Design of Multistep Aging Treatments of 2099 (C458) Al-Li Alloy

M. Romios, R. Tiraschi, C. Parrish, H.W. Babel, J.R. Ogren, and O.S. Es-Said

(Submitted June 22, 2005)

Multistep artificial aging treatments coupled with various natural aging times for aluminum lithium 2099 alloy (previously called C458) are discussed to obtain mechanical tensile properties in the T6 condition that match those in the T861 condition, having a yield strength in the range of 414-490 MPa (60-71 ksi), an ultimate strength in the range of 496-538 MPa (72-78 ksi), and 10-13% elongation. Yield and ultimate tensile strengths from 90-100% of the strength of the as-received material (in the T861 condition) were obtained. The highest tensile strengths were consistently obtained with two-step, low-to-high temperature artificial aging treatments consisting of a first step at 120 °C (248 °F) for 12-24 h followed by a second step between 165 and 180 °C (329-356 °F) for 48-100 h. These T6-type heat treatments produced average yield and ultimate strengths in the longitudinal direction in the range of 428-472 MPa (62.1-68.5 ksi) and 487-523 MPa (70.6-75.9 ksi), respectively, as well as lower yield strength anisotropy when compared with the as-received material in the T861 condition.

1. Introduction

Al-Li C458 was developed in 1997. Research data indicate that it has superior physical and mechanical properties over traditional 2xxx and 7xxx series aluminum alloys, and it overcomes many shortcomings, especially mechanical anisotropy, of previous Al-Li alloys (Ref 1). Because of the alloy's maturity, it was designated 2099 by the Aluminum Association in 2004, and the alloy will be referred to by its current name for the rest of the manuscript.

When compared with its predecessor 2090, 2099 has less planar anisotropy, higher transverse ductility, excellent stress corrosion cracking resistance (SCCR), and excellent toughness, and like 2090 it has excellent cryogenic properties (Ref 2). Al-Li alloys are preferred to other aluminum alloys for aerospace applications primarily because they have a low density, offering 10-25% weight savings in some aerospace applications (Ref 1, 3). Al-Li 2099 has been found to save up to 14% weight on major structural components of aircraft wings (Ref 3) and 21% for some cryogenic tanks. Al-Li alloys generally have high specific modulus and excellent fatigue and cryogenic toughness properties, as compared with traditional aluminum alloys.

All of the mechanical tensile data collected from the T6 heat treatments reported here are compared with those of 2099 alloy in the as-received T861 condition, which has been solution treated, stretched 6%, and age hardened. In the T8 temper, the plastic deformation from the 6% preage stretch introduces dis-

M. Romios, R. Tiraschi, J.R. Ogren, and **O.S. Es-Said,** Loyola Marymount University, Mechanical Engineering Department, One LMU Drive Los Angeles, CA 90045; and **C. Parrish** and **H.W. Babel,** Boeing Company, 5301 Bolsa Ave, Huntington Beach, CA 92647. Contact e-mail: oessaid@lmu.edu. **Fig. 1** One-piece dome for a cryogenic tank (2219 alloy)

locations, which act as preferential nucleation sites in the matrix for the primary strengthening phase, T_1 (Al₂CuLi) (Ref 4). It was found that the nucleation of the T_1 precipitates, at dislocations induced by plastic deformation, enhances the strength and aging kinetics of 2099 (Ref 4). A key disadvantage of Al-Li alloys is the need for cold work to attain peak mechanical strength. The non-cold worked 2099 does not tend to form the $T₁$ precipitates in the matrix, only at the grain boundaries, and any T_1 precipitates in the matrix of 2099 are likely the result of dislocations remaining after the solution heat treatment (Ref 4). More abundant and finer precipitation of T_1 in the matrix of 2099 directly correlates to increased strength and ductility (Ref 4). In addition, cold working can add cost and complexity to the manufacturing of some parts, and in some aerospace applications, such as one-piece domes for cryogenic tank applications (Fig. 1), is not possible.

The aim of this study is to develop T6 aging treatments for 2099 alloy capable of achieving strength and ductility compa-

rable to that of the alloy in the T861 condition, primarily to eliminate the 6% preage stretch used in the T8 treatment. That is, complex shaped parts could be heat treated to attain a T6 strength comparable to the T8 strength without stretching. These T6 heat treatments could be used as replacements for T8 heat treatments on parts that are complexly shaped and cannot be cold worked in manufacturing.

U.S. Patent 4 861 391 (Ref 5) describes a method for producing an Al-Li alloy having a combination of enhanced strength and fracture toughness. The patent describes a two-step, low-to-high temperature artificial aging process. The process documented by Rioja et al. (Ref 5) involves aging at or below a first temperature of 93 °C (200 °F) for 12-100 h, then further aging at a second temperature above the first but below 219 °C (425 °F) until the desired strength is reached. When comparing tensile yield strengths of one-step versus two-step artificial aging, it was found that the strengths obtained from two-step aging were consistently higher. The patent hypothesizes that when solute atom clustering occurs in the first step, a more even distribution of variously sized strengthening precipitates form in the second step (Ref 5).

This study focuses on the design of aging treatments to maximize tensile strength while maintaining acceptable ductility. The results from mechanical testing of 2099 alloy plate material subjected to several variations of aging treatments are discussed. The data show that tensile strengths comparable to that of the material in the T861 condition can be obtained by a T6 type aging treatment not requiring the preage stretch or any form of cold work. The aging treatments discussed are designed based on the principle that, in the artificial aging process, low temperatures tend to enhance nucleation of strengthening precipitates and higher temperatures tend to enhance the growth of such precipitates (Ref 5). The goal of the study is the optimization of such aging treatments where the combination of times and temperatures produce high ultimate and yield tensile strengths with minimum sacrifice in ductility.

2. Experimental Procedure

The 2099, 1.27 cm (0.5 in.) plate material in this study was supplied by Alcoa (Pittsburgh, PA). The nominal chemical composition of the aluminum alloy by weight percent is 2.4- 3.0Cu, 1.6-2.0Li, 0.4-1.0Zn, 0.1-0.5Mg, 0.1-0.5Mn, 0.05- 0.12Zr, 0.02-0.5Si, 0.03-0.7Fe, and 0.1Ti. Most of the coupons used in this study were machined from 2099 plate material in the T861 condition, while others were machined from plate material in either the T351 or the TF condition. Coupons were machined into tensile bars 2.54 cm (1 in.) wide, 20 cm (8 in.) long, and either 0.635 cm (0.25 in.) or 1.27 cm (0.5 in.) thick, with a gauge width of 1.27 cm (0.5 in.). Where material permitted, sets of three coupons each were machined at orientations of 0° (longitudinal), 45°, and 90° (transverse) relative to the plate-rolling direction, for a maximum of nine coupons per aging treatment. Results represent the average of three tensile tests for each orientation. Tensile tests were performed on Tinius Olsen Universal (Tinius Olsen, Horsham, PA) and Instron 4505 testing machines (Instron, Norwood, MA). Separate hardness bars were prepared and hardness tests were performed on the Rockwell B hardness scale using the Wilson/Rockwell hardness tester. Forty-nine heat-treatment experiments were performed in total. Fourteen of those that resulted in the highest tensile strengths are discussed here and compared with the plate material in the as-received T861 condition.

3. Results and Discussion

The experimentally designed heat treatments summarized here demonstrate that 2099 alloy plate material with ultimate and yield tensile strength comparable to that in the T861 condition can be obtained with a T6 type heat treatment. The processes for several of the best performing heat treatments are summarized in Table 1, while the mechanical properties of those processes are summarized in Table 2. In Tables 1 and 2, Heat Treatment 0 identifies the as-received properties of the C458-T861 1.27 cm (0.5 in.) plate material, with tensile strengths and elongation as provided by Alcoa (Ref 6). With the exception of Heat Treatment 0, all the heat treatments summarized in Tables 1 and 2 included a solution treatment of 549 °C (1020 °F) for 2 h immediately followed by quenching in room temperature water.

The most successful artificial aging treatments in terms of tensile strength were two-step, low-to-high temperature processes. The objective of these two-step artificial aging treat-

ments is to nucleate precipitates in the first step and coarsen the precipitates in the second step. In one study that focused on optimizing the two-step artificial aging treatment, the results from the tensile tests pointed to an optimal temperature combination of 120 °C (248 °F) in the first step and 180 °C (356 °F) in the second step (Ref 7). Various combinations of first and second step aging times were tested, including a three trial experiment that aged samples at 120 °C for 12 h and then varied the second step aging time at 180 °C (356 °F) for 24, 50, and 100 h (Heat Treatments 1, 2, and 3) (Ref 7).

Tensile strength was found to increase when going from 24-50 h for the second aging step. For example, the yield strength in the 45° orientation increased by about 5% from 413 MPa for Heat Treatment 1 to 433 MPa for Heat Treatment 2, exceeding that of the T861 condition (Heat Treatment 0 in Table 2). However, the yield strength then decreased for Heat Treatment 3, dropping about 4% from 433 to 415 MPa in the 45° orientation. This suggests that slight overaging occurs when aged for up to 100 h at 180 $^{\circ}$ C (356 $^{\circ}$ F) (Ref 7).

Heat Treatment 4 tested a two-step artificial aging treatment of 120 °C (248 °F) for 24 h and 180 °C (356 °F) for 48 h (Ref 7). Comparing Heat Treatment 4 with Heat Treatment 2 shows a slight improvement in tensile strength, suggesting that the longer first step aging time allows for an increase in strength. For example, the yield and ultimate strengths in the 0° orientation increased from 441 and 489 MPa for Heat Treatment 2 to 456 and 504 MPa for Heat Treatment 4, respectively. Heat Treatment 5 is similar to Heat Treatment 4 with a first artificial aging step of 120 \degree C (248 \degree F) for 24 h but with a second artificial aging step at 180 °C (356 °F) for 75 h, which is 50% longer than that of Heat Treatment 4 (Ref 8). However, the yield and ultimate strength values decreased by 2.4-5% between Heat Treatments 4 and 5. This comparison suggests again that the high temperature of 180 °C (356 °F) when used as the second step artificial aging temperature for times greater than 50 h results in overaging and reduction in tensile strength.

Heat Treatment 4 resulted in tensile strength values that were from 93 to 98% of the strength in the as-received T861 condition. The yield and ultimate strengths in the 0° orientation measured from this aging treatment were 456 and 504 MPa, respectively, compared with 490 and 524 MPa for the T861 condition (Ref 7). A later attempt to optimize this aging treatment for maximum tensile strength was performed with the same first step artificial aging time and temperature of 120 °C (248 °F) for 24 h but with a second artificial aging step at a slightly lower temperature of 165 °C (329 °F) to avoid overaging (Ref 9). This aging treatment (Heat Treatment 6) resulted in a 1.5-3.5% increase in yield and ultimate tensile strength as compared with Heat Treatment 4. For example, the yield and ultimate strengths in the 0° orientation increased from 456 and 504 MPa for Heat Treatment 4 to 472 and 523 MPa for Heat Treatment 6, respectively.

Heat Treatment 6 produced the highest tensile strength of all the heat treatments tested in this study. Performed on 1.27 cm (0.5 in.) plate material in the T861 starting condition, this heat treatment included a two-step artificial aging schedule of 120 °C (248 °F) for 24 h and 165 °C (329 °F) for 100 h (Ref 9). This resulted in strength values that were from 96-99% of the strength in the T861 condition, including an average yield strength in the 45° orientation that was 11 MPa greater than that of the as-received material (yield strength of 425 MPa for Heat Treatment 6 compared with 414 MPa for the as-received T861). The yield and ultimate strengths in the 0° orientation measured from Heat Treatment 6 were 472 and 523 MPa, respectively, compared with 490 and 524 MPa for the T861 condition. However, when this heat treatment was performed on plate material in the T351 starting condition (Heat Treatment 14), the strength was 5-6% lower (Ref 10). This suggests that the starting condition may make a difference in the materials response to the heat treatment.

While the majority of artificial aging treatments tested were of the two-step, low-to-high temperature variety, one of the three-step processes is worth mentioning. This heat treatment, indicated as Heat Treatment 7 in Tables 1 and 2, was performed on 1.27 cm (0.5 in.) plate material initially in the T861 starting condition. The three-step artificial aging treatment consisted of 140 °C (284 °F) for 18 h, 180 °C (356 °F) for 48 h, and 110 °C (230 °F) for 72 h (Ref 11). Heat Treatment 7 resulted in strengths that were from 95% to 98% of that in the as-received T861 condition. The yield and ultimate strengths in the 0° orientation measured from this aging treatment were 463 and 510 MPa, respectively, compared with 490 and 524 MPa for the T861 condition (Ref 11).

Heat Treatment 7 was designed as a combination of high-

to-low and low-to-high temperature processes. The first step, midtemperature treatment for a short duration of time is performed so that the solute atoms cluster to form nuclei for the formation and growth of precipitates, while the second step, high temperature treatment causes the precipitates to grow (Ref 5). The third step, low temperature for a long duration of time, is intended to cause new precipitates to form, but at a much slower rate and more evenly distributed (Ref 12).

Three of the most successful high-to-low temperature twostep aging treatments (Heat Treatments 8, 9, and 10) resulted in significant strength values. Heat Treatment 8 was performed with a first step temperature at the lower 154 $^{\circ}$ C (309 $^{\circ}$ F), versus 163 °C (325 °F) for Heat Treatments 9 and 10 (Ref 10). Consequently, Heat Treatment 8 produced 11% lower yield strength but 6% higher ultimate strength values in the 0° orientation when compared with Heat Treatments 9 and 10. The ultimate strength values measured for Heat Treatment 8 in the 0° and 45° orientations were 95 and 90% of the T861 condition, respectively (Ref 10). However, the yield strength values for Heat Treatments 8, 9, and 10 were significantly below the goal, especially in the 45° orientation.

It was observed that in all of the high-to-low temperature two-step artificial aging treatments, the yield strength values, especially in the 45° orientation where no coupons averaged over 400 MPa (58 ksi), were significantly lower than those of the as-received. Coincidentally, all of the high-to-low temperature experiments were performed with a natural aging time of 96 h and with plate material of a different lot in either the T351 or TF starting condition (Ref 10).

It is not clear whether it is the high-to-low temperature aging process, the long natural aging time, the lot chemistry, or the starting condition that resulted in the significantly low yield strength values for the 45° orientation. Tensile data tend to show that the longer natural aging time does not adversely affect the yield strength of the alloy. All of the tensile coupons in the 45° orientation for the high-to-low temperature artificial aging treatments were taken from the TF plate material, which perhaps explains the significantly lower strength values recorded for this orientation from these aging treatments. Most of the heat treatments performed with plate material of the lot in the T351 condition resulted in lower than desired yield strength values, and those performed with plate material of the lot in the TF condition resulted in even lower yield strength values.

Heat Treatments 11, 12, and 13 are all characterized by having a first step artificial aging time at 121 $^{\circ}$ C (250 $^{\circ}$ F) of 48 h, twice as long as previous studies (Ref 10). Heat Treatments 11 and 12 perform well in the 0° orientation, with yield strength values reaching 86-88% of the T861 condition and ultimate strengths reaching 93% of the T861 condition. The artificial aging schedule for Heat Treatments 11 and 12 is similar to Heat Treatment 6, with the exception of the longer first step time (48 h as compared with 24 h) and a slightly lower second step temperature (154 °C as compared with 165 °C). In addition, Heat Treatments 11 and 12 were performed with plate material that was initially in the T351 condition, as opposed to the T861 condition plate material that was used for Heat Treatment 6 (Ref 10). Consequently, the tensile strength values in the 0° orientation for Heat Treatments 11 and 12 were 6.5- 10.6% lower when compared with those of Heat Treatment 6. This difference in tensile strength may be a result of the starting condition of the plate material, as was the case when Heat Treatments 6 and 14 were compared. Although, the longer first step aging time may also have been a factor.

Fig. 2 Effect of first step artificial aging temperature on tensile strength. Data points are from several low-to-high temperature artificial aging treatments consisting of various first and second step times and temperatures.

Comparing Heat Treatments 11 and 12 reveals that natural aging time does not have a significant effect on the tensile strength of the artificially aged material. Heat Treatments 11 and 12 consisted of the same solution treatments and same artificial aging schedule, but Heat Treatment 11 included a natural aging time of 2 days, while Heat Treatment 12 included a natural aging time of 24 days (Ref 10). The yield and ultimate strength values in the 0° orientation for these two heat treatments were within 3% of each other, with Heat Treatment 12 having slightly lower yield and ultimate strengths. Additional heat treatments with natural aging times of 2 and 24 days, but with identical solution treatments and artificial aging schedules, revealed the same trend (Ref 10). Subsequent natural aging studies did show that the hardness of samples increased significantly within the first two days and continued to increase slightly up to 10 days (Ref 10). However, in comparing Heat Treatments 11 and 12, when samples are artificially aged, 2-4 days of natural aging preformed after solution treatment and prior to artificial aging is sufficient to obtain T6 peak strength.

Heat Treatment 13, which is similar to Heat Treatments 11 and 12 with the exception of a very short second step artificial aging time (24 h) at a slightly higher temperature (177 °C) (Ref 10), resulted in tensile strength values in the 0° orientation that were about 5% lower than those of Heat Treatments 11 and 12. This is likely a consequence of the short second step aging time not allowing the precipitates to grow to a significant size. The better performing heat treatments included second step artificial aging times that were much longer.

Figures 2-4 are plots of tensile strength data obtained from several low-to-high temperature artificial aging treatments consisting of various combinations of 1st and 2nd step aging times and temperatures. Comparing the first-step artificial aging temperature versus tensile strength (Fig. 2) appears to indicate that the strength peaks between temperatures of 120-150 °C (248- 302 °F) (Fig. 4). A similar pattern is obtained when the firststep artificial aging time is compared with tensile strength (Fig. 3), indicating a possible peak first-step aging time between 24 and 48 h. The second-step artificial aging temperature appears to produce maximum tensile strength between 160 and 180 °C (320 and 356 °F) (Fig. 4). The second-step artificial aging time appears to be less influential on the resulting strength, but is dependent on the aging temperature. It is recommended that the region of the first step aging cycle between temperatures of 120-145 °C (250-290 °F) and durations of 24-48 h, along with

Fig. 3 Effect of first step artificial aging time on tensile strength. Data points are from several low-to-high temperature artificial aging treatments consisting of various first and second step times and temperatures.

Fig. 4 Effect of second step artificial aging temperature on tensile strength. Data points are from several low-to-high temperature artificial aging treatments consisting of various first and second step times and temperatures.

a complementary second step cycle, should be investigated to pinpoint a possible peak-age T6 condition.

In the majority of the best performing T6 heat treatments, the percent elongation at fracture was typically 20% less than that of the material in the T861 condition. This may be caused by cracks or imperfections on the surface of the coupons. Another possible explanation for the lower than desired ductility may be that longer times at the higher temperatures caused the precipitates at the grain boundaries to coarsen. This has the effect of immobilizing the slip systems in the lattice structure and increasing strength, but reducing ductility. The lower than desired elongation is a trend consistent with many of the heat treatments of this study, particularly those resulting in the highest strength values.

The anisotropy of Al-Li alloys has been noted as a disadvantage. Anisotropy is quantified here as the percent difference between the highest and lowest values of strength among the 0°, 45°, and 90° orientations. The anisotropy in both yield strength and ultimate strength were computed and compared with the anisotropy of the as-received T861 condition. The best heat-treatment processes reported here actually show an improvement in anisotropy over the as-received material, particularly in yield strength. The as-received material exhibited 15% anisotropy in yield strength, whereas the best two-step, lowto-high artificial aging treatments exhibited between 5 and 10% anisotropy in yield strength. The anisotropy in ultimate strength was on average the same as the as-received material, ranging from 4.5 to 9.3%. The high-to-low, two-step artificial aging treatments, which exhibited significantly low yield strength in the 45° orientation, showed very high anisotropy. The percent anisotropy is summarized in Table 2.

4. Conclusions

T6 heat treatment processes, consisting of multistep artificial aging treatments, of 2099 Al-Li alloy can be optimized to produce tensile strength that is comparable to that of the material in the T861 condition. Tensile and yield strengths of up to 99% of the strength in the T861 condition can be obtained.

In general, higher aging temperatures and longer aging times result in higher strengths, although strength will drop and ductility may be sacrificed if over-aging occurs at higher temperatures. However, when considering multistep aging processes, care should be taken in choosing times and temperatures for each step; longer times and higher temperatures are a general improvement in strength but should be optimized to produce the desired material properties.

The multistep artificial aging treatments that produce excellent ultimate and yield tensile strength consistently result in considerably less ductility than that of the material in the T861 condition.

The yield strength anisotropy recorded for the multistep artificial aging treatments was significantly lower than that of the material in the T861 condition, a considerable achievement for overcoming one noted disadvantage of the T861 condition.

Low yield strengths, especially in the 45° orientation, were experienced when the material was aged with a high-to-low temperature artificial aging treatment, where the first-step aging temperature is higher than the second step. However it is not clear whether the lower yield strength was the result of the artificial aging treatment or the TF starting condition of the plate material.

Acknowledgments

The authors gratefully acknowledge the National Science Foundation and their Research Experiences for Undergraduates program (Grant No. EEC-0353668), Alcoa for their donation of materials, Loyola Marymount University and the Boeing Company for their sponsorship of the research, and all the students and faculty involved in the generation of this work.

References

- 1. R.G. Buchheit, D. Mathur, and P.I. Gouma, Grain Boundary Corrosion and Stress Corrosion Cracking Studies of Al-Li-Cu Alloy AF/C458, Ohio State University, Columbus, OH, undated
- 2. D.C. Vander Kooi, W. Park, and M.R. Hilton, Characterization of Cryogenic Mechanical Properties of Aluminum-Lithium Alloy C458, *Scr. Mater.,* Vol 41 (No. 11), 1999, p 1185-1190
- 3. R.J. Rioja, C.J. Warren, M.D. Goodyear, M. Kulak, and G.H. Bray, Al-Li Alloys for Lower Wings and Horizontal Stabilizer Applications, *Mater. Sci. Forum,* Vol 242, 1997, p 255-260
- 4. B.M. Gable, A.A. Csontos, and E.A. Starke, The Role of Mechanical Stretch on Processing-Microstructure-Property Relationships of AF/C 458, *Mater. Sci. Forum,* Vol 331-337, 2000, p 1341-1346
- 5. R.J. Rioja, E.L. Colvin, A.K. Vasudevan, and B.A. Cheney, Aluminum Alloy Two-Step Aging Method and Article, U.S. Patent No. 4 861 391, 1989
- 6. G.B. Venema and R.J. Rioja, The Manufacture of C458 Plate at Davenport Works and Lot Release Properties, Proceedings from the Aluminum-Lithium Workshop, ALCOA and Air Force Research Laboratory (AFRL), Wright-Patterson AFB, Bass Lake Lodge, OH, 1998
- 7. C. Parrish, J. Barba, H.M. Oh, J. Peraza, J. Foyos, E.W. Lee, and O.S. Es-Said, Alternate Heat Treatments of C458 Aluminum Lithium Alloy, *Mater. Sci. Forum,* Vol 331-337, 2000, p 655-662
- 8. E. Acosta, A. Dakessian, E. Monge, and C. Parrish, Design Process of C458 Aluminum Lithium Alloy, Loyola Marymount University, Los Angeles, CA, 2000
- 9. M. Romios, R. Suchit, R. Nahman, and C. Jones, Heat Treatment

Design Process for C458 Aluminum Lithium Alloy, Loyola Marymount University, Los Angeles, CA, 2000

- 10. R. Tiraschi, F. Gaxiola, K. Heung, H. Nassar, H. Babel, C. Parrish, J. Foyos, J. Ogren, and O.S. Es-Said, On Optimizing the Mechanical Strength of C458 Al Alloy for Cryogenic Tank Applications by Various Heat Treatments, Loyola Marymount University, Los Angeles, CA, 2003
- 11. N. Abourialy, K. Trechter, and G. Wallace, Experimental Design of a Heat Treatment Process for C458-T6 Aluminum-Lithium Alloy, Loyola Marymount University, Los Angeles, CA, 2000
- 12. R.J. Rioja and R.S. James, Heat Treatments of Aluminum-Lithium Alloys, U.S. Patent No. 5 076 859, 1991