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**THERMOMECHANICAL PROCESSING OF
5083 ALUMINUM TO INCREASE STRENGTH
WITHOUT INCREASING SUSCEPTIBILITY TO
STRESS CORROSION CRACKING**

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SUBJECT DESCRIPTORS

Al-Mg Alloys
5083 Aluminum
Stress Corrosion Cracking
Thermomechanical Forming
Al-Mg Precipitation
Thermomechanical Processing

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THERMOMECHANICAL PROCESSING OF 5083 ALUMINUM TO INCREASE STRENGTH WITHOUT INCREASING SUSCEPTIBILITY TO STRESS CORROSION CRACKING

Carl M. Edstrom and Jack J. Blakeslee

Abstract. Aluminum alloy 5083 presents a stress corrosion problem when the metal becomes sensitized; i.e., continuous precipitate of Al-Mg phase along the grain boundaries. By controlling the amount of strain hardening from deformation and by selecting the proper processing temperature, the continuous precipitate can be avoided.

SUMMARY

5083 aluminum with 25% cold work must be processed above 215 °C or below 70 °C to avoid forming continuous precipitate in the grain boundaries which makes the material susceptible to stress corrosion cracking. Time at temperature above 215 °C should be held to minimum (less than 30 minutes) to retain some strength from the 25% cold work.

INTRODUCTION

A hemishell made at Rocky Flats uses 5083 alloy aluminum. This formed hemishell requires a minimum yield strength of 234 MPa (34 ksi) at the equator and the finished formed sphere must pass an Alternate Immersion Stress Corrosion Cracking Test, ASTM G44-75.¹

Initially the parts were formed from 19 mm ($\frac{3}{4}$ " thick aluminum blanks heated to 175 °C. The blanks came from 25.4 mm (1" thick 5083 H321 plate cold rolled to 19 mm ($\frac{3}{4}$ " to increase the strength and satisfy the minimum yield strength of 234 MPa (34 ksi). To obtain the ductility necessary to form the part, the blank required soaking one hour at 175 °C. Stress corrosion, testing C-rings cut from the formed parts, resulted in greater than 90% failure.²

This paper documents the steps followed to develop the final forming procedure used to meet the strength and corrosion requirements for the part.

STRESS CORROSION CRACKING TEST PROCEDURE

The specifics on the test to establish whether a material is susceptible to stress corrosion cracking is given in ASTM G44-75 specification entitled "Standard Recommended Practice for Alternate Immersion Stress Corrosion Cracking in 3.5% Sodium Chloride Solution." Generally, a C-ring specimen is machined from the formed part (refer to Figures 1 and 2) so that when stressed by the aluminum screw, the stress is across the short transverse direction of the plate. The stress applied is 75% of the yield strength. The stressed C-rings are immersed 10 minutes in the salt solution and 50 minutes in air on a repeating cycle, 24 hours a day for 30 days. Periodic visual inspections of the C-rings are made for cracks.

STRESS CORROSION CRACKING FAILURE

After experiencing the high rate of stress corrosion cracking failure with the first parts formed, the as-received plate was examined. The alternate immersion stress corrosion cracking test performed on C-rings cut from the as-received plate resulted in an unacceptable failure rate from three of the four stocked plates.² Metallographic examination of the as-received plate revealed continuous precipitate along the grain boundaries (Figure 3).

Literature Search

Because of the unacceptable stress corrosion cracking results with the as-received plate, Alcoa and Reynolds were contacted for guidance in controlling the continuous grain boundary precipitate. Alcoa sent Technical Paper No. 14, "Development of Wrought Aluminum-Magnesium Alloys," written by E. H. Dix, W. A. Anderson, and M. Bryon Shumaker in 1958³ and two other reports written under Navy contract telling how to test for stress corrosion susceptibility.^{4, 5} Technical Paper No. 14 proved to be a valuable reference for establishing fabricating practices which minimized stress corrosion susceptibility.

Figure 26 of Technical Paper No. 14, which is included in this report as Figure 4 shows that 5083 H12 aluminum (with 4.5% nominal magnesium content) can become susceptible to stress corrosion cracking after being subjected for 2 years to a temperature between 75 – 350 °F (25 – 175 °C). Also from this figure it is seen that 4.5% magnesium becomes completely soluble in aluminum at 500 °F (260 °C). This provided the guide for the experiments to produce parts of 5083 aluminum without stress corrosion cracking susceptibility.

Experiments were necessary because Technical Paper No. 14 showed susceptibility to stress corrosion cracking after two years exposure to the specific temperatures in Figure 4, where in forming our 5083 aluminum part, the aluminum would be exposed to temperatures for less than three hours. Also, Figure 4 applies to H12 material which is equivalent to 18% cold work and our 5083 aluminum has a minimum of 25% equivalent cold work. The paper stated that an increase in retained cold work in the aluminum-magnesium alloys increased the susceptibility for stress corrosion cracking.

Another valuable aspect of Technical Paper No. 14 was the correlation shown between continuous Al-Mg precipitate and susceptibility to stress corrosion cracking. This correlation greatly increased the speed for obtaining experimental results. Metallographic examination was much quicker than machining C-rings and waiting 30 days for stress corrosion results.

Solutionizing Temperature

The first step in the experiments was to remove the continuous precipitate found in the as-received 5083 aluminum plate. This was accomplished by heating to 260 °C, holding one hour at temperature and water quenching (Figure 5), and also by fully annealing three hours at 400 °C and water quenching (Figure 6).

Cold Work and Forming Temperature Versus Stress Corrosion Cracking Susceptibility

Since our material contains more cold work and would be subjected to shorter times at temperature than the material used to generate (Figure 4) the following experiment was performed. Samples of 5083 aluminum plate of 4.8% Mg, 0.8% Mn composition were solutionized at 260 °C (1 hr) and 400 °C (3 hr) and water quenched, cold rolled from 25 mm (1") to 19 mm ($\frac{3}{4}$ ") thickness (25%) and subjected to temperatures from 66 °C (150 °F) to 204 °C (400 °F) for various times, and water quenched at the end of the soak. The specific times and temperatures are outlined in Table 1 along with a written description of precipitate. Figures 7 and 8 provide the visual description of the precipitate forms; dispersed or continuous. These data show that it requires more than 10 minutes at any temperature for the $\frac{3}{4}$ " thick aluminum to develop a continuous precipitate. Also, all samples held two hours at temperature between 150 °C and 190 °C (300 °F and 375 °F) exhibited continuous precipitate; i.e., susceptibility to stress corrosion.

When comparing the susceptibility to stress corrosion cracking from data shown in Table 1 with the curve shown in Figure 4, there appears to be a discrepancy. The Table indicates that susceptibility to stress corrosion cracking develops if the 5083 metal (4.8% Mg) is heated at 190 °C (375 °F) or less, where Figure 4 indicates the susceptible temperature to be 175 °C (350 °F) or less. This difference comes from the differences in cold work of the metals tested. Figure 4 shows test results on H12 temper approximates 18% cold work, whereas the metal tested for Table 1 had 25% cold work. Also, note that the results in Table 1 for the lower

TABLE 1. Continuous Precipitate Formation Versus Time at Temperature

Solutionized at 260 °C		Solutionized at 400 °C	
Time and Temperature	Prec. Form	Time and Temperature	Prec. Form
None	Dispersed	None	Dispersed
16 hr at 66 °C (150 °F)	Dispersed	16 hr at 66 °C (150 °F)	Dispersed
10 min. at 177 °C (350 °F)	Dispersed	10 min. at 177 °C (350 °F)	Dispersed
10 min. at 190 °C (375 °F)	Dispersed	10 min. at 190 °C (375 °F)	Semi-Continuous
30 min. at 190 °C (375 °F)	Dispersed	30 min. at 190 °C (375 °F)	Continuous
10 min. at 204 °C (400 °F)	Dispersed	10 min. at 204 °C (400 °F)	Dispersed
2 hr at 150 °C (300 °F)	Continuous	2 hr at 150 °C (300 °F)	Continuous
2 hr 10 min. at 177 °C (350 °F)	Continuous	2 hr 10 min. at 177 °C (350 °F)	Continuous
2 hr 10 min. at 190 °C (375 °F)	Continuous	2 hr 10 min. at 190 °C (375 °F)	Continuous
2 hr 10 min. at 204 °C (400 °F)	Semi-Continuous	2 hr 10 min. at 204 °C (400 °F)	Continuous

Notes:

1. All samples solutionized, cold rolled 25% reduction prior to heating for stabilization.
2. It requires longer than 10 minutes at any temperature to form a continuous precipitate.

temperatures do not compare to Figure 4 curve because the heating times for the data in Table 1 were less than two years.

Sensitizing by Simulated Cleaning

In processing, the hemishells are cleaned and dried for 24 hours at 150 °C. This temperature is within the sensitization (susceptibility to stress corrosion cracking) sections of the curve in Figure 4. Experiments were performed to determine if a prior processing method would retard the sensitization from the 24 hours, 150 °C drying operation. Also, another drying cycle of 4 hours at 200 °C was examined. The metal was solutionized 1 hour at 260 °C and water quenched, cold rolled from 25 mm (1") to 19 mm (3/4") for the 25% reduction, and from 25 mm (1") to 13 mm (1/2") for the 50% reduction, and given a thermal stabilizing treatment. Table 2 provides a list of these stabilizing treatments and resulting metallography and mechanical properties after the simulated cleaning-drying process. Every sample which was heated for 24 hours at 150 °C developed continuous precipitate in the grain boundaries (Figures 9 and 10). With the exception of one sample, heating the metal 4

hours at 200 °C did not change the precipitate form nor form a continuous precipitate in the grain boundaries. Why the one sample cold worked 25% and stabilized 30 minutes at 215 °C produced continuous, grain boundary precipitate after 4 hours at 200 °C is unknown.

Stress Corrosion Cracking Results on Formed Hemishells

Metal solutionized following two practices (1 hour at 260 °C and 3 hours at 400 °C) and cold rolled from 25 mm to 19 mm (1" to 3/4") was formed into hemishells. The forming temperatures used 1 hour soak at 170 °C, 10 minute soak at 210 °C, and 10 minute soak at 230 °C.

Continuous precipitate was present in the microstructure of the parts heated 1 hour at 170 °C for forming (Figure 11). The precipitate was dispersed in the parts formed at 210 °C and 230 °C (Figures 12 and 13).

The stress corrosion cracking results correlated to the presence of, or lack of, continuous precipitate. Seven of the eight C-rings machined from the parts

TABLE 2. Sensitization From Cleaning-Drying Operation

Sample Number	Cold Work (% Red.)	Stabilizing Treatment	Clean-Dry Treatment	Metallography (See Note)	Tensile Strength MPa (ksi)	Yield Strength MPa (ksi)	Elongation (%)
1	25%	30 min. 215 °C	24 hr 150 °C	A	348.8 (50.6)	230.9 (33.5)	13.2
4	25%	10 min. 215 °C	24 hr 150 °C	A	348.1 (50.5)	235.1 (34.1)	13.0
7	25%	30 min. 230 °C	24 hr 150 °C	A	341.9 (49.6)	224.7 (32.6)	14.3
10	25%	10 min. 230 °C	24 hr 150 °C	A	344.0 (49.9)	224.7 (32.6)	13.3
13	25%	10 min. 250 °C	24 hr 150 °C	A	341.9 (49.6)	222.7 (32.3)	14.8
16	50%	30 min. 230 °C	24 hr 150 °C	A	347.8 (51.9)	250.3 (36.3)	13.0
19	50%	30 min. 250 °C	24 hr 150 °C	A	357.8 (51.9)	236.5 (34.3)	17.0
2	25%	30 min. 215 °C	4 hr 200 °C	A	341.9 (49.6)	224.7 (32.6)	13.0
5	25%	10 min. 215 °C	4 hr 200 °C	B	342.6 (49.7)	229.6 (33.3)	13.0
8	25%	30 min. 230 °C	4 hr 200 °C	B	337.8 (49.0)	224.1 (32.5)	12.5
11	25%	10 min. 230 °C	4 hr 200 °C	B	340.0 (49.3)	225.4 (32.7)	14.5
14	25%	10 min. 250 °C	4 hr 200 °C	B	339.2 (49.2)	223.4 (32.4)	13.3
17	50%	30 min. 230 °C	4 hr 200 °C	B	355.7 (51.6)	249.6 (36.2)	13.5
20	50%	30 min. 250 °C	4 hr 200 °C	B	351.6 (51.0)	235.8 (34.2)	17.0
3	25%	30 min. 215 °C	None	B	341.9 (49.6)	232.3 (33.7)	12.8
6	25%	10 min. 125 °C	None	B	342.6 (49.7)	233.0 (33.8)	11.8
9	25%	30 min. 130 °C	None	B	337.1 (48.9)	222.7 (32.3)	14.3
12	25%	10 min. 230 °C	None	B	345.4 (50.1)	231.6 (33.6)	15.0
15	25%	10 min. 250 °C	None	B	344.0 (49.9)	226.8 (32.9)	14.0
18	50%	30 min. 230 °C	None	B	358.5 (42.0)	250.9 (36.4)	14.0
21	50%	30 min. 250 °C	None	B	348.8 (50.6)	235.8 (34.2)	14.0

Note: Metallography A equals continuous precipitate in grain boundaries (refer to Figures 9 and 10).

Metallography B equals non-continuous precipitate (refer to Figures 9 and 10).

formed after heating 1 hour at 170 °C cracked in the stress corrosion cracking test. None of the C-rings machined from the parts formed at 210 °C and 230 °C cracked. The C-rings that failed cracked with 7 days exposure (Table 3).

C-Ring Orientation Versus Stress Corrosion Cracking

One set of C-rings (eight per hemishell) were machined from a hemishell formed after 1 hour at 170 °C to ensure sensitization. These C-rings were oriented so that four would be stressed in the longitudinal direction and four in the long transverse direction.

No C-rings stressed in the longitudinal or long transverse direction cracked; whereas seven of the eight

C-rings taken from another hemishell formed as a duplicate at 170 °C did crack when stressed in the short transverse direction. This experiment demonstrates that unless the part is strained across the short transverse direction, failure owing to stress corrosion cracking will not occur.

MECHANICAL PROPERTIES

Solutionizing Temperature

Yield strength in the parts formed from the metal solutionized for three hours at 400 °C prior to cold rolling 25% was less than the yield strength for the parts formed from the metal solutionized 1 hour at 260 °C (Table 4).

TABLE 3. Alternate Immersion Stress Corrosion Cracking Test.

Part Processing		C-Ring Stress Direction	No C-Rings Tested	Number of C-Rings Cracked
Solutionize Temperature	Forming Temperature			
3 hr 500 °C	1 hr 170 °C	Longitudinal	4	None
3 hr 400 °C	1 hr 170 °C	Long Transverse	4	None
3 hr 400 °C	1 hr 170 °C	Short Transverse	8	7 in 7 Days
2 hr 260 °C	1 hr 170 °C	Longitudinal	4	None
2 hr 260 °C	1 hr 170 °C	Long Transverse	4	None
2 hr 260 °C	10 min. 210 °C	Longitudinal	3	None
2 hr 260 °C	10 min. 210 °C	Long Transverse	3	None
3 hr 400 °C	10 min. 210 °C	Longitudinal	3	None
3 hr 400 °C	10 min. 210 °C	Long Transverse	4	None
3 hr 400 °C	10 min. 210 °C	Short Transverse	8	None
2 hr 260 °C	10 min. 230 °C	Longitudinal	3	None
2 hr 260 °C	10 min. 230 °C	Long Transverse	4	None
3 hr 400 °C	10 min. 230 °C	Longitudinal	4	None
3 hr 400 °C	10 min. 230 °C	Long Transverse	4	None
3 hr 400 °C	10 min. 230 °C	Short Transverse	8	None

TABLE 4. Yield Strength of Formed Hemishells

Forming Temperature	Average Yield Strength ¹		Average Yield Strength ² Solutionize 400 °C		Average Yield Strength ² Solutionize 260 °C	
	(MPa)	(ksi)	(MPa)	(ksi)	(MPa)	(ksi)
170 °C	236	34.2	232	33.7	239	34.6
210 °C	232	33.6	227	32.9	236	34.3
230 °C	232	33.7	230	33.4	234	34.0

¹ Average of four tests.² Average of two tests.**Forming Temperature**

Yield strength generally decreased as the forming temperature increased (Table 4).

The tensile and yield strengths were not significantly decreased by the thermal drying cycles. The average tensile strengths for metal with no thermal drying, 24 hours at 150 °C, and 4 hours at 200 °C, respectively, were 50.1, 50.6, and 49.9 ksi. The average yield strengths for the same respective thermal drying cycles were 33.8, 33.7, and 33.4 ksi (refer to Table 2).

SUBSTITUTE STRESS CORROSION TEST

Because the Alternate Immersion Stress Corrosion Cracking Test is expensive owing to machining time on the C-ring and very time consuming, it was desirable to examine an accelerated test for susceptibility stress corrosion. Two exfoliation corrosion tests developed through the Aluminum Association, ASSET (Ammonium-Salt Solution Exfoliation Test) and NAWLT (Nitric Acid Weight Loss Test) detect continuous grain boundary precipitate in 24 or 48 hours of testing. It was thought that one of these tests could serve as the

quality check in place of the 30 day Alternate Immersion Stress Corrosion Cracking Test. To check correlation of the current Alternate Immersion Stress Corrosion Cracking Test with the ASSET and NAWLT tests, two approximate 2" X 2" sections were cut from the formed hemishells and subjected to the two tests; the results compared to C-rings cut from the same parts but tested by the Alternate Immersion SCC test.

The ASSET and NAWLT tests did not prove to be as definite as the Alternate Immersion Stress Corrosion Cracking Test for determining metal susceptible to stress corrosion cracking (refer to Appendix A).

TESTING SPECIFICS

Mechanical Property Specimens

All tensile specimens obtained from drawn hemishells were R4 type (2" long by 0.160" gage diameter). These tensile bars were machined from an area near the equator. The axis of the tensile bar followed the longitude of the hemishell.

All tensile specimens obtained from rolled plate such as for the simulated cleaning test were either R1 (5½" long by 0.505" gage diameter) or R2 (3½" long by 0.357" gage diameter) type.

Metallography Samples

The metallographic samples cut from drawn hemishells came from near the equator. The surface examined metallographically is that which touches when two hemishells are brought together to form a sphere.

The metallographic samples from plate were cut so that the surface examined was the plate edge. This showed elongated grains with long grain boundaries to examine for continuous precipitate.

EXPERIMENTAL CONCLUSIONS

1. Both solutionizing treatments, one hour at 260 °C and three hours at 400 °C, each

followed by a water quench, solutionized the magnesium-aluminum precipitate. The two hours at 260 °C treatment is preferred because it provides higher mechanical properties in the formed part.

2. The presence of continuous precipitate increased the chances for susceptibility of stress corrosion cracking.
3. Subjecting 5083 aluminum with residual strain equivalent to 25% cold work or more, to the temperature between 70 °C and 190 °C caused precipitation of the Mg-Al compound along the grain boundaries. Continuous precipitate formed in 30 minutes at temperatures of 150 °C to 190 °C, but required longer than 16 hours to form at 70 °C.
4. To prevent formation of continuous precipitate in grain boundaries during forming the part, the metal must be heated above 210 °C or below 70 °C.
5. No combination of cold working reduction and thermal stabilizing treatment within the restraint of holding 234 MPa (34 ksi) minimum yield strength was found which prevented continuous precipitate from forming after 24 hours at 150 °C, the drying procedure followed during part cleaning.
6. The substitute drying procedure, in part cleaning, of 4 hours at 200 °C did not create continuous precipitate in metal which had been stabilized at 215 °C or higher.
7. When stressing C-rings known to be susceptible to stress corrosion cracking (continuous grain boundary precipitate) across the longitudinal or long transverse directions, no failures occurred. However, C-rings from the same material failed in stress corrosion cracking tests when stressed in the short transverse direction.
8. The ASSET (Ammonium-Salt Solution Exfoliation Test) and NAWLT (Nitric Acid Weight Loss Test) were less definitive in determining susceptibility to stress corrosion cracking than the Alternate Immersion SCC Test. Therefore, the quicker, less expensive

tests cannot substitute for the Alternate Immersion SCC Test.

Technical Paper No. 14³ states (in reference to the Al-Mg phase diagram) at room temperature the estimated solubility of magnesium in aluminum is quite low. Thus, magnesium in excess of the solubility limit, would tend to form precipitate particles of an aluminum-magnesium compound. Fortunately, precipitation occurs only very slowly and to a limited extent at atmospheric temperatures. It is important to remember, however, that an increase in both temperature and degree of strain hardening by cold deformation accelerates the precipitation process. Based on this statement and other data in Technical Paper No. 14³, I would predict that the parts cold worked 25%, formed at 210 °C or greater, and free of Mg precipitate after forming, would form continuous precipitate after 15 - 20 years at room temperature and be susceptible to stress corrosion cracking in a corrosive environment.

Attention to the following can prevent stress corrosion cracking of 5083 aluminum parts in service.

1. First form a part without the Al-Mg precipitate and avoid subjecting the part to temperatures between 70 °C - 190 °C. This buys a 15 - 20 year waiting period before the magnesium precipitates and makes the 5083 part susceptible to stress corrosion cracking.
2. Avoid stressing the part across the short transverse direction and/or keep short transverse surface from exposure to the corrosive environment.
3. Real environment is normally not as severe as the Alternate Immersion Stress Corrosion Cracking Test which simulates 20 years at the beach at Fort Judith, Rhode Island, so the part will not fail within 30 days after becoming susceptible to stress corrosion cracking. Any corrosive medium less severe than the 3.5% sodium chloride in the Alternate Immersion SCC Test buys time before failure occurs. Also any stress level less than the 75% of yield strength used in the Alternate Immersion SCC Test buys time before failure occurs.

RECOMMENDATIONS

The recommended fabricating practice for the 5083 aluminum parts is:

1. Solutionize 1 hour at 260 °C and water quench.
2. Cold roll 25% using a cross rolling pass sequence.
3. Heat the blanks 10 - 30 minutes at >215 °C.
4. Form.

It is also recommended that the current drying practice in the cleaning operation be changed. The temperature needs increasing from 150 °C to 200 °C or 215 °C and the time at temperature needs decreasing to a maximum of 4 hours to protect from mechanical property loss.

REFERENCES

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3. E. H. Dix, Jr., W. A. Anderson, and M. B. Shumaker, "Development of Wrought Aluminum Magnesium Alloys," Technical Paper No. 14, 1958; Alcoa Research Laboratories.
4. E. J. Czyryca and H. P. Hack, "Corrosion of Aluminum Alloys in Exfoliation - Resistant Tempers Exposed to Marine Environments for Two Years," prepared under TOP 21, Task Area SF 54541702, Task 14626 at Naval Ship Research and Development Center, Bethesda, Maryland, 20084.
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ILLUSTRATIONS
(Figures 1 through 13)

FIGURE 1. C-Ring Orientation With Hemishell

FIGURE 2. Dimensions of C-Ring Specimens

FIGURE 3. Continuous Precipitate
Found in the As-Received 5083 H321
Plate. Etch: 10% phosphoric acid at 50 °C

FIGURE 4. Effect of Temperature on Susceptibility to Stress-Corrosion Cracking. (Time of heating—2 years.)

FIGURE 5. Precipitate Form in Solutionized and Rolled 5083 Plate. Solutionized 1 hour at 260 °C, rolled 25% reduction. Etch: 10% phosphoric acid at 50 °C.

400X

FIGURE 6. Precipitate Form in Solutionized and Rolled 5083 Plate. Solutionized 3 hours at 400 °C, rolled 25% reduction. Etch: 10% phosphoric acid at 50 °C.

400X

FIGURE 7. Continuous Precipitate in 5083 Aluminum. Solutionized, rolled, stabilized.

Actual sample solutionized at 400 °C and stabilized 2 hours, 10 minutes at 350 °F (177 °C) but photo also represents microstructures from:

<u>Solutionized 260 °C</u>	<u>Solutionized 400 °C</u>
Stabilized -	
2 hr at 150 °C	2 hr at 150 °C
2 hr 10 min. at 177 °C	30 min. at 190 °C
2 hr 10 min. at 190 °C	2 hr 10 min. at 190 °C
	2 hr 10 min. at 204 °C

FIGURE 8. Dispersed Precipitate in 5083 Aluminum. Solutionized, rolled, stabilized.

Actual sample solutionized at 400 °C and stabilized 10 minutes at 177 °C. Photo also represents microstructure from:

Solutionized 260 °C

Stabilized -

16 hr at 66 °C
10 min. at 177 °C
10 min. at 190 °C
30 min. at 190 °C
10 min. at 204 °C

Solutionized 400 °C

16 hr at 66 °C
10 min. at 204 °C

FIGURE 9. Simulated Cleaning-Drying Sensitizing Effect

General: Metal solutionized 1 hr at 260 °C water quenched.
Cold rolled from 25 mm (1") to 18 mm ($\frac{3}{4}$ ") -25%,
Heated 20 min. at 215 °C, water quenched, then:

Heated 24 hr at 150 °C.

Etch: 10% Phosphoric Acid at 50 °C.

400X

Heated 4 hr at 200 °C.

Etch: 10% Phosphoric Acid at 50 °C.

400X

No simulated drying.

Etch: 10% Phosphoric Acid at 50 °C.

400X

FIGURE 10. Simulated Cleaning-Drying Sensitizing Effect

General: Metal solutionized 1 hr at 260 °C, water quenched.
Cold rolled from 25 mm (1") thickness to 13 mm
(½") -50%. Heated 30 min. at 230 °C, water
quenched, then:

Heated 24 hr at 150 °C.

Etch: 10% Phosphoric Acid at 50 °C.

400X

Heated 4 hr at 200 °C.

Etch: 10% Phosphoric Acid at 50 °C.

400X

No simulated Drying

Etch: 10% Phosphoric Acid at 50 °C.

400X

**FIGURE 11. Continuous Precipitate Found in
Parts Formed After One Hour at 170 °C**

Sample E 8

Solutionized at 400 °C.

Etch: 10% Phosphoric Acid at 50 °C.

500X

Sample E 10

Solutionized at 260 °C

Etch: 10% Phosphoric Acid at 50 °C.

500X

**FIGURE 12. Dispersed Precipitate Found in
Part Formed After 10 Minutes at 210 °C**

Sample E 12

Solutionized at 260 °C.

Etch: 10% Phosphoric Acid at 50 °C.

500X

Sample E 14

Solutionized at 400 °C.

Etch: 10% Phosphoric Acid at 50 °C.

500X

**FIGURE 13. Dispersed Precipitate Found in
Parts Formed After 10 Minutes at 230 °C**

**Sample E 16
Solutionized at 260 °C.
Etch: 10% Phosphoric Acid at 50 °C.
500X**

**Sample E 18
Solutionized at 400 °C
Etch: 10% Phosphoric Acid at 50 °C
500X**

APPENDIX A

**Exfoliation and Stress Corrosion Cracking Tests of
5083 Aluminum Alloy**

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Component Development
CHEMISTRY RESEARCH AND DEVELOPMENT

KWIC Index

Stress Corrosion Cracking
5083 Aluminum
Corrosion

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INTRODUCTION

An alternate immersion stress corrosion cracking (SCC) test has been employed to determine the susceptibility of 5083 Al to SCC.¹ The 30-day ASTM method² was adopted and M-documented as a quality acceptance test for the Hatch parts.

Further studies have been performed to determine whether two short duration (24-48 hours) exfoliation* corrosion test methods, the Ammonium-Salt Solution Exfoliation Test (ASSET) and the Nitric Acid Weight Loss Test (NAWLT)³ could be correlated to the alternate immersion test and microstructure. The ASSET has been approved by the Aluminum Association for determining aluminum alloy susceptibility to exfoliation. The NAWLT has been evaluated by the Aluminum Association as a promising method for determining susceptibility of aluminum alloys to intergranular corrosion and exfoliation corrosion.

The results of ASSET tests performed concurrently by three Aluminum Association members (Alcoa, Reynolds, and Kaiser) concluded that the susceptibility to exfoliation was primarily related to a microstructure with elongated grains and a relatively continuous precipitate of Al-Mg phase along these grain boundaries.³ Since sensitized 5083 Al exhibits such microstructure, the exfoliation tests were performed on that alloy to identify possible correlation with the Alternate Immersion SCC test.

In another evaluation test program³ performed by five Aluminum Association members, the weight loss results of the NAWLT test correlated well with the degree of grain boundary attack seen in the ASSET test. This study also explores this possible correlation of NAWLT test with the Alternate Immersion SCC test.

EXPERIMENTAL PROCEDURE

ASSET Test

Six sample specimens of 5083 Al were obtained for the ASSET test by sectioning Hatch parts with varying heat treatments as shown in Table 1.

*Exfoliation is that form of corrosion which proceeds laterally from the sites of initiation along grain boundaries parallel to the surface. The corrosion products force the metal upwards resulting in a layered surface appearance.

TABLE 1. ASSET Test Sample Description

Part Number	Specimen Surface Area	Fabrication Heat Treatment	
		Solutionizing Temperature (°C)	Forming Temperature (°C)
E8	11.53	3 hr at 400-WQ ^a	1 hr at 170
E10	10.57	2 hr at 260-WQ	1 hr at 170
E12	14.25	2 hr at 260-WQ	10 min. at 210
E14	12.15	3 hr at 400-WQ	10 min. at 210
E16	11.44	2 hr at 260-WQ	10 min. at 228
E18	13.18	3 hr at 400-WQ	10 min. at 228

a. Water quench

Following the procedures outlined by the Aluminum Association for the ASSET, the sample specimens were degreased in a 2 vol % detergent (Oakite Aluminum Cleaner) solution at room temperature for 5 minutes with sonication. The specimens were then rinsed for 5 minutes in flowing, cascading distilled water and then allowed to air dry for one hour.

After degreasing, the sample specimens were etched for 1 minute in 5 wt % sodium hydroxide at 82 °C and then rinsed in distilled water. The specimens were next desmutted for 30 seconds in concentrated nitric acid at room temperature, rinsed with distilled water, and allowed to air dry.

The sample specimens were then immersed for 24 hours at 66 ± 1 °C in the corrodent solution which consisted of:

- 1.0 M ammonium chloride
- 0.25 M ammonium nitrate
- 0.01 M ammonium tartrate
- 3 g/l hydrogen peroxide (10 ml of 30% stock solution per liter)

The sample specimens were immersed in sufficient quantity of the corrodent solution to provide a minimum volume to surface area ratio of 50 ml/in².

After 24 hours immersion, the sample coupons were removed from the corrodent solution, rinsed in distilled water and soaked in concentrated nitric

TABLE 2. NAWLT Test Sample Description^a

Part Number	Specimen Surface Area (in ²)	Specimen Initial Weight (gm)
E8	12.86	123.4093
E10	12.61	114.2792
E12	13.24	126.6035
E14	12.95	125.5441
E16	12.74	122.0268
E18	13.20	128.9102

a. Heat treatment of these duplicate samples was identical to the parameters in Table 1.

acid 23 ± 2 °C until clean (45–60 seconds). The specimens were then rinsed in distilled water, air dried, and visually inspected for exfoliation.

NAWLT Test

Six sample specimens (duplicates of the ASSET test specimens) were selected for the NAWLT test as shown in Table 2.

Preparation of these samples followed the procedures outlined for the NAWLT test by the Aluminum Association.³ This included ultrasonic aqueous cleaning for 5 minutes at 23 ± 1 °C, followed by an etch in NaOH and desmutted in HNO₃ as previously described for the ASSET tests.

The cleaned specimens were weighed to the nearest 0.1 mg and immersed in 70 wt % nitric acid (125 ml/in² – volume to surface area ratio) at 30 ± 1 °C for 24 hours. After 24 hours storage the specimens were removed, rinsed in distilled water, air dried, and reweighed to the nearest 0.1 mg.

Stress Corrosion Cracking Alternate Immersion Test

To correlate the ASSET and NAWLT test results to the Alternate Immersion SCC test, the C-rings tested were machined from the same or duplicate processed Hatch parts from which the ASSET and

TABLE 3. Identity of C-Rings

Part Number	Solutionize (°C)	Forming Temperature (°C)	C-Ring Stress Direction	Number C-Rings
E8	3 hr 400	1 hr 170	Longitudinal	4
E8	3 hr 400	1 hr 170	LT	4
E10	2 hr 260	1 hr 170	Longitudinal	4
E10	2 hr 260	1 hr 170	LT	4
E12	2 hr 260	10 min. 210	Longitudinal	3
E12	2 hr 260	10 min. 210	LT	3
E14	3 hr 400	10 min. 210	Longitudinal	3
E14	3 hr 400	10 min. 210	LT	4
E16	2 hr 260	10 min. 230	Longitudinal	3
E16	2 hr 260	10 min. 230	LT	4
E18	3 hr 400	10 min. 230	Longitudinal	4
E18	3 hr 400	10 min. 230	LT	4
E9	3 hr 400	1 hr 170	Short Trans.	8
E15	3 hr 400	10 min. 210	Short Trans.	8
E19	3 hr 400	10 min. 230	Short Trans.	8

Note: LT = Long Transverse

NAWLT samples were cut; Table 3 provides the identity of the C-rings.

Briefly, the Alternate Immersion SCC test consists of exposing stressed C-rings for 30 days to alternate immersion in a 3.5 wt % sodium chloride solution. The stress level is 90% of yield strength.

RESULTS

ASSET Test

The results of the ASSET tests which are based on visual assessment are summarized in Table 4.

Although pit blistering, i.e. "lipping" of pits was exhibited by samples E10 and E16, no evidence of exfoliation was observed for any of the samples tested. The ASSET test results appeared to be inconsistent because there are essentially only three variables (E8 and E10, E12 and E14; and E16 and E18). The processing procedures for these pair samples are nearly identical and the

TABLE 4. ASSET Test Results

Sample Part, Identifiable	Sensitized Condition	Visual Assessment
E8	Sensitized ^a	Light pitting
E10	Sensitized ^a	Heavy pit blistering
E12	Borderline sensitization ^b	Medium pitting
E14	Borderline sensitization ^b	Light pitting
E16	No sensitization ^c	Heavy pit blistering
E18	No sensitization ^c	Medium pitting

a. Forming temperature 170 °C

b. Forming temperature 210 °C

c. Forming temperature 228 °C

TABLE 5. NAWLT Test Results

Part Number Identification ^a	Specimen Surface Area (in ²)	Specimen Weight Loss (mg)	Weight Loss per Unit Area (mg/in ²)
E8	12.86	184.7	14.4
E10	12.61	219.7	17.4
E12	13.24	167.2	12.6
E14	12.95	149.3	11.5
E16	12.74	167.7	13.2
E18	13.20	160.0	12.1

a. Heat treatments previously described in Table 5.

microstructure of the two samples in the pair are identical; yet the pair samples reacted to the ASSET test with different sensitivity to the etch.

A sample specimen of as-received 5083 Al plate (surface area – 30.410 in²), known to be sensitized, was next submitted to the ASSET. The sensitized material exhibited heavy pit blistering but again, no exfoliation was detected.

NAWLT Test

Table 5 summarizes the results of the NAWLT.

The paired sample weight losses did show some indication that E8 and E10 (the sensitized samples)

TABLE 6. Alternate Immersion Stress Corrosion Cracking Test

Part Number	Stress Direction	Metallographic Assessment of Sensitization ^a	Number of C-Rings Cracked
E8	Longitudinal	Sensitized	None
E8	LT ^b	Sensitized	None
E10	Longitudinal	Sensitized	None
E10	LT	Sensitized	None
E12	Longitudinal	Not Sensitized	None
E12	LT	Not Sensitized	None
E14	Longitudinal	Not Sensitized	None
E14	LT	Not Sensitized	None
E16	Longitudinal	Not Sensitized	None
E16	LT	Not Sensitized	None
E18	Longitudinal	Not Sensitized	None
E18	LT	Not Sensitized	None
E9	Short Trans.	Sensitized	7 of 8 in 7 days
E15	Short Trans.	Not Sensitized	None
E19	Short Trans.	Not Sensitized	None

a. Continuous precipitate in grain boundaries is defined as being sensitized.

b. LT = Long Transverse

had greater weight loss; however, none of the samples exhibited excessive weight loss as defined by Aluminum Association criteria, i.e. >100 mg/in². Neither the surface appearance nor weight loss values provided enough difference to distinguish the different degree of sensitization.

A sample specimen of as-received 5083 Al plate (surface area – 30.257 in², weighing 384.3084), known to be sensitized, was also submitted to the NAWLT. A metal loss rate of 54.9 mg/in² was measured for the specimen after the HNO₃ exposure. This rate is much greater than the thermally processed samples, however not excessive according to Aluminum Association NAWLT criteria.

Alternate Immersion Stress Corrosion Cracking Test

Table 6 displays the results of the Alternate Immersion SCC test. None of the 44 C-rings

stressed in the longitudinal and long transverse directions failed in the 30 day test; even though the metallographic examination of parts E8 and E10 showed a sensitized condition of continuous precipitate in grain boundaries. However, when the C-rings were stressed in the short transverse direction (parts E9, E15, and E19) the C-rings from part E9 did crack. Metallographic examination of part E9 showed the same degree of continuous precipitate in the grain boundaries as in part E8.

DISCUSSION

The NAWLT test did show a slightly greater weight loss for the sensitized material (E8 and E10); however, the amount of weight loss seen does not exceed the 100 mg/in² excessive weight loss criteria set by the Aluminum Association. The difference between the nonsensitized samples (E12, 14, 16, and 18) and sensitized sample (E8 and E10) is too small to be distinguishable as sensitized without other information.

Our less than encouraging results from the ASSET and NAWLT as a substitute for the more expensive and time consuming Alternate Immersion SCC test versus the encouraging results of the Aluminum Association members is probably related to the degree of sensitization. None of the part forming processes followed in the experiment produced as heavy a continuous grain boundary precipitate as found in the as-received 5083 H113 plate.

CONCLUSIONS

1. Neither the ASSET nor NAWLT test can be substituted for the Alternate Immersion Stress Corrosion Cracking Test to determine the

susceptibility of stress corrosion cracking of the 5083 aluminum parts produced for the W70 and B77 programs.

2. The degree of sensitization produced by practices followed in the production of test parts for this experiment does not present a stress corrosion cracking problem when the stresses are across the longitudinal and long transverse directions.

RECOMMENDATION

Since the NAWLT test did show a slight distinction between the sensitized and nonsensitized samples it is recommended that the time in solution be increased from 24 to 48 hours. Maybe this increased exposure will further separate the weight loss values between the two material conditions.

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