively studied by traditional methods, to the point that it appears as an example in ASTM G47-79¹³ and was used in the interlaboratory test program¹¹. It was chosen for this study because tensile specimens were already available and there were severe time constraints associated with the decision on whether to specify an anodized coating for the hull components. Since this temper is more

susceptible to SCC than the T7351, it also served to highlight any improvements that anodizing might produce.

2. BACKGROUND ON THE BREAKING LOAD METHOD

The raw data for the breaking load method is obtained by measuring the "apparent tensile strength" of smooth tension specimens that have been stressed to pre-determined levels in a "constant strain" fixture and then exposed to the corrosive environment for set periods of time. The apparent tensile strength of each specimen is calculated using its original cross-sectional area without consideration for any thinning of the specimen due to general corrosion or stress corrosion cracking. This data provides an indication of the change in load carrying capability of the specimens as a function of exposure time and externally-applied stress. Reductions in the apparent tensile strength for stressed specimens beyond those found for unstressed specimens are assumed to be due to stress corrosion cracking.

Statistical analysis of the breaking load data is based on the assumption that the specimen breaks at its weakest point. If a specimen develops SCC, it may contain a wide spectrum of crack sizes but fracture will always occur at the largest crack. As a result, the distribution of all crack sizes within a group of specimens is not as important as the distribution of the largest cracks. The extreme value distribution pioneered by Gumbel¹⁴ applies to the largest cracks, assuming that the distribution of all crack sizes is exponential. These assumptions were supported by the work of Aziz¹⁵, who characterized pitting depth with an exponential distribution and showed that the maximum pit depth followed the related extreme value distribution. Since SCC is also related to the microstructure, the breaking load data for specimens suffering from SCC should follow an extreme value distribution as well. However, since fracture stress is related to the largest flaw size in an inverse manner, the appropriate distribution for a fracture stress would be an extreme value distribution of smallest values¹⁰. This distribution is bell-shaped but skewed to the left, so a group of replicate tests is likely to produce a wider range of fracture stresses below the most probable value than above.

The probability for survival, with survival defined as no failure at the stress of interest during the period of exposure, is expressed by the following equation, attributed to Gompertz¹⁶:

$$P = \exp(-e^z) \quad (1)$$

where P is the probability of survival and Z is the reduced variate, which has the form:

$$Z = (S - \hat{\mu}) / \hat{\sigma} \quad (2)$$

where S is the exposure stress and $\hat{\mu}$ and $\hat{\sigma}$ are estimates of the distribution location and scale parameters, respectively. These parameters, which are comparable to the mean and standard deviation of the normal distribution, are determined using the probability plotting method¹⁷. The breaking strengths of the replicate specimens in a test group are ranked in descending order and plotted against the expected value of each ranked observation. Specimens which broke prior to tensile testing are assumed to belong to the distribution, and hence affect the expected values, but are not available for plotting.

The expected value of the i^{th} observation is estimated by the R^{th} fractile of the distribution, where:

$$R_i = (i - \frac{1}{2}) / N \quad (3)$$

and N is the sample size. For the extreme value distribution of smallest values, the expected value of the i^{th} observation is:

$$E_i = -\ln[-\ln(R_i)] \quad (4)$$

If the data from a test group follows an extreme value distribution, a plot of E_i versus the ranked breaking strengths will yield a straight line. The slope, m , and intercept, b , of this line are determined by linear regression and provide estimates of $\hat{\mu}$ and $\hat{\sigma}$ for a large sample size, denoted by $\hat{\mu}_m$ and $\hat{\sigma}_m$, from the following equations:

$$\hat{\mu}_m = -\frac{b}{m} \quad (5)$$

$$\hat{\sigma}_m = -\frac{1}{m} \quad (6)$$

Before their use in Equation 2, these parameters must be adjusted to account for the small sample size employed in our experiments according to the following equations:

$$\hat{\sigma} = \hat{\sigma}_m (\sigma_m / \sigma_n) \quad (7)$$

$$\hat{\mu} = \hat{\mu}_n - \hat{\sigma}_n y_n + \hat{\sigma} \bar{y}_n \quad (8)$$

where n is the number of the N specimens which survived to be tensile tested, $\sigma_n = \pi/\sqrt{6}$, $y_n = 0.577$, and σ_n and \bar{y}_n are found in a table extrapolated from the work of Gumbel¹⁸ by Sprowls and co-workers¹⁹.

One of the advantages reported for the breaking load method is the ability to estimate the survival probability P (Equation 1) with reasonable confidence as the actual probability of survival for a resistant material approaches 1.0. Such a material normally displays very few failures in traditional time-to-failure methods. As a result, a prohibitively large number of specimens would be required to determine a value of P with reasonable confidence.

A more meaningful use of breaking load data can be obtained by calculating the tensile stress at which 99% of the specimens could be expected to survive a specified exposure stress for a specified length of time. This 99% survival stress (S_{99}) is determined using the following equation:

$$\begin{aligned} S_{99} &= \hat{\mu} + \hat{\sigma} \cdot \ln[\ln(0.99)^{-1}] \\ &= \hat{\mu} - 4.60 \cdot \hat{\sigma} \end{aligned} \quad (9)$$

and not only permits a direct comparison of stressed and unstressed specimen performances, but also provides a numerical value for comparing materials. Furthermore, the 99% survival stress can be used with fracture mechanics theory to estimate the equivalent flaw size that would not be exceeded in 99% of the SCC specimens that survived. A plot of this flaw size as a function of time could provide information about crack growth rates.

The 99% survival stress obtained for a given material at a specified exposure stress and length of exposure does not generally correspond to a threshold stress below which SCC would not be expected to occur within the specified exposure time. However, a series of S_{99} values can be used to obtain a statistically-defined threshold stress, S_{th} , such as the stress for which there is a 95% confidence of a probability of survival greater than 99%. This threshold stress is determined by plotting S_{99} against the exposure stress for each exposure time and calculating the intersection of each best fit line, obtained by linear regression analysis, with the line defined by S_{99} equalling the exposure stress. The mean, m , and standard deviation, s , of these

intersection points are calculated and then used to determine the lower confidence limit, LCL, from the following equation:

$$LCL = S_{th} = m - \frac{t_{n-1} \cdot s}{\sqrt{n}} \quad (10)$$

where n is the number of different exposure times (intersection points) and the value of t_{n-1} is obtained from a table of the cumulative t distribution²⁰ for a one-tailed t -test with 95% confidence. The value of S_{th} provides a single number which can be used to rate the behaviour of different alloys in the same environment or, in our case, the same alloy with different surface treatments.

3. BOX-COX TRANSFORMATIONS

A major problem with the original breaking load method that was identified by the interlaboratory test program is the extreme sensitivity of the value of the 99% survival stress to differences in the standard deviation of different test groups. Colvin and Emptage¹¹ showed an example of data from an identical test group (five specimens of AA 7075-T651 exposed to alternating immersion in 3.5% sodium chloride for 6 days at 138 MPa) from two different laboratories. From the number of specimens which failed prior to tensile testing, it was clear that one group had suffered more damage than the other. However, the calculated value of S_{99} was lower for the group with more survivors, simply because there was a greater variation between individual breaking strengths within that group. Furthermore, eliminating the worst performing specimens (those that broke prior to tensile testing) from the calculations of μ and σ biased the test results, leading to higher values of S_{99} for groups in which some specimens failed than would occur if those specimens had barely survived to be tested. Use of the extreme value distribution could also give rise to negative values for S_{99} , and hence for S_{th} , which are physically impossible. The obvious first approach of setting negative values of S_{99} to zero was shown to result in misleading conclusions. Finally, the statistical procedure could only be applied to the individual stress/time test groups. As a result, the method could not be used to obtain an overall estimate of the variance which would account for inconsistencies not related to resistance to SCC, such as machining finish and location within the exposure apparatus.

The Box-Cox transformation was proposed to address the problems encountered with the extreme value distribution. Successful implementation of the transformation is predicated on the assumption

that the variance increases in some manner with decreasing residual strength. This assumption seems reasonable for specimens which are experiencing stress corrosion cracking. Prior to the initiation of SCC, there should only be a small variation in the breaking strengths of individual specimens. However, as cracks grow, and the breaking strength decreases, there should be an ever increasing variation in crack lengths resulting from even slight differences in crack growth rates between specimens.

The first step in the procedure involves a preliminary transformation of the original breaking strength values, X , by the following equation:

$$X_{tr} = \frac{100 \cdot X}{X_0} \quad (11)$$

where X_0 is the average breaking load for no exposure for the given alloy or temper. The value of X_{tr} provides the percent retention of the original breaking strength and therefore standardizes the results for alloys with different uncorroded strength levels.

The average, m , and the standard deviation, s , are calculated for each individual stress/time test group with more than one specimen which survived to be tensile tested. The slope, α , of the best fit straight line is determined from the plot of $\ln(s)$ versus $\ln(m)$ for each different alloy or, in our case, different corrosive environment. This plot describes the nature of the relationship assumed to exist between the breaking strength and the variance. If the plot is not linear, the Box-Cox procedure cannot be applied to the system under investigation. If a meaningful value of α can be determined, the Box-Cox transformation is a power transformation which has the form:

$$Y = (100/100^\lambda) \cdot (X_{tr}^\lambda - 1) \quad (12)$$

where the power of transformation, λ , is equal to $1 - \alpha$. The constant, $100/(100)^\lambda$, gives rise to transformed values of Y in the range from 0 to 100, which is the same range as the values of X_{tr} .

As shown in Figure 1, the Box-Cox transformation essentially stretches the residual strength axis for large strength values and compresses the axis for small values. As a result, the transformation provides a means of discriminating between alloys with high levels of resistance to SCC. Such alloys would be difficult to separate using traditional pass/fail criteria. Another important effect of the

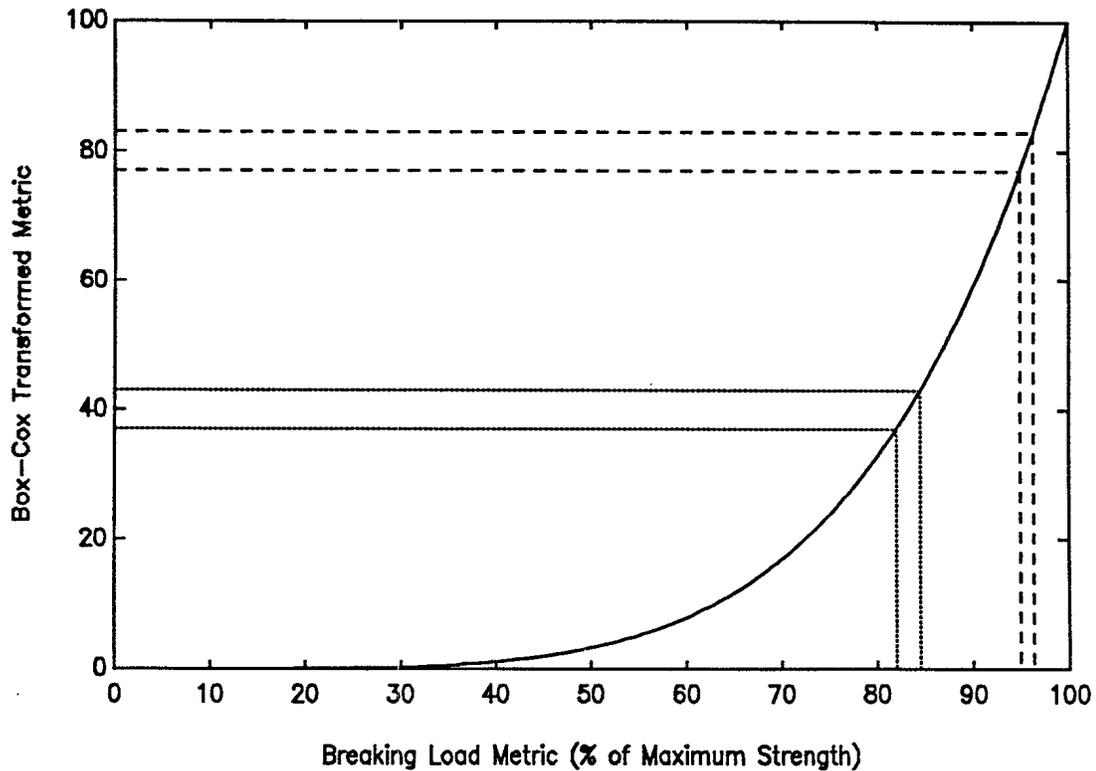


Figure 1. Box-Cox transformation curve for the example of $\lambda=5$. The dashed lines illustrate the transformation of the variance for one hypothetical test group while the dotted lines illustrate that for another test group with lower breaking strength and larger variance.

transformation is also illustrated in Figure 1. The variances of the individual stress/time test groups, which increase with decreasing mean breaking strength, are all approximately equal after transformation. Furthermore, the transformed data follows a normal distribution in many cases. As a result, standard statistical techniques, such as t-tests and ordinary analysis of variance²¹ (ANOVA), can be used. Since the variance is equivalent throughout the transformed Box-Cox metric, least significant differences and lower confidence limits are easily calculated.

Once the value of λ has been determined, statistically plausible values of Y are generated for the specimens which fail prior to tensile testing. A specimen which fails on the day it is scheduled to be tensile tested is assigned a value of Y equal to the transform of the exposure stress, Y_{exp} . A specimen which fails prior to the final day is assigned a value of Y obtained by generating a random number over the

interval $(0, Y_{exp})$. This procedure is simple, conservative and allows the use of the analysis of variance technique.

A simple way of analyzing a set of transformed data would involve the determination of the mean and standard deviation of each cell (stress/time test group) in the data table. Since each cell has the same number of "observations", the pooled estimate of the standard deviation, s_p is:

$$s_p = \sqrt{\frac{(s_1^2 + s_2^2 + \dots + s_r^2)(N-r)}{r(N-r-c)}} \quad (13)$$

where N is the total number of observations (specimens), r is the number of data cells and c is the number of specimens that failed prematurely. The smallest difference in the means of two cells that is statistically significant, the least significant difference or LSD, is given by:

$$LSD = t_v s_p \sqrt{\frac{2}{n}} \quad (14)$$

where n is the number of observations per cell, the degrees of freedom, v , are given by:

$$v = N - r - c, \quad (15)$$

and t_v is obtained from a table of the cumulative t distribution²⁰ for a two-tailed t -test with 95% confidence. The value of LSD can be used to compare two cells to determine whether or not the data in them comes from two populations with different means.

In order to compare cells from two different alloys or corrosive environments, the estimated variances from the two data sets must first be pooled. For data sets 1 and 2, with variance estimates, s_1^2 and s_2^2 , and degrees of freedom, v_1 and v_2 , respectively, the pooled standard deviation is given by:

$$s_p = \sqrt{\frac{v_1 s_1^2 + v_2 s_2^2}{v_1 + v_2}} \quad (16)$$

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

$$LCL = m_{BC} - \frac{t_v s_p}{\sqrt{n}} \quad (17)$$

where m_{BC} is the average Box-Cox transformed value and t_v is obtained from a table of the cumulative t distribution²⁰ for a one-tailed t -test. The LCL values can then be transformed back to either the X_{tr} or the original X metrics.

4. EXPERIMENTAL PROCEDURE

Round tension specimens with a diameter of 3.18 mm, a gauge length of 25.4 mm and an overall length of 50.8 mm were machined from a 76.2 mm thick plate of AA 7075-T651 in the short transverse orientation in accordance with ASTM Standard G49-76²². Chemical analysis of this plate is shown in Table 1. One group of eighty-five specimens was hard anodized under industrial conditions by the Ship Repair Unit (Pacific) according to U.S. Military Specification MIL-A-8625F Type III. A second group received a sulphuric acid anodized coating according to MIL-A-8625F Type II. A third group was left as-machined.

Tensile stresses of 0, 69, 138 or 207 MPa were applied to the specimens using a wedge-type, constant-strain frame as shown in Figure 2. The frames were assembled finger-tight and then placed in a commercially-available, synchronous loading device that was developed by Alcoa Research Laboratories²³. Operation of the device caused the inward movement of the wedge-shaped side pieces of the stressing frame, producing an uniaxial tensile stress in the specimen. This stress was related directly by Hooke's law and Young's modulus to the strain of the specimen, which was measured with an MTS model 623.13B-20 extensometer that had a 12.7 mm gauge length. Friction between the various components of a stressing frame maintained a constant strain after removal of the frame from the loading device.

Sets of five replicate specimens of each of the four stress levels for each of the three surface treatments were exposed to

TABLE 1. Chemical Composition of AA 7075-T651 Specimens

Element	Cu	Mg	Zn	Mn	Fe	Si	Cr	Ti	Al
Weight percent	1.39	2.45	5.92	0.06	0.15	0.09	0.2	0.03	rem

alternate immersion in sea water in accordance with the procedure described in ASTM Standard G44-75²⁴ for 2, 4, 7 or 10 days. A total of eighty specimens were exposed for each surface treatment. The specimens were continuously immersed in the sea water for 10 min of each hour and then allowed to dry for the remaining 50 min. This cycle was achieved by placing a pump on a timer. While the pump was operating, it maintained sufficient flow to ensure that all the specimens in a tank were immersed in sea water. When the timer shut the pump off, the tank was quickly emptied through a gravity drain. The sea water, which had a temperature of 10°C throughout the experiment, was obtained directly from the Strait of Juan de Fuca and used on a once-through basis.

Each surviving specimen was removed from the stressing frame after its scheduled length of exposure, rinsed in deionized water, dried and tension tested in an Instron 4204 tensile testing machine in accordance with ASTM Standard E8-89²⁵. The apparent tensile strength was then calculated on the basis of the original cross-sectional area. Finally, five specimens of each surface treatment, which were not stressed or exposed to sea water, were broken in the tensile testing machine to determine the initial breaking strength.

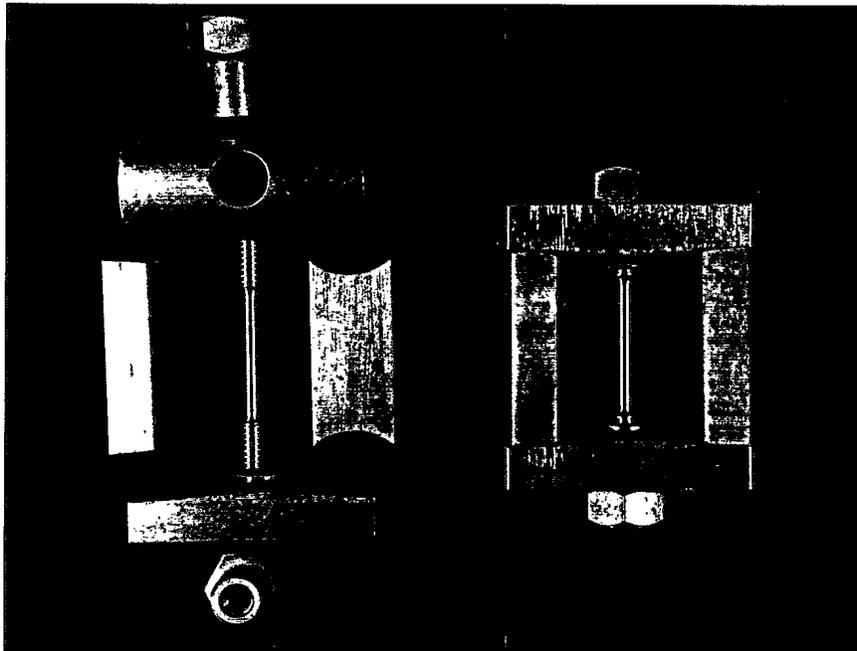


Figure 2. Exploded view of the components of the stressing frame, including the specimen, and the final stressed assembly.

5. RESULTS AND DISCUSSION

The residual breaking strengths of the uncoated, sulphuric acid anodized and hard anodized specimens exposed to alternate immersion in sea water are shown in Tables 2, 3 and 4, respectively. In these tables, a specimen that failed prior to tensile testing is marked by an "f" followed by the number of the day during which it failed or a "?" if that day was not determined. Ten uncoated specimens failed under the highest external load while no unstressed specimens failed, indicating that, as is already known, AA 7075-T651 is susceptible to stress corrosion cracking in sea water. Even without statistical analysis, the fact that only one hard anodized specimen failed suggests that hard anodizing improved the alloy's resistance to SCC. A total of five sulphuric acid anodized specimens failed, suggesting that sulphuric acid anodizing also had a beneficial effect, although not as pronounced as hard anodizing.

TABLE 2. Residual Breaking Strengths (MPa) of Uncoated AA 7075-T651 Specimens Exposed to Alternate Immersion in Sea Water

Stress	0 MPa	69 MPa	138 MPa	207 MPa
Time (days)				
0	518 514 513 502 491			
2	507 507 502 499 496	516 507 502 500 497	510 507 503 481 472	472 469 452 451 329
4	511 506 499 498 498	513 506 504 502 489	521 497 496 473 396	511 476 453 449 431
7	500 487 487 486 480	456 411 212 173 150	504 382 285 f? f?	f? f? f? f? f?
10	536 464 457 419 398	388 252 197 175 58	245 234 f? f? f?	f? f? f? f? f?

TABLE 3. Residual Breaking Strengths (MPa) of Sulphuric Acid Anodized AA 7075-T651 Specimens Exposed to Alternate Immersion in Sea Water

Stress	0 MPa	69 MPa	138 MPa	207 MPa
Time (days)				
0	509 502 500 497 481			
2	513 506 501 501 499	512 509 508 499 489	508 496 493 490 464	505 491 428 417 353
4	507 505 504 498 494	525 499 494 492 484	504 503 502 375 358	420 417 400 361 270
7	512 507 506 498 487	503 487 480 473 462	493 478 266 189 180	505 427 354 342 214
10	538 520 505 501 486	498 404 219 167 156	196 124 115 93 f?	222 f? f? f? f?

The calculated value of the 99% survival stress for each test group for each surface treatment is shown in Table 5, along with the correlation coefficient, r , of the best fit line determined for the plot of E_1 (Equation 4) versus the ranked breaking strengths. Specimens which were known to have failed during the last day of exposure were assigned a breaking strength equal to the externally-applied stress for these calculations. A sample calculation of S_{99} appears in Appendix 1. A value of $r=-1$ indicates that the data within each cell followed the extreme value distribution exactly. Visual inspection of the r values reveals that this distribution did provide a reasonable description of the behaviour of the specimens within a test group. However, the sample size was too small to completely rule out other distributions.

The effects of the exposure time and the externally-applied stress on the values of S_{99} are shown graphically in Figure 3 for

TABLE 4. Residual Breaking Strengths (MPa) of Hard Anodized AA 7075-T651 Specimens Exposed to Alternate Immersion in Sea Water

Stress	0 MPa	69 MPa	138 MPa	207 MPa
Time (days)				
0	510 509 504 504 498			
2	515 511 507 504 503	522 511 510 507 505	519 505 504 503 472	514 511 504 502 436
4	518 515 506 502 499	511 509 507 505 499	514 508 499 434 434	436 426 413 314 224
7	520 511 511 507 505	514 512 504 502 500	516 509 507 503 498	514 501 277 274 f?
10	514 506 499 499 495	507 504 504 489 460	522 422 332 237 210	479 327 237 219 207

uncoated specimens, Figure 4 for sulphuric acid anodized specimens and Figure 5 for hard anodized specimens. Comparison of these three figures does not reveal any substantial improvement in resistance to stress corrosion cracking as a result of either form of anodizing during the first four days of exposure to sea water. However, by the seventh day, an appreciable benefit from anodizing can be observed. Neither of the anodized groups showed any significant reduction in the value of S_9 , for specimens exposed for 7 days at 69 MPa, while the value for the uncoated specimens had dropped to -632 MPa, which indicated that only the uncoated specimens were suffering from SCC at that stress level and length of exposure. By the tenth day, the sulphuric acid anodized specimens at 69 MPa had begun to suffer from SCC as well. The hard anodized specimens did not experience SCC at 69 MPa for any length of exposure.

TABLE 5. Calculated Values of 99% Survival Stress, S_{99} , and Correlation Coefficient, r , for Each Test Group for Each Surface Treatment

	Stress	0 MPa	69 MPa	138 MPa	207 MPa
Time					
Uncoated AA 7075-T651					
0 days	S_{99} r	443 -0.979			
2 days	S_{99} r	475 -0.965	457 -0.916	393 -0.9532	51 -0.889
4 days	S_{99} r	463 -0.868	450 -0.969	190 -0.958	275 -0.928
7 days	S_{99} r	440 -0.879	-632 -0.889	-1260 -1.00	-7790 -
10 days	S_{99} r	131 -0.930	-497 -0.964	48 -1.00	- -
Sulphuric Acid Anodized AA 7075-T651					
0 days	S_{99} r	436 -0.971			
2 days	S_{99} r	467 -0.876	449 -0.979	394 -0.969	80 -0.978
4 days	S_{99} r	470 -0.971	394 -0.866	-33 -0.888	1 -0.957
7 days	S_{99} r	446 -0.986	391 -0.975	-666 -0.888	-257 -0.985
10 days	S_{99} r	394 -0.961	-698 -0.887	-339 -0.924	- -
Hard Anodized AA 7075-T651					
0 days	S_{99} r	478 -0.972			
2 days	S_{99} r	477 -0.921	468 -0.869	394 -0.937	286 -0.890
4 days	S_{99} r	459 -0.938	481 -0.990	224 -0.900	-180 -0.962
7 days	S_{99} r	474 -0.896	468 -0.922	467 -0.976	-1070 -0.894
10 days	S_{99} r	456 -0.907	373 -0.941	-438 -0.946	-475 -0.843

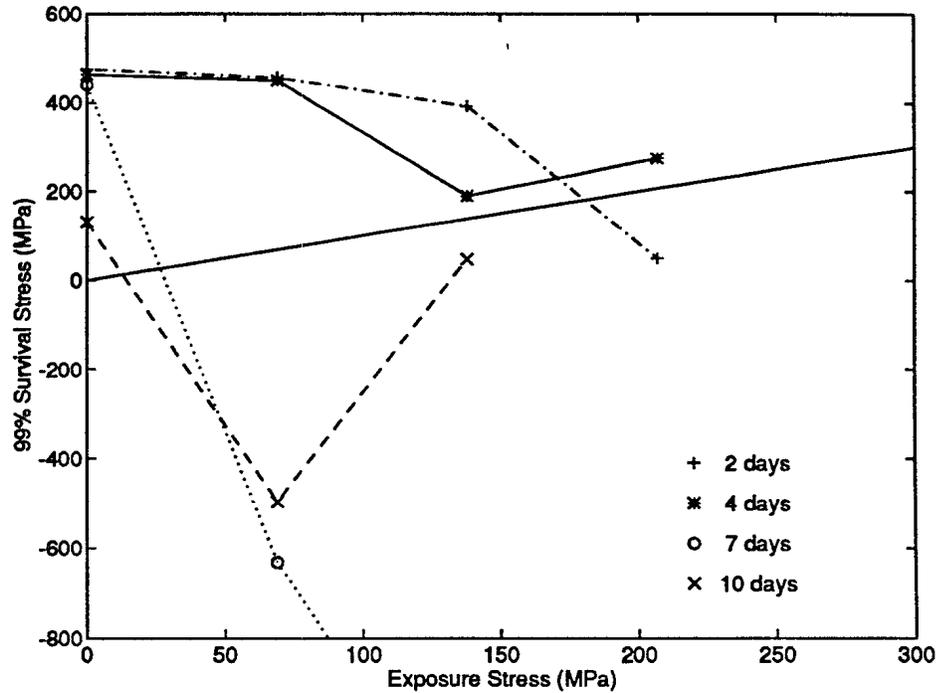


Figure 3. Plot of 99% survival stress versus externally-applied exposure stress for uncoated specimens of AA 7075-T651.

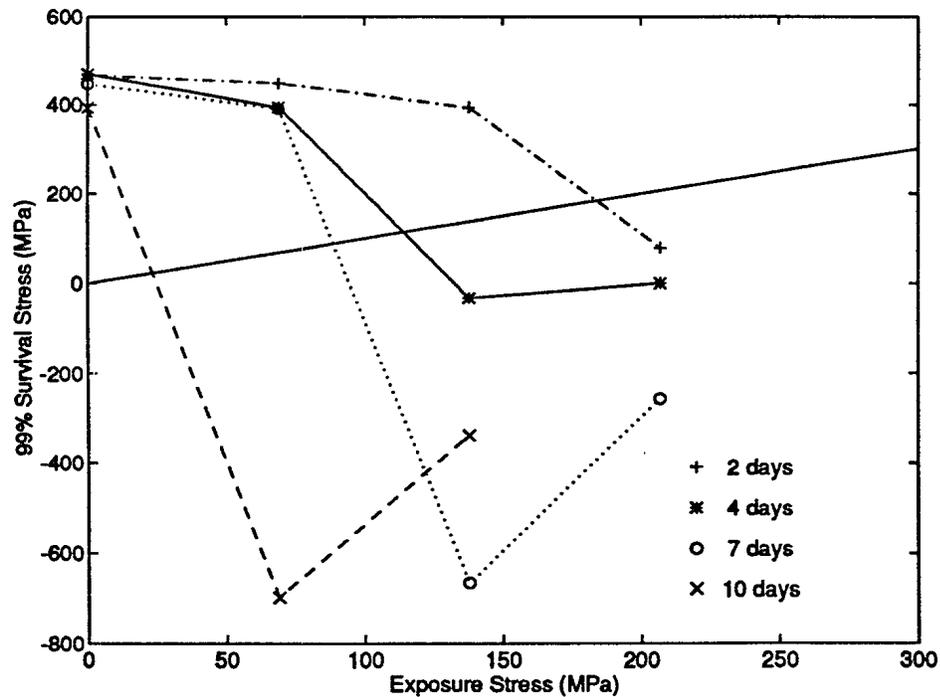


Figure 4. Plot of 99% survival stress versus externally-applied exposure stress of sulphuric acid anodized specimens of AA 7075-T651.

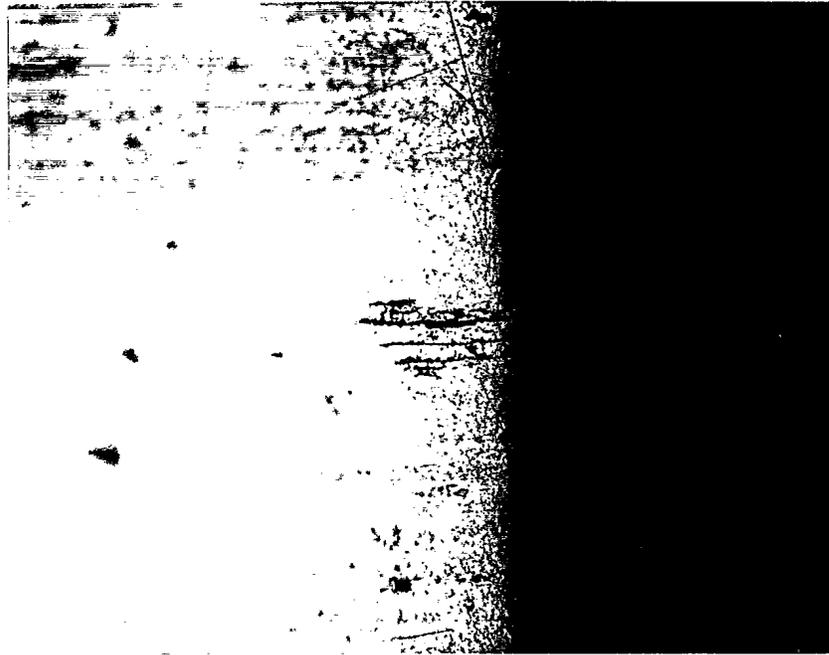


Figure 6. Intergranular cracking of uncoated AA 7075-T651 exposed to alternate immersion in sea water for 10 days. Etched with Keller's reagent. 250x.

the range reported by the interlaboratory test program¹¹, which varied from -119.5 MPa to 59.4 MPa. The ranges of the values for the different surface treatments do not overlap and lead to the conclusion that both sulphuric acid and hard anodizing improve the resistance of AA 7075-T651 to stress corrosion cracking, with hard anodizing providing the greater benefit.

As noted in the introduction to the Box-Cox transformation, successful implementation of the transformation is based on the assumption that the natural logarithm of the standard deviation of each individual stress/time test group increases linearly with decreasing

TABLE 6. Threshold Stresses for Each of the Three Surface Treatments

Surface Treatment	Threshold Stress (MPa)	
	Pessimistic	Optimistic
Uncoated	-47	-13
Sulphuric Acid Anodized	15	23
Hard Anodized	52	71

TABLE 7. Slope of \ln (Standard Deviation) versus \ln (Mean of Normalized Breaking Strengths), Correlation Coefficient and Power Parameter for Each of the Three Surface Treatments

Surface Treatment	Slope (α)	Correlation Coefficient (r)	Power Parameter (λ)
Uncoated	-2.02	-0.487	3.02
Sulphuric Acid Anodized	-1.78	-0.546	2.78
Hard Anodized	-6.99	-0.873	7.99

natural logarithm of the mean breaking strength. The correlation coefficients shown in Table 7 indicate a rather poor fit to the data for the uncoated and sulphuric acid anodized specimens. Nevertheless, the slopes did result in power parameters that served to stretch the residual strength axis for large strength values and compress the axis for small values. However, the transformed data may not follow a normal distribution or have variances for the individual stress/time groups that are approximately equal. As a result, calculations of the least significant difference and the analysis of variance for the uncoated and sulphuric acid anodized specimens may not be strictly valid.

The mean of the Box-Cox transformed data for each stress/time group for the uncoated specimens is plotted in Figure 7 as a function of the externally-applied stress. Similar graphs for the sulphuric acid anodized and hard anodized specimens appear in Figure 8 and Figure 9, respectively. Each graph also shows the value of the least significant difference, LSD, at the 95% confidence level for that surface treatment. These values were found to be 25.4 for the uncoated specimens, 28.2 for the sulphuric acid anodized specimens and 34.4 for the hard anodized specimens.

The Box-Cox transformation served to confirm several of the conclusions that were reached using the original breaking load method and removed the anomalous behaviour that was observed at higher stress levels as a result of the original statistical procedure. As suggested by the original method, a statistically-significant reduction in the breaking strength at the 69 MPa stress level occurred by the 7th day of exposure for the uncoated specimens, occurred between the 7th and 10th

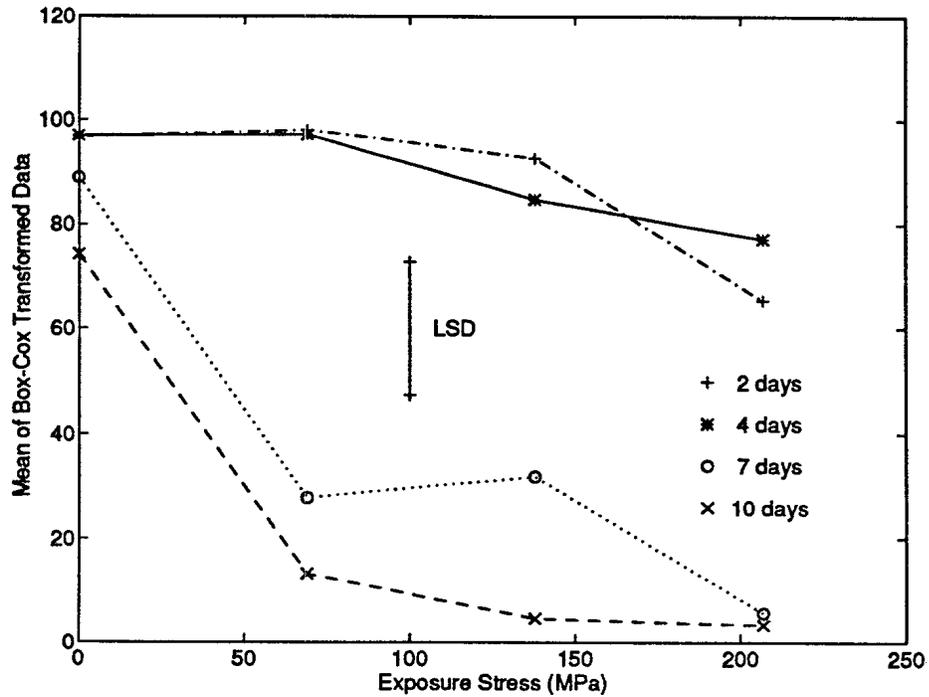


Figure 7. Plot of the mean of the Box-Cox transformed data for each test group versus exposure stress for uncoated AA 7075-T651.

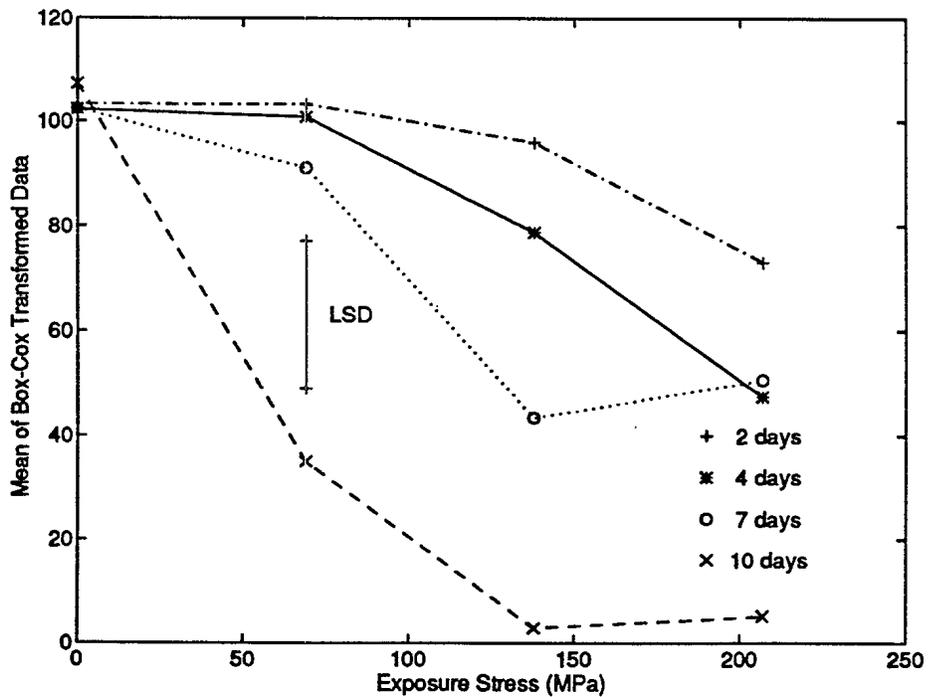


Figure 8. Plot of the mean of the Box-Cox transformed data for each test group versus exposure stress for sulphuric acid anodized AA 7075-T651.

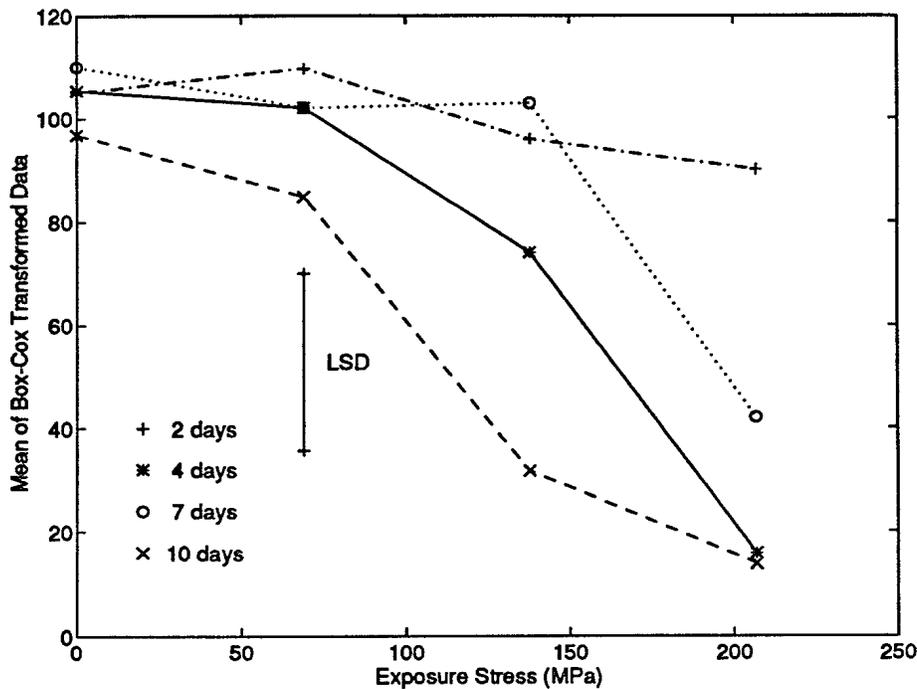


Figure 9. Plot of the mean of the Box-Cox transformed data for each test group versus exposure stress for hard anodized AA 7070-T651.

days for the sulphuric acid anodized specimens and did not occur at all for the hard anodized specimens. At the 138 MPa stress level, the Box-Cox transformation showed a significant reduction in the breaking strength by the 7th day for the uncoated specimens, by the 7th day for the sulphuric acid anodized specimens as well, and between the 7th and 10th days for the hard anodized specimens. The hard anodizing therefore continued to offer improved resistance to SCC at this higher stress level. At the 207 MPa stress level, the transformation showed a significant reduction in the breaking strength of the uncoated and sulphuric acid anodized specimens within two days of exposure that was not observed for the hard anodized specimens, again demonstrating the improvement provided by hard anodizing.

The behaviour of the 4 day, 207 MPa test group of uncoated specimens was assumed to be anomalous given the significant reduction in breaking strength already observed after 2 days at that stress level. Such results will occasionally occur as a result of the small sample size within each test group.

The difference between the means of the transformed data for

the 10 day, 0 Mpa group and the control group (0 days, 0 MPa) for the uncoated specimens was found to be 25.9, which was slightly greater than the LSD of 25.4, and indicated that the reduction in breaking strength was statistically significant. As shown in Figure 6, this reduction was caused by the onset of intergranular cracking. Any reduction in the breaking strength of the sulphuric acid or hard anodized specimens at 0 MPa was not significant and intergranular cracking was not detected on these specimens.

An analysis of variance table involving all three variables (stress level, exposure time and surface treatment) for the transformed data appears in Table 8. In this table, the four means appearing under the four different applied stress levels were calculated using all specimens that were tested under each load, independent of the length of exposure and the surface treatment. Similarly, the four means appearing under the four different exposure times involved all specimens for each exposure time, independent of the applied load and the surface treatment. Finally, the three means appearing under the three different surface treatments involved all specimens for each surface treatment, independent of the stress level and length of exposure. The error shown under the grand (overall) mean represents the standard error for each stress/time test group for each alloy. The standard error for each treatment mean appears in brackets beside that mean. The table shows that the addition of an externally-applied stress had a significant effect on the degradation of AA 7075-T651 specimens exposed to alternate immersion in sea water. Furthermore, the degradation increased with increasing level of stress. The table also shows that the degradation increased with each increasing length of exposure. Finally, anodizing is shown to provide a significant improvement in resistance to SCC, with hard anodizing providing a greater benefit than sulphuric acid anodizing.

Unfortunately, an overall table like Table 8 masks some of the differences in behaviour resulting from the different surface treatments. For example, the table indicates that an external load of 69 MPa resulted in a significant decrease in breaking strength. However, an ANOVA table for the hard anodized specimens by themselves shows that a load of 69 MPa did not significantly affect the breaking strength of these specimens (Appendix 3a). Significant SCC did not occur until the 138 MPa stress level. The extent of damage at the 69 MPa level on the uncoated specimens was so severe (Appendix 3b) that it caused the difference in the treatment means between 0 MPa and 69 MPa to be significant when all three treatments were combined in Table 8.

Table 8. Analysis of Variance Table for the Box-Cox Transformed Residual Breaking Strengths for All Three Surface Treatments

Stress	0 MPa	69 MPa	138 MPa	207 MPa	Grand Mean
Mean	99.2 (3.0)	80.5 (3.0)	61.6 (3.0)	40.8 (3.0)	70.5
Time	2 days	4 days	7 days	10 days	Error
Mean	94.1 (3.0)	81.9 (3.0)	66.6 (3.0)	39.4 (3.0)	10.3
Surface Treatment	Uncoated	Sulphuric Acid Anodized	Hard Anodized		
Mean	59.9 (2.6)	71.5 (2.6)	80.2 (2.6)		
Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F ratio	$F_{0.99}$
Mean	1	1190000	1190000		
Stress	3	113000	37600	71.2	3.95
Time	3	100000	33400	63.2	3.95
Surface Treatment	2	16600	8280	15.6	4.79
Stress-Time Interaction	9	28400	3150	6.0	2.56
Stress-Surface Interaction	6	9860	1640	3.1	2.96
Time-Surface Interaction	6	2460	4100	7.8	2.96
Three Factor Interaction	18	17900	994	1.9	2.19
Error	192	102000	529		
Total	240	1610000			

6. CONCLUSIONS

The breaking load method showed that, at a minimum, anodizing delayed the onset of stress corrosion cracking for specimens exposed to alternate immersion in sea water at 69 MPa, with hard anodizing providing a greater benefit than sulphuric acid anodizing. Anodizing was also shown to delay the onset of intergranular cracking in the absence of an externally-applied stress. Intergranular cracking was observed on the uncoated specimens after 10 days of exposure but not on any of the anodized specimens. The method also yielded values for a statistically valid threshold stress for each surface treatment that

supported the conclusion that both sulphuric acid and hard anodizing improve the resistance of AA 7075-T651 to SCC, with hard anodizing providing the greater benefit. The value of S_{th} for the uncoated specimens was well within the range reported by an interlaboratory program designed to test the reproducibility of the method and to evaluate alternative statistical procedures.

The extreme value distribution was found to provide a reasonably good fit to the data, but its use did give rise to several problems previously noted in the interlaboratory test program. The procedure involving Box-Cox transformations did alleviate these problems and served to confirm the conclusions reached using the original method, even though the statistical calculations may not have been strictly valid. It also showed that hard anodizing continued to offer improved resistance to SCC at higher stress levels.

Analysis of variance of the transformed data confirmed the conclusion that anodizing provided a significant improvement in resistance to SCC, with hard anodizing providing the greater benefit. Thus, the decision to hard anodize the aluminum in the pressure hull of the underwater vehicle has been supported by these experiments. Furthermore, the hard anodizing provides an excellent substrate for a paint system which would result in an even greater reduction in the possibility of unexpected failure due to stress corrosion cracking.

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APPENDIX 1. Sample Calculation of 99% Survival Stress

The ranked breaking strengths for the five uncoated specimens exposed to sea water for 4 days at 138 MPa are shown in the following table, along with the expected value of each ranked observation, E_i .

Observation i	$R_i =$ $(i-1/2)/5$	$E_i =$ $-\ln[-\ln(R_i)]$	Breaking Strength (MPa)
1	0.1	-0.834	521
2	0.3	-0.186	497
3	0.5	0.367	496
4	0.7	1.031	473
5	0.9	2.250	396

A plot of E_i versus the ranked breaking strengths yields a best-fit line with slope, m , equal to -0.0235, intercept, b , equal to 11.7, and correlation coefficient, r , equal to -0.958. The value of r near -1 indicates that the extreme value distribution provides a good fit to the data.

Initial estimates of the distribution location and scale parameters, $\hat{\mu}$ and $\hat{\sigma}$, are found to be:

$$\hat{\mu}_- = -\frac{b}{m} = 499 \quad (1)$$

$$\hat{\sigma}_- = -\frac{1}{m} = 42.5 \quad (2)$$

These parameters are adjusted to account for the small sample size using the following equations:

$$\hat{\sigma} = \hat{\sigma}_-(\sigma_n/\sigma_n) = 42.5((\pi/\sqrt{6})/0.7932) = 68.7 \quad (3)$$

$$\begin{aligned} \hat{\mu} &= \hat{\mu}_- - \hat{\sigma}_- \bar{y}_n + \hat{\sigma} \bar{y}_n \\ &= 499 - 42.5(0.577) + 68.7(0.4565) \\ &= 506 \end{aligned} \quad (4)$$

The value of the 99% survival stress, S_{99} , is then calculated:

$$\begin{aligned} S_{99} &= \hat{\mu} - 4.60 \cdot \hat{\sigma} \\ &= 506 - 4.60(68.7) \\ &= 190 \text{ MPa} \end{aligned} \quad (5)$$

APPENDIX 2. Sample Calculation of the Threshold Stress, S_{th} .

The first step in determining the statistically-defined threshold stress involves calculation of the slope, B , and intercept, A , of the best-fit line obtained from a plot of the 99% survival stress, S_{99} , versus the exposure stress, S_{exp} , for each length of exposure. The point of intersection of this line with the line defined by $S_{99} = S_{exp}$ is given by:

$$S'_{th} = -\frac{A}{B-1} \quad (1)$$

As noted in the discussion, a range of values can be obtained depending on whether or not anomalous points arising from the statistical procedure are included in the calculation. Inclusion of these points results in a higher value of S_{th} than would be obtained without them. The results for the sulphuric acid anodized specimens with these anomalous points included appear in the following table:

Exposure Length	Slope	Intercept	S'_{th} (MPa)
2 days	-1.76	530	192
4 days	-2.66	483	132
7 days	-4.59	454	81.1
10 days	-5.31	152	24.1

The mean, m , and standard deviation, s , of the values of S'_{th} are determined and used to calculate S_{th} :

$$\begin{aligned} S_{th} &= m - t_{0.95(3)} \cdot \frac{s}{\sqrt{4}} \\ &= 107 - 2.353 \cdot \frac{71}{\sqrt{4}} \\ &= 23 \text{ MPa} \end{aligned} \quad (2)$$

If the 4 day, 207 MPa point, the 7 day, 207 MPa point and the 10 day, 138 MPa point are removed from the calculation, the results become:

Exposure Length	Slope	Intercept	S'_{th} (MPa)
2 days	-1.76	530	192
4 days	-3.64	528	114
7 days	-8.07	613	67.7
10 days	-15.8	394	23.4

APPENDIX 2. (cont.)

The value of S_{th} was then found to be:

$$\begin{aligned} S_{th} &= 99 - 2.353 \cdot \frac{72}{\sqrt{4}} \\ &= 15 \text{ MPa} \end{aligned} \quad (3)$$

APPENDIX 3(a). Analysis of Variance Table for the Box-Cox Transformed Residual Breaking Strengths of Hard Anodized Specimens of AA 7075-T651.

Stress	0 MPa	69 MPa	138 MPa	207 MPa	Grand mean:
Mean	104 (6.2)	99.8 (6.2)	76.1 (6.2)	40.4 (6.2)	80.2
Time	2 days	4 days	7 days	10 days	Error
Mean	100 (6.2)	74.4 (6.2)	89.3 (6.2)	56.8 (6.2)	12.4
Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F ratio	$F_{0.99}$
Mean	1	514000	514000		
Stress	3	51300	17100	22.2	4.13
Time	3	21300	7110	9.21	4.13
Interaction	9	15200	1690	2.19	2.72
Error	64	49400	772		
Total	80	652000			

APPENDIX 3(b). Analysis of Variance Table for the Box-Cox Transformed Residual Breaking Strengths of Uncoated Specimens of AA 7075-T651.

Stress	0 MPa	69 MPa	138 MPa	207 MPa	Grand Mean
Mean	89.2 (4.1)	59.1 (4.1)	53.5 (4.1)	37.9 (4.1)	59.9
Time	2 days	4 days	7 days	10 days	Error
Mean	88.2 (4.1)	89.0 (4.1)	38.5 (4.1)	23.9 (4.1)	8.1
Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F ratio	$F_{0.99}$
Mean	1	287000	287000		
Stress	3	27700	9240	28.2	4.13
Time	3	68100	22700	69.1	4.13
Interaction	9	13400	1480	4.52	2.72
Error	64	21000	328		
Total	80	417000			

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4. AUTHORS (Last name, first name, middle initial) LENARD, Derek R. and STARRATT, Darlene L.			
5. DATE OF PUBLICATION (month and year of publication of document) November 1994	6a. NO. OF PAGES (total containing information. Include Annexes, Appendices, etc.) 32	6b. NO. OF REFS (total cited in document) 25	
7. DESCRIPTIVE NOTES (the category of the document, e.g. technical report, technical note or memorandum. If appropriate, enter the type of report, e.g. interim, progress, summary, annual or final. Give the inclusive dates when a specific reporting period is covered.) DREP Technical Memorandum 94-126			
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The breaking load method was used to determine the effects of alternate immersion in sea water on uncoated, sulphuric acid anodized and hard anodized specimens of AA 7075-T651 in order to evaluate the benefits of anodizing the aluminum pressure hull of an autonomous underwater vehicle to reduce the risk of stress corrosion cracking. The original test method showed that anodizing delayed the onset of stress corrosion cracking of specimens subjected to an externally-applied stress of 69 MPa, with hard anodizing providing a greater benefit than sulphuric acid anodizing. Anodizing was also shown to delay the onset of intergranular cracking in the absence of an external stress. The value of the threshold stress determined for the uncoated specimens was within the range reported by an interlaboratory test program.

The extreme value distribution was found to provide a good fit to the data, but its use did give rise to several problems previously noted in the interlaboratory program. A new statistical procedure involving Box-Cox transformations did alleviate these problems and served to confirm the conclusion reached using the original method, even though the statistical calculations may not have been strictly valid. The new procedure also showed that hard anodizing continued to offer improved resistance to stress corrosion cracking at higher stress levels. Finally, analysis of variance of the transformed data confirmed the conclusion that anodizing provided a significant improvement in resistance to stress corrosion cracking, with hard anodizing providing the greater benefit.

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