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The Effects of Retrogression and Reaging on Aluminum Alloy 2099 (C458)

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The objective of this study was to investigate the feasibility of performing retrogression and reaging (RRA) heat treatments on 2099 aluminum-lithium alloy. The retrogression temperatures were 200-250 °C and retrogression times were 5-60 min. Half of the samples were exposed to a salt fog environment. Interestingly, the samples exposed to salt spray had consistently higher mechanical tensile properties than those which were not exposed.

Keywords aluminum-lithium alloys, heat treatable aluminum alloys, retrogression and reaging

1. Introduction

Aluminum-lithium alloys were developed for the aircraft industry with the intention of reducing weight thus allowing increased payload and fuel efficiency. It started in late 1950s with alloy 2020 being used in aircraft structures to the early 1990s with 2195 being used for the space shuttle external tank. Al 2099 today has many applications in aerospace. 2099 is used in all applications from structures and plating to cryogenic containers. Age-hardenable alloys that are peak aged to the T6 or T8 temper possess the highest strength. For the 7xxx series aluminum alloys, when they are peak aged, they are highly susceptible to stress corrosion cracking (SCC) which can lead to premature failure. One way to remedy this problem is to over age the material to a T7 temper but this causes the aluminum alloy to lose 10-17% of its strength. A solution to this problem is retrogression and reaging (RRA). In general, RRA is applied as a method to extend the service life of aging aircrafts. RRA is recommended for use in components that are life limited by corrosion due to decreasing fatigue strength and tensile properties (Ref 1-8).

Retrogression and reaging (RRA) is a heat treatment process that was developed to allow aluminum alloys to display T7 corrosion resistance with T6 strength. This process has proven successful with 7xxxx series aluminum alloys but was not tested with aluminum-lithium alloys. The heat treatment process of RRA involves a material being heated to a high temperature just below the solvus line for a short period of time. This step of the process is called retrogression. The material is then reaged back to its peak strength to provide high strength and SCC resistance. It was reported that retrogression is essentially a grain boundary precipitate coarsening treatment (Ref 9).

Talianker and Cina (Ref 3) indicated that the presence of dislocations is the primary factor in determining susceptibility of alloys to SCC rather than the precipitate structure. The beneficial effects of RRA are due to the disappearance of these dislocations. While studying the microstructures, T6, and RRA conditions of 7xxx series aluminum, it was found that the only differences were the size and size distribution of matrix participate, and coarsening of grain boundary precipitates in the RRA condition.

Romios et al. (Ref 10) subjected T8 samples to a T6 two-step aging heat treatment process. This study was aimed to develop T6 aging treatments that were capable of achieving strength and ductility comparable to that of an alloy in the T861 (stretched prior to aging) condition. Parts heat treated to a T6 temper had strength and ductility comparable to the T8 temper but without stretching before aging. Samples were aged at low temperatures to nucleate particles. Then they were further aged at a second temperature above the first temperature to grow these particles until a desired strength was reached. The tensile and yield strengths had increased up to 99% of the T861 condition (Ref 10). A similar T6 two-step heat treatment process was utilized in this study.

The objective of this research was to determine the effects of RRA on the stress corrosion resistance of Al 2099 (C458) as well as to determine the optimal combination of retrogression time and temperature to maximize mechanical properties and minimize susceptibility to SCC. The effectiveness of the RRA process was measured by comparing the mechanical properties of the RRA samples to the as-received samples in the T8 temper. Half of the samples were exposed to a salt spray to simulate a corrosive environment; the other half were not exposed. All the RRA samples were compared to the two-step heat-treated T6 temper (Ref 10) and the as-received T8 temper to examine how the RRA treatment maintained the peak strength and SCC resistance properties.

2. Experimental Procedure

The alloys were retrogressed at 200, 220, 240, 250, and 260 °C for 5 min, 10 min, 20 min, 40 min, and 1 h. They were

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then reaged at 120 °C for 12 h for the first step and reaged at 180 °C for 16 h in the second step in process 1. In process 2, they were reaged at 120 °C for 12 h for the first step and then reaged at 180 °C for 24 h.

The nominal chemical composition of Al 2099 (C458) is shown in Table 1. The alloy was received as 0.91 m (3 ft) × 1.22 m (4 ft) × 0.013 m (0.5 in.) plates in the T8 temper. Venema and Rioja (Ref 11) showed that the 2099 (previously AF/C458) alloy in the T8 condition exhibits ultimate strengths between 503 MPa (73 ksi) and 524 MPa (76 ksi), yield strengths between 420 MPa (61 ksi) and 490 MPa (71 ksi) with a percent elongations between 7.4 and 8.8%. The orientation relevant to this study is the longitudinal direction with ultimate strength of 524 MPa (76 ksi), yield strength of 490 MPa (71 ksi) and 10.7% elongation. The ultimate and yield strengths, and percent elongation in the as-received condition with and without salt spray are shown in Table 2 and 3.

2.1 Retrogression

Retrogression was conducted using a salt bath furnace and an oil bath furnace between 5-60 min and 200-250 °C. Following retrogression, and before reaging, the samples were naturally aged for 24 h. All retrogression treatments were performed in an oil bath. Initially a salt bath was used; however, the salt residue took longer time to remove before tensile testing. A Blue M Magni oil bath (Model: MW-1155C-2) was used for the oil bath, and a McEnglevan Speedy Melt Salt bath furnace (Model: P812) was used for the salt bath retrogression treatments.

Table 1 Chemical composition of alloying elements in Al 2099 (C458), Lot No. 596050

Chemical composition of Al 2099 (C458)							
Alloying element	Cu	Li	Zn	Mg	Zr	Ti	Mn
wt.%	2.58	1.73	0.60	0.26	0.09	0.01	0.25

2.2 Reaging

A two-step heat treatment process was performed to bring the 2099 aluminum at T6 temper to a T8 type strength and ductility without the 6% stretch used to achieve the T8 temper (Ref 10). In this study, two different reaging processes were conducted. Process one had a first stage at 120 °C for 12 h, and the second stage took place at 180 °C for 16 h. Process 2 had a first stage at 120 °C for 12 h and the second stage took place at 180 °C for 24 h (Table 4).

2.3 Salt Fog Test

After the reaging process, the samples were exposed to a salt fog test. Half the samples were exposed and half the samples were not. Salt fog testing exposure was performed in accordance with ASTM B 117 (Ref 12).

2.4 Mechanical Properties

Following the retrogression, the remaining oil or salt residues was removed by grinding with silicon carbide (grit 180) paper or by wiping any loose residue off. Tensile specimens were prepared and tested in accordance with ASTM standard E8. The tensile samples were rectangular plate specimens, with 203.2 mm (8.0 in.) total length, 50.8-mm (2.0 in.) gage length, 12.7-mm (0.5 in.) width, 57.15-mm (2.25 in.) length of reduced section and 12.7-mm (0.5 in.) radius of fillet. The thickness of the samples was reduced to 6.35 mm (0.25 in.). The grips had 50.8-mm (2.0 in.) length, and 19.05-mm (0.75 in.) width. Specimens were machined using standard milling machines and a CNC machine. Tensile test were performed on an Instron 4505 test frame at a constant cross head speed of 1.27 mm/min (0.05 in./min). All RRA samples were tested in duplicates. Only in the as-received condition, more than two samples were tested.

3. Results and Discussion

On observing the data, a few trends can be seen. The first observation is that the samples that were exposed to a salt environment showed better tensile strength results as compared

Table 2 2099 T8 as-received properties (no salt spray exposure)

	2099 T8 as-received properties (no RRA)		
	σ_{ult}, MPa (ksi)	σ_{yield}, MPa (ksi)	Percent elongation
Sample 1	546.8 (79.3)	495.8 (71.9)	10.2
Sample 2	531.6 (77.1)	468.9 (68.0)	9.8
Sample 3	531.6 (77.1)	483.3 (70.1)	Broke outside the gage length
As-received T8 (average) [average of sample 1, 2 and 3]	536.7 (77.8)(a)	482.7 (70.0)(a)	10.0(a)
Standard deviation	7.2	11.0	0.2
T6 modified to resemble T8 (process 1) (Ref 10)	530.9 (77.0)(a)	482.7 (70.0)(a)	11.0(a)
Ref 11	524 (76.0)(a)	490 (71.0)(a)	10.7(a)

The percent elongation was not calculated for samples which broke outside the gage length. The position of the fracture was slightly away from the gage, and the samples never failed in the radius or in the grip
(a) Average numbers

Table 3 2099 T8 as-received properties (salt spray exposure)

2099 T8 as-received properties (no RRA)			
	σ_{ult}, MPa (ksi)	σ_{yield}, MPa (ksi)	Percent elongation
Sample 1	559.9 (81.2)	522.0 (75.7)	Broke outside the gage length
Sample 2	557.1 (80.8)	517.1 (75.0)	8.3
Sample 3	538.5 (78.1)	N/A	Broke outside the gage length
Sample 4	533.7 (77.4)	489.5 (71.0)	Broke outside the gage length
Sample 5	533.0 (77.3)	475.0 (68.9)	Broke outside the gage length
Sample 6	559.2 (81.1)	500.6 (72.6)	8.8
As-received T8 (average) 7emsp;[average of samples 1 through 6]	546.9 (79.3)(a)	500.8 (72.6)(a)	8.6(a)
Standard deviation	12.0	17.4	0.3
T6 modified to resemble T8 (process 1) (Ref 10)	530.9 (77.0)(a)	482.7 (70.0)(a)	11.0(a)
Ref 11	524 (76.0)(a)	490 (71.1)(a)	10.7(a)

The percent elongation was not calculated for samples which broke outside the gage length. The position of the fracture was slightly away from the gage, and the samples never failed in the radius or in the grip
(a) Average numbers

Table 4 Definition of reaging processes for Al 2099

Aging processes for Al 2099 (C458)	
Aging process 1	Aging process 2
1. Aging at 120 °C for 12 h 2. Aging at 180 °C for 16 h	1. Aging at 120 °C for 12 h 2. Aging at 180 °C for 24 h

to the samples that were not exposed to a corrosive environment. However, the percent elongation was reduced. It was speculated that the samples that were not exposed to a corrosive environment would have had better results than the samples that were exposed. Further testing will be required to see why the corrosive environment provided better strength results for the exposed samples. It should be noted, however, that according to ASTM B117 (Ref 12), the exposure zone should be maintained at 35 °C. This temperature is not warm enough to have influenced the aging of the specimens in 168 h (1 week) during the salt fog test.

As retrogression temperature increases, the mechanical tensile strength values began to decrease over retrogression exposure time. Most of the samples at lower retrogression temperatures experienced (process 2) an increase in values at the 10-min exposure mark and then experienced a drop. As the temperature increased, the trend started to resemble a linear decrease.

Table 2 includes the data comparison for Al 2099 that has been subjected to the two-step heat treatment process (Ref 10) that did not undergo the RRA process nor salt spray exposure. Table 3 shows the tensile properties of samples that have not undergone the RRA process but were exposed to salt spray solution. From Table 2, for the salt spray exposure, the ultimate strength of the two-step T6 temper is only 6.2 MPa higher than the T8 standard (Ref 10) with equal yield strengths. Both conditions have almost the same ductility. This shows that with a two-step low temperature to high temperature T6 heat treatment process, it is possible to produce T8 results without the cumbersome pre-stretch that is required of the T8 temper (Ref 10). Table 3 also shows similar results as Table 2. In fact

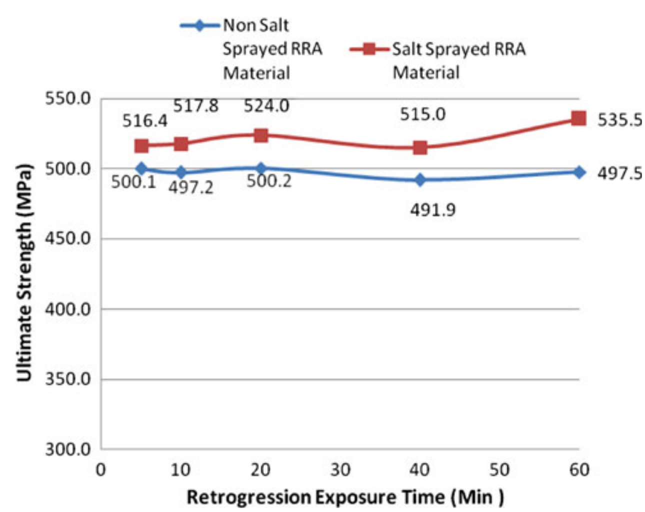

Fig. 1 Ultimate strength vs. retrogression exposure time in RRA material with retrogression exposure temperature at 200 °C after reaging process 1

the samples that have been exposed to the salt spray have shown improvement over the T8 temper strength values. The ultimate strength for the two-step T6 was 15.9 MPa higher than the T8 and the yield strength of the two-step T6 being 17.9 MPa higher than the T8 values (Ref 10). The salt-sprayed samples showed significant improvement in strength values over the samples not exposed to the corrosive environment. A slight drop in percent elongation is observed.

The samples that had a retrogression temperature of 200 °C, process 1 (Fig. 1, 2), showed a relatively linear data from 5 to 40 min. However, the strength values rose for both the ultimate strength and yield strength from 40 to 60 min. This rise could be a result of solute reversion (Ref 13). When comparing process 2 (Fig. 3, 4) to process 1 (Fig. 1, 2), they showed similar results. While process 1 had achieved its greatest strength at 60 min, process 2 achieved it at the 10 min mark. There was a slight general increase in strength values in process 1.

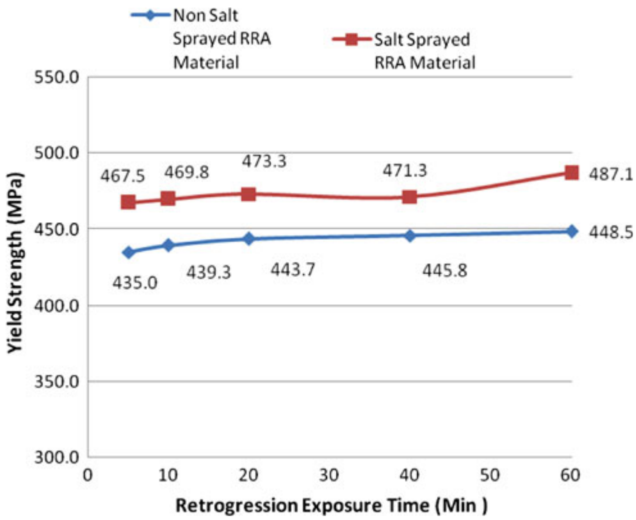

Fig. 2 Yield strength vs. retrogression exposure time in RRA material with retrogression exposure temperature at 200 °C after reaging process 1

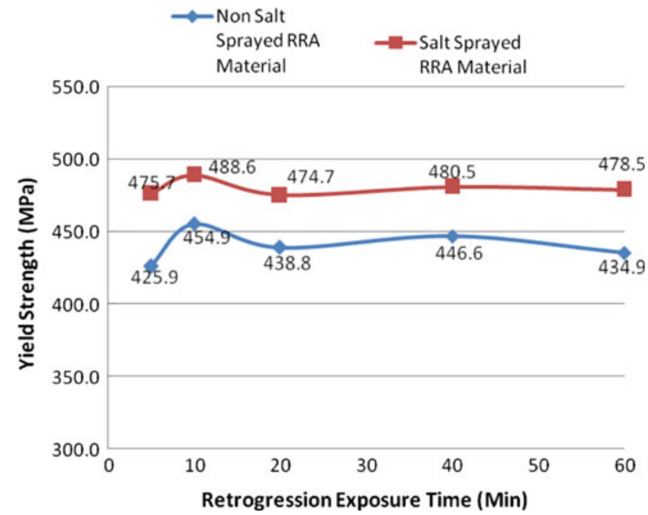

Fig. 4 Yield strength vs. retrogression exposure time in RRA material with retrogression exposure temperature at 200 °C after reaging process 2

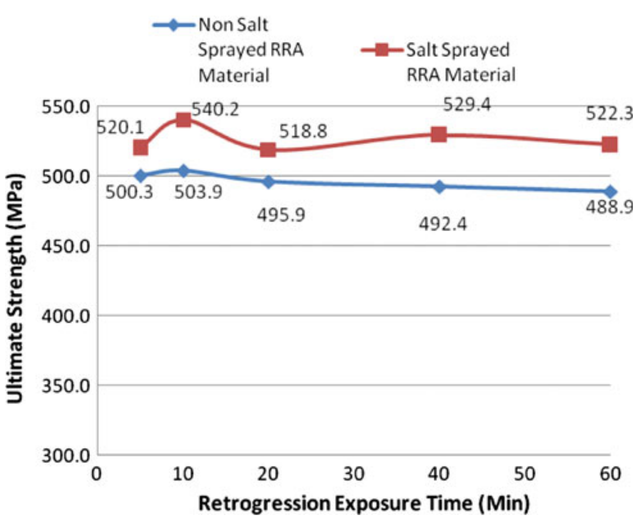

Fig. 3 Ultimate strength vs. retrogression exposure time in RRA material with retrogression exposure temperature at 200 °C after reaging process 2

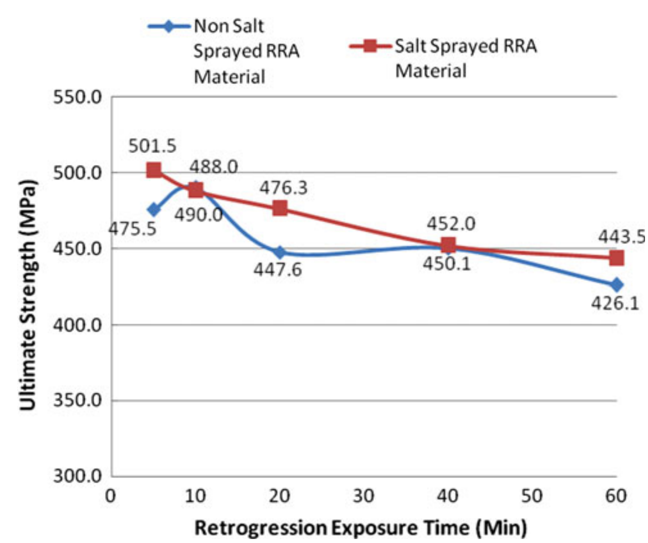

Fig. 5 Ultimate strength vs. retrogression exposure time in RRA material with retrogression exposure temperature at 250 °C after reaging process 1

Comparing the 200 °C (Fig. 1-4) with the 250 °C retrogression temperatures (Fig. 5-8) a significant change in the mechanical properties is observed. While the graphs from the 200 °C samples showed a relatively constant linear data, the 250 °C sample shows a declining path. For 250 °C process 1 (Fig. 5, 6), the ultimate strength showed a decreasing path. The yield strength experienced a rise in properties values at the 10 min mark followed by a continued drop in mechanical property value. At 250 °C process 2 (Fig. 7, 8), the ultimate strength follows a decreasing linear path. For the non-salt-sprayed samples, the yield strength experiences a slight rise in property values from 5 to 20 min before following a decreasing path. The salt-sprayed samples strength values followed a decreasing path through out. Samples that were exposed to the salt spray environment showed slightly better properties compared to the non-salt-sprayed samples. The mechanical tensile strength properties showed a steady decline as the retrogression temperature increased.

Table 5 and 6 present the percent elongation data for non-salt exposed and salt exposed, respectively. The percent elongation ranges from 8.8 to 16%. This means that RRA process did not significantly affect the percent elongation of the material. In Table 5, under the 240 °C process 2, all the samples broke out sides of the gage length. The reason for this is unclear; it is probably due to an experimental error in machining. Since the samples broke outside of the gage length, percent elongation was not calculated.

In the 10-min retrogression exposure time, process 1 (Fig. 9, 10), the tensile mechanical properties experience a dip at 240 °C. The strength values begin to rise again from 240

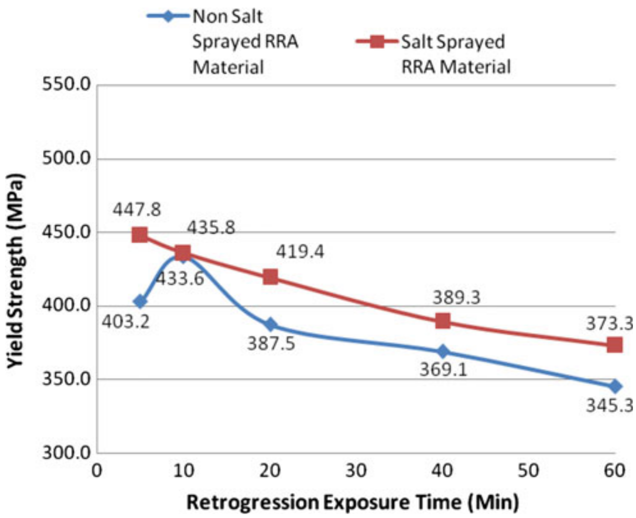

Fig. 6 Yield strength vs. retrogression exposure time in RRA material with retrogression exposure temperature at 250 °C after reaging process 1

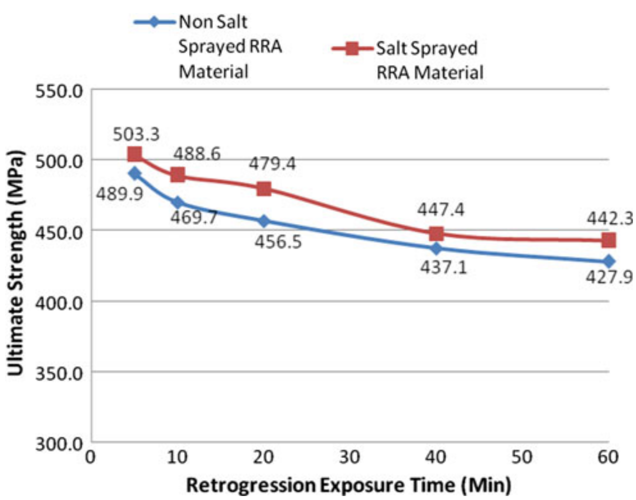

Fig. 7 Ultimate strength vs. retrogression exposure time in RRA material with retrogression exposure temperature at 250 °C after reaging process 2

to 250 °C. This rise could be due to solute reversion (Ref 13). Process 2 (Fig. 11, 12) does not follow the trend of process 1; instead, the ultimate strength values experience a steady decline. The yield strength values experience a slight plateau from 200 to 220 °C and then they decline again. The salt-exposed samples had better strength values.

In the 60-min retrogression exposure time, process 1 (Fig. 13, 14) and process 2 (Fig. 15, 16), the tensile strengths show initial high values which then quickly decrease until 240 °C. For the ultimate strength, the values for the salt-exposed and non-salt-exposed samples were almost equal at higher temperatures. The same is true for the yield strength values for process 1 but for process 2 the salt-sprayed samples had consistently higher values.

In Table 2, 3, and 5, the percent elongation was not calculated for some samples because fracture occurred outside the gage length. The position of the fracture was immediately

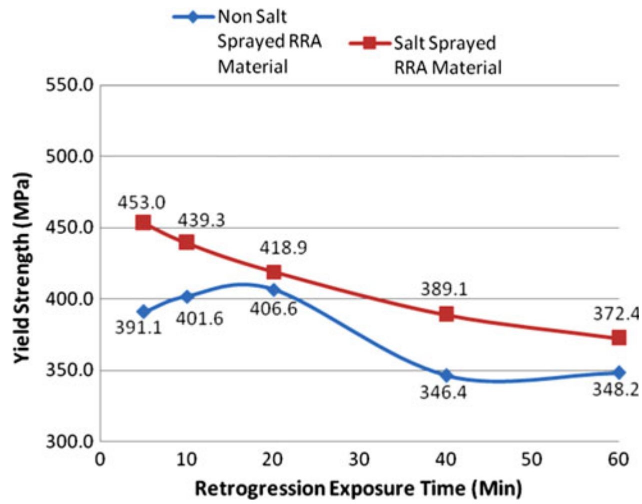

Fig. 8 Yield strength vs. retrogression exposure time in RRA material with retrogression exposure temperature at 250 °C after reaging process 2

Table 5 Percent elongation data for non-salt-exposed samples

Non-salt spray				
Percent elongation (process 1|process 2)				
	Temp., °C			
Time, min	200	220	240	250
5	16.0|11.2	10.4|11.2	11.4|N/A	11.2|11.4
10	8.7|9.6	10.0|11.2	12.0|N/A	9.8|9.8
20	10.0|7.5	13.0|11.0	10.3|N/A	8.8|10.0
40	9.5|8.1	9.6|11.3	10.2|N/A	10.8|9.1
60	10.3|8.8	10.7|11.2	11.2|N/A	10.1|9.1

The percent elongation was not calculated for samples which broke outside the gage length. The position of the fracture was slightly away from the gage, and the samples never failed in the radius or in the grip

Table 6 Percent elongation data for salt exposed samples

Salt spray				
Percent elongation (process 1|process 2)				
	Temp., °C			
Time, min	200	220	240	250
5	10.2|9.8	9.9|9.1	9.3|11.0	9.3|10.0
10	8.8|9.7	8.8|9.3	10.0|10.6	10.8|9.7
20	7.5|8.7	9.6|9.4	10.0|9.5	8.0|8.5
40	10.9|10.4	9.2|9.8	9.5|10.4	8.8|8.9
60	9.6|8.8	8.6|9.8	10.1|10.0	9.7|10.2

outside the gage and it never failed in the radius or in the grip. It is interesting to note that for alloy 2099 the percent elongation was not reduced significantly after the salt spray test. This is in contrast to the 2195 alloy (Ref 14) where there was a significant deterioration in the percent elongation after the salt spray

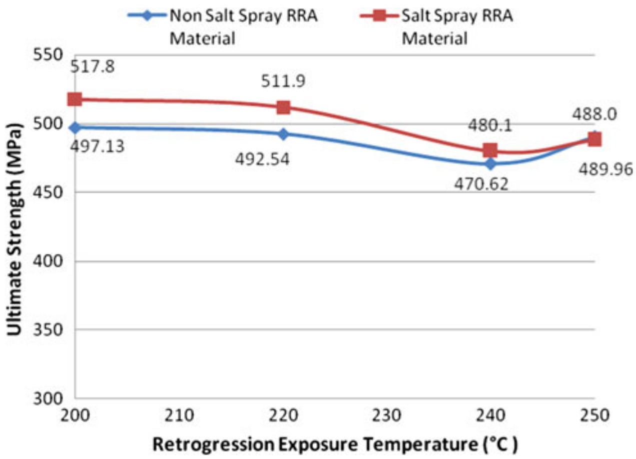

Fig. 9 Ultimate strength vs. retrogression exposure temperature in RRA material with retrogression exposure time at 10 min after reaging process 1

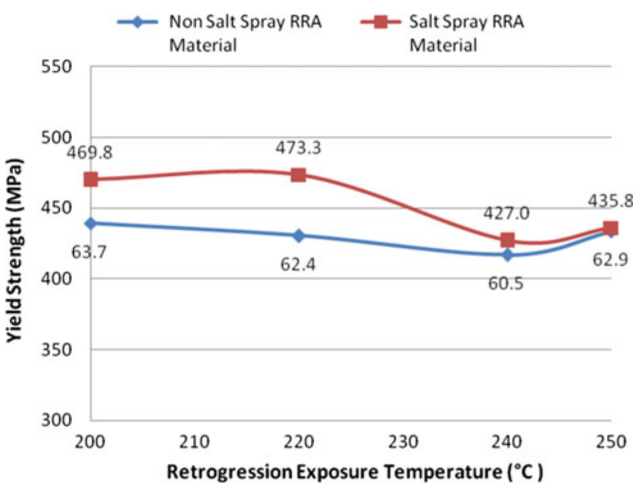

Fig. 10 Yield strength vs. retrogression exposure temperature in RRA material with retrogression exposure time at 10 min after reaging process 1

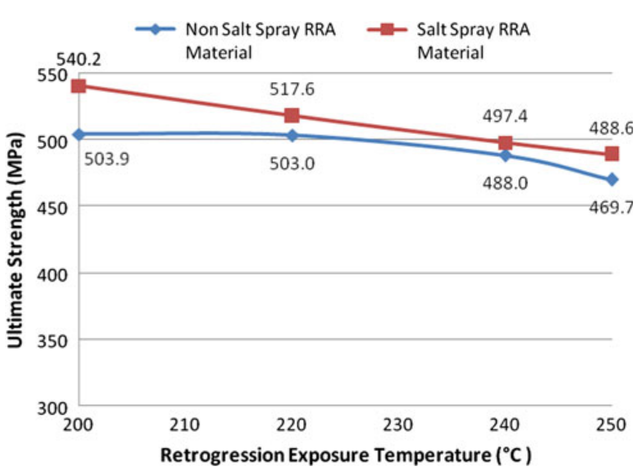

Fig. 11 Ultimate strength vs. retrogression exposure temperature in RRA material with retrogression exposure time at 10 min after reaging process 2

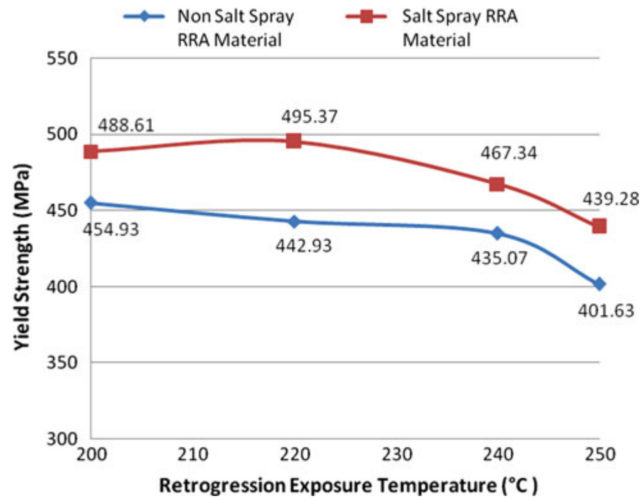

Fig. 12 Yield strength vs. retrogression exposure temperature in RRA material with retrogression exposure time at 10 min after reaging process 2 in non-salt-sprayed samples

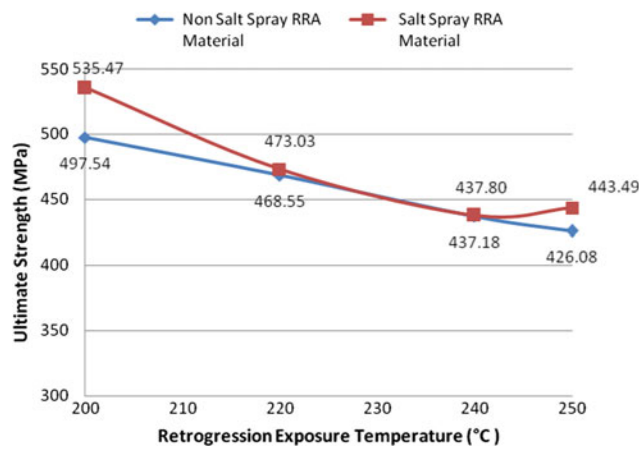

Fig. 13 Ultimate strength vs. retrogression exposure temperature in RRA material with retrogression exposure time at 60 min after reaging process 1

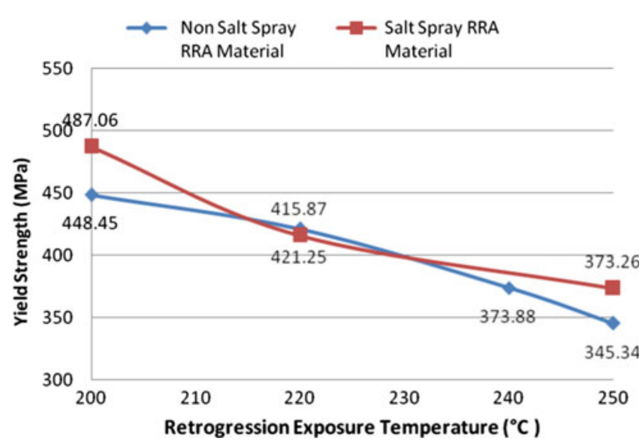

Fig. 14 Yield strength vs. retrogression exposure temperature in RRA material with retrogression exposure time at 60 min after reaging process 1

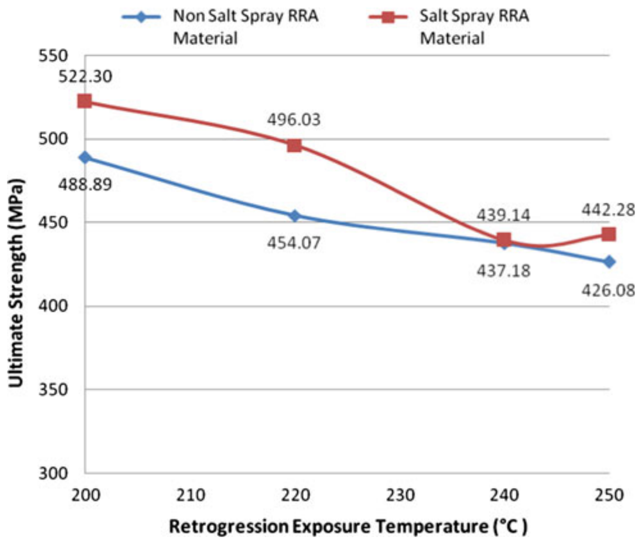

Fig. 15 Ultimate strength vs. retrogression exposure temperature in RRA material with retrogression exposure time at 60 min after reaging process 2

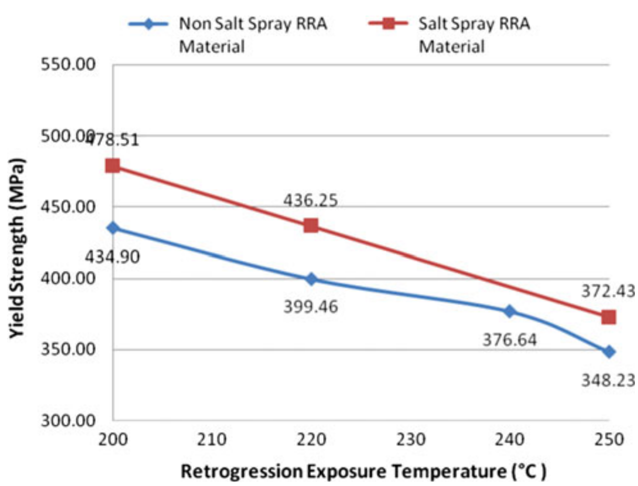

Fig. 16 Yield strength vs. retrogression exposure temperature in RRA material with retrogression exposure time at 60 min after reaging process 2

exposure. This appears to be a function of the material itself in terms of its chemical composition and thermomechanical history.

The microstructures of the fracture tensile samples (Fig. 17a-g) were examined at different magnifications (100×, 1000×, and 2000×). All the micrographs indicated a mixed mode fracture; there are areas of equiaxed dimples (ductility) and intergranular fracture (brittleness).

4. Summary

Retrogression and reaging had beneficial effects in terms of maintaining strength values as well as stress corrosion cracking

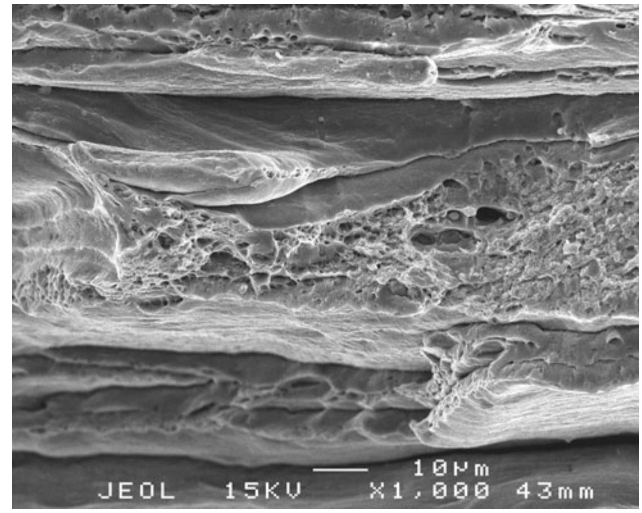

Fig. 17 SEM micrograph of 2099 alloy at 1000× magnification (Al 2099 process 2, 200 °C for 40 min)

resistance properties for Al 2099. The best RRA processes were at 200 °C for any time (5, 10, 20, 40, and 60 min). This is true for both processes 1 and 2. The RRA samples exhibited mechanical property values that were close to those of the as-received two-step T6 heat temper and the T8 temper. Samples that were exposed to the corrosive environment produced slightly better tensile results. As retrogression exposure time or temperature increased, the strength decreased. After comparing processes 1 and 2, it would appear that process 2 is marginally higher in strength values.

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