Chapter 25 – CORROSION CONTROL USING RETROGRESSION AND RE-AGING (RRA)

D. Raizenne and X. Wu
Institute for Aerospace Research
National Research Council of Canada
Ottawa, Ontario
CANADA

P. Sjöblom, R. Rondeau and S. Kuhlman
University of Dayton Research Institute
Dayton, Ohio
USA

ABSTRACT

Stress Corrosion Cracking (SCC) of primary structural components manufactured from Al 7075-T6 forgings, extrusions and plate products continues to plague aging aircraft. A two-step heat treatment, Retrogression and Re-Aging (RRA), applied to 7XXX series aluminum alloys in the T6 temper has been shown to greatly enhance the SCC resistance of these materials with minimal trade-off in strength. The RRA technology has reached a level of technical maturity where it can be transitioned to actual aircraft components. There are two ways that the RRA heat treatment process can be carried out. The shop RRA process utilizes conventional heat-treating equipment such as oil bath and furnace. The in-situ RRA process uses state-of-the-art hot bonder heat conduction technology coupled with real-time dissolution kinetics algorithms to heat treat selected areas of large parts. This paper describes the development of both the shop and in-situ RRA processes.

25.1 INTRODUCTION

The aluminum alloy 7075 in the peak aged T6 heat treatment condition has been widely used for aircraft structural applications. Corrosion damage in the form of Stress Corrosion Cracking (SCC), exfoliation and pitting is often the reason why 7075-T6 components are replaced. When improved corrosion resistance is required, alloy Al 7075 is often used in the over-aged temper T73, but in comparison to 7075-T6, there is a strength penalty of 10 to 15%, which precludes the use of the T73 temper when material substitution is considered. An alternative is to perform a retrogression and re-aging heat treatment on parts in the T6 condition. The RRA process has been shown to improve corrosion resistance to levels approaching those of the T73 condition. An RRA heat treatment will however result in a five to seven percent reduction in tensile strength from the T6 temper. An RRA heat treatment involves two steps: a **retrogression** step in the preferred temperature range of 180 to 200°C for 30 to 40 minutes, followed by a **re-aging** step at 120°C for 24 hours. The RRA process was first developed by Cina and Gan [1],[2] in the mid 1970s and was further developed at the National Research Council of Canada (NRC) by Wallace et al. [3]-[9] to the more practical time and temperature combinations described above. The RRA process has been successfully applied to extrusions and aircraft components up to 0.75 inch (20 mm) thick [10]. A comprehensive review of RRA work is provided in Reference [11].

The RRA process is illustrated through a series of metallurgical transformations in Figure 25-1. Starting in the T6 condition, retrogression is applied in the range 180 – 200°C to cause hardness and yield strength to fall rapidly through Stage 1 as Guinier-Preston (G-P) zones dissolve and the solid solution is enriched in the strengthening elements. Stage 2 is a transient period of hardness recovery as the remaining η‘ grows to a near optimum size distribution, but it begins to fall again as this η‘ coarsens excessively and begins

transforming to η. In Figure 25-1, it is important to note that the electrical conductivity and corrosion resistance increase steadily with hold time at the retrogression temperature. Therefore, if the goal is to increase corrosion resistance with minimal loss of original strength, then retrogression should be carried out to point D in Figure 25-1. The recovery of strength during re-aging may be due to the reformation of G-P zones or the formation of additional η‘ directly from the supersaturated solid solution or via the initial formation of G-P zones. The net result of the RRA process is a material with a strength slightly below that of a single step aged T6 material together with resistance to environmentally assisted cracking equivalent to that of an over-aged T73 material. Temperatures at the low end of the range quoted above require longer times to reach the optimum microstructural condition, which make it easier to achieve temperature uniformity in thicker section parts and greater uniformity of properties.

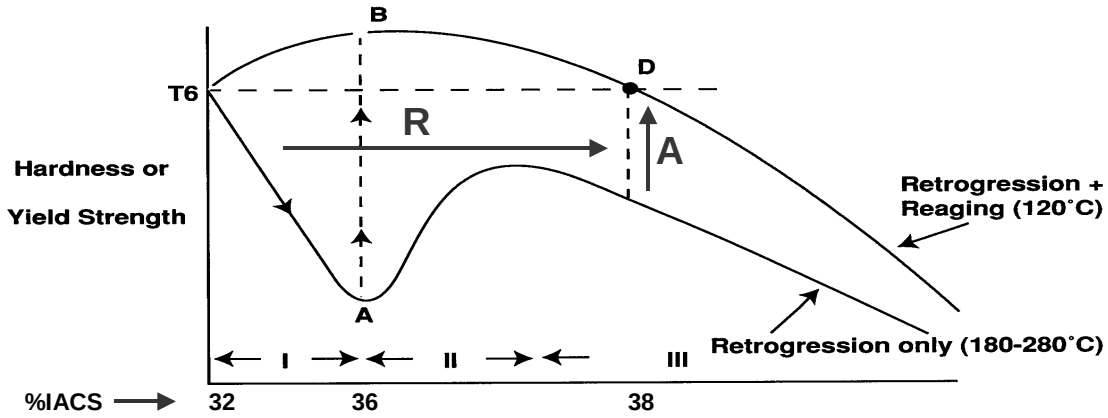


Figure 25-1: Schematic of the Retrogression and Re-Aging Process.

From 1999 to 2005 the USAF (through UDRI)), the Canadian Department of National Defence and NRC carried out a joint program to further develop the RRA process so that is could be applied to service components that were particularly susceptible to stress corrosion cracking. Al 7075-T6 extrusions and forgings with histories of SCC damage were identified on the C-130 and C-141 aircraft as ideal candidates for the RRA process [12]-[18].

25.2 KINETICS MODELLING OF THE PRECIPITATION PROCESS IN 7000 SERIES ALUMINUM ALLOYS

The 7000 series aluminum alloys are precipitation hardenable alloys. The peak strength is developed through the T6 temper while the T73 temper produces superior corrosion resistance. These temper specific properties are controlled by the following precipitation sequence [19]:

Solid Solution →	Guinier-Preston (GP) Zones →	η‘ →	η →	T	(1)
	Metastable	Metastable	Pseudostable	Equilibrium	
Particle Size	35 Å	200x50 Å	500 Å	----	
Solvus	150°C	250°C	370°C	190°C	

G-P zones are essentially coherent spherical solute clusters of Zn, Mg and Cu, while η‘, $MgZn_2$ or more correctly $Mg(ZnCuAl)_2$ appears as discrete platelet particles that are semi-coherent with the matrix and η is a non-coherent form of the same phase appearing as rods or plates. Other phases such as Tau-$Mg_{32}(AlZn)_{49}$

may appear at longer aging times while primary S-phase (Al_2CuMg) may appear in some of the newer alloys such as Al 7050 with high Cu and Mg contents.

Let x_1, x_2 and x_3 denote the relative phase fraction of G-P zones, η‘ and η precipitates, the above precipitation sequence, Equation (1) (neglecting formation of the T phase), can be reasonably well described by a set of differential equations, based on the first-order reaction kinetics, as:

$$\frac{dx_i}{dt} = k_{if}(1-\sum_{j=i}^{3} x_j) - k_{id}x_i \qquad \text{(i = 1,2,3)} \qquad (2)$$

where k_{if} and k_{id} are the formation and dissolution rate constants for the i-th precipitate type (i = 1, 2, 3) respectively. This set of kinetics equations recognizes the order of thermodynamic stability of each precipitate. For each precipitate, the formation rate is set to null above its solvus temperature.

Equation (2) allows us to evaluate the total phase content in a 7XXX aluminum alloy at different heat treatment conditions, and thus to find optimized phase fractions that can yield a good combination of strength and stress corrosion resistance. The set of fraction numbers (G-P zone dissolution and η‘ and η formation) can then be used to control the actual retrogression and re-aging heat treatment in ‘real time’ to ensure that the desired properties are obtained in practice. The key to this process is monitoring the part’s temperature during the RRA process.

Depending upon the part’s as-received electrical conductivity, threshold levels of η‘ and η are used to determine the optimum time for a retrogression treatment [20]. Each retrogression treatment is thus optimized for strength and corrosion resistance. This is illustrated in Figure 25-2 where the heat-up profiles are shown for three parts of similar shape heated in three different air-recirculating ovens. Optimized retrogression treatments (ramp to retrogression temperature, hold and cool to 50°C) ranged from 50.5 to 72.5 minutes. It is very important to understand the role of convective and conductive heat transfer – thus the requirement to monitor the temperature of each part using a device such as a thermocouple.

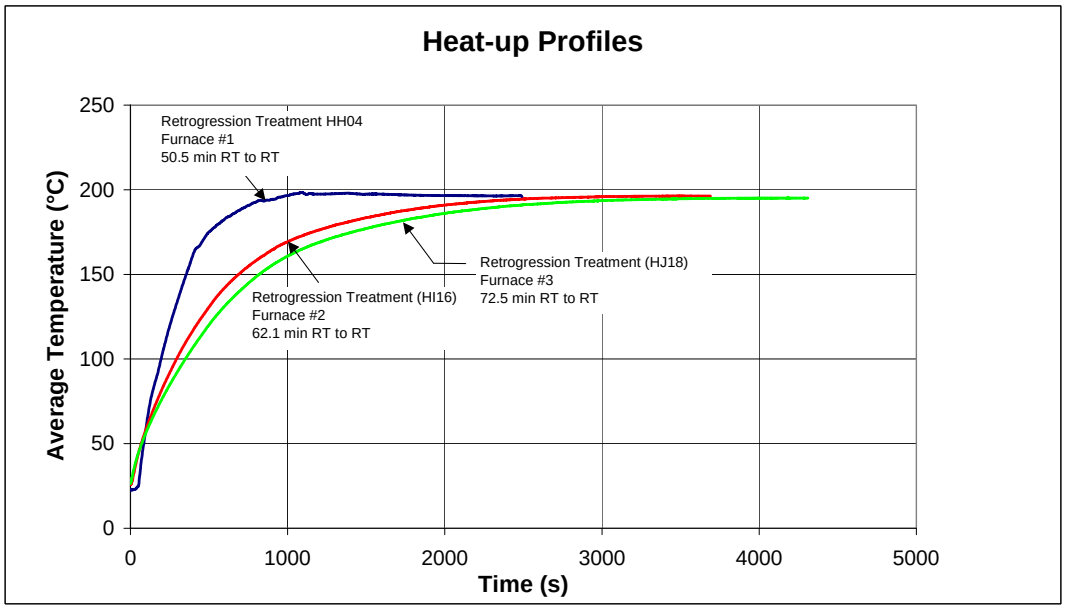


Figure 25-2: Comparison of Heat-Up and Retrogression Times for C-130 Sloping Longeron Sections in Three Different Air-Recirculating Furnaces. By using real-time kinetics modelling, the three sections were optimized for strength and corrosion resistance.

25.3 MATERIAL TEST PROGRAM

The objectives of the RRA material test program were twofold. The first objective was to verify the improvement in corrosion resistance through exfoliation and stress corrosion cracking tests. The second objective was to ensure that the drop in static strength caused by the RRA process did not lead to strength below minimum specified levels:

- Increase the electrical conductivity from 32 – 34 % IACS to a minimum of 38% IACS;
- Improve exfoliation corrosion rating to EXCO A;
- Improve SCC resistance to the T73 temper;
- Meet MIL-HDBK-5H A-Basis allowables for tensile strength, yield strength and ductility;
- Maintain T6511 temper fracture toughness; and
- Maintain T6511 temper fatigue crack growth rates.

For the material test program 2000 tests were carried out. They include corrosion tests, tensile tests, compression tests, bearing tests, fracture and fatigue tests, warpage and distortion tests, electrical conductivity and hardness tests. The effect of the RRA process on protective coatings such as anodizing was also evaluated.

25.4 RRA PROCESS DEVELOPMENT

Two RRA treatment processes were developed. The first, an immersion process, developed at UDRI, used an oil bath to carry out RRA treatments on small parts such as the C-130 Paratrooper Door Longeron (PDL). The second is an '*in situ*' process developed at NRC and UDRI. The in-situ process shown in Figure 25-3 uses a 16 zone hot bonder that is coupled with the 'real-time' dissolution kinetics algorithms described above to heat treat 'selected' areas of large parts such as the C-130 sloping longeron and the C-141 landing gear main frame.

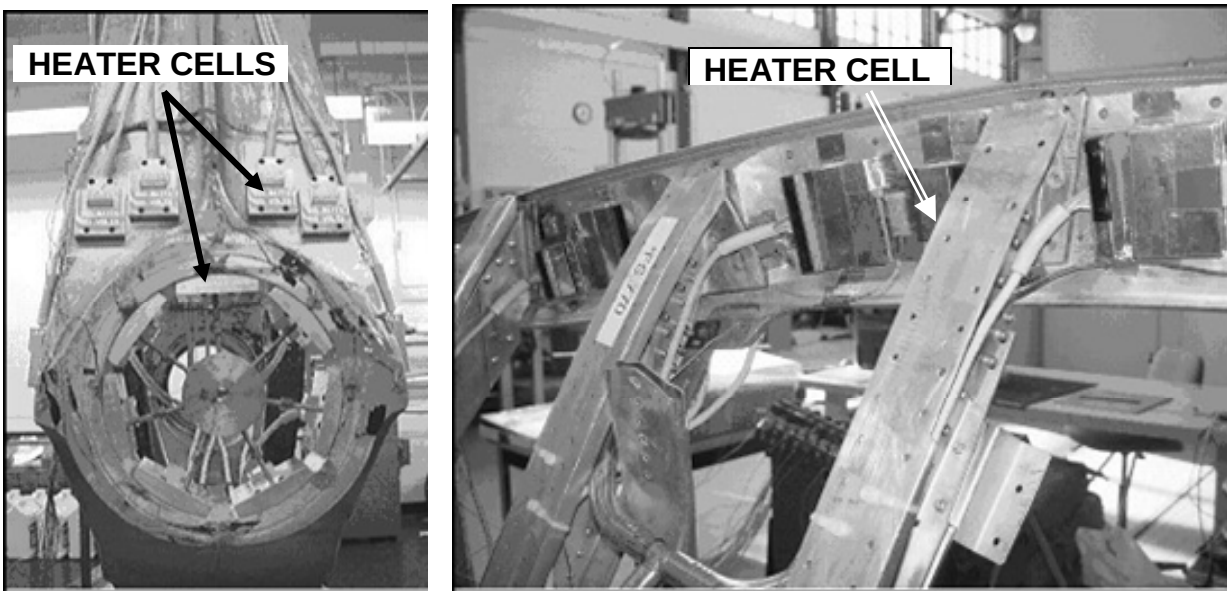


Figure 25-3: *In Situ* RRA Process on C-141 Landing Gear Main Frame and C-130 Sloping Longeron Using Zimac Heater Cells.

Detailed processing specifications for the two RRA processes were developed. For the *in situ* RRA process, the processing specification addresses the following areas:

- Closed form thermal analysis to determine heat transfer requirements.
- Preparation of drawings for heater cell, thermocouple and active cooling placement.
- Electrical conductivity for screening the material's initial condition.
- Retrogression treatment at 195°C using dissolution kinetics modeling for optimized retrogression time.
- Re-aging treatment at 120°C for 24 hours.
- Electrical conductivity and hardness surveys to determine the material's final condition.

25.5 CERTIFICATION REQUIREMENTS

No formal exercise to generate A and B basis allowables for the RRA process was undertaken. The USAF certification approach focused on strength reduction rather than on meeting MIL-HDBK-5H static strength allowables. What this means in practice is that the USAF was satisfied with no greater than a seven percent reduction in 'as-received' static strength. Typically the 'as-received' static strength for extruded product such as the C-130 sloping longeron is at least seven percent greater than the MIL-HDBK-5H A-Basis static strength allowable. Because each component will be screened using electrical conductivity before an RRA treatment is carried out, the process specification ensures that the component's final static strength will meet the MIL-HDBK-5H A-Basis static strength allowable. In addition, the USAF also have access to the Lockheed Martin design data for the C-130 and they have determined that for those candidate RRA parts the seven percent knockdown in static strength is within the static margin-of-safety for those specific components.

25.6 C-130 SLOPING LONGERON

The C-130 sloping longeron (Figure 25-4) is an Al 7075-T6511 extrusion 26 ft (7.9 m) in length with a maximum section thickness of 0.78 in. (19.6 mm). C-130 sloping longerons are replaced on a regular basis during depot level maintenance because of pitting corrosion and stress corrosion cracking damage. Corrosion damage is limited to two sections of the longeron between fuselage stations FS770 and FS835. For the sloping longeron 8 of the 26 feet were in-situ RRA treated starting at FS737 and extending to FS835. The RRA qualification trials were carried out as follows:

1) Service exposed longeron material was used for:

   a) Corrosion, static strength, fatigue and fracture coupon tests;

   b) Dissolution kinetics model optimization; and

   c) Heat transfer hardware and software development.

2) New extruded Al-7075-T6511 material was then used to ensure that the service exposed longeron material did not influence the coupon test results and the RRA process parameters. A processing specification was written for the in-situ RRA process as applied to the sloping longeron.

3) Two new sloping longerons were then purchased. The first sloping longeron was in-situ RRA treated following the processing specification. The longeron was then sectioned for tensile strength verification. The second sloping longeron was then in-situ RRA treated.

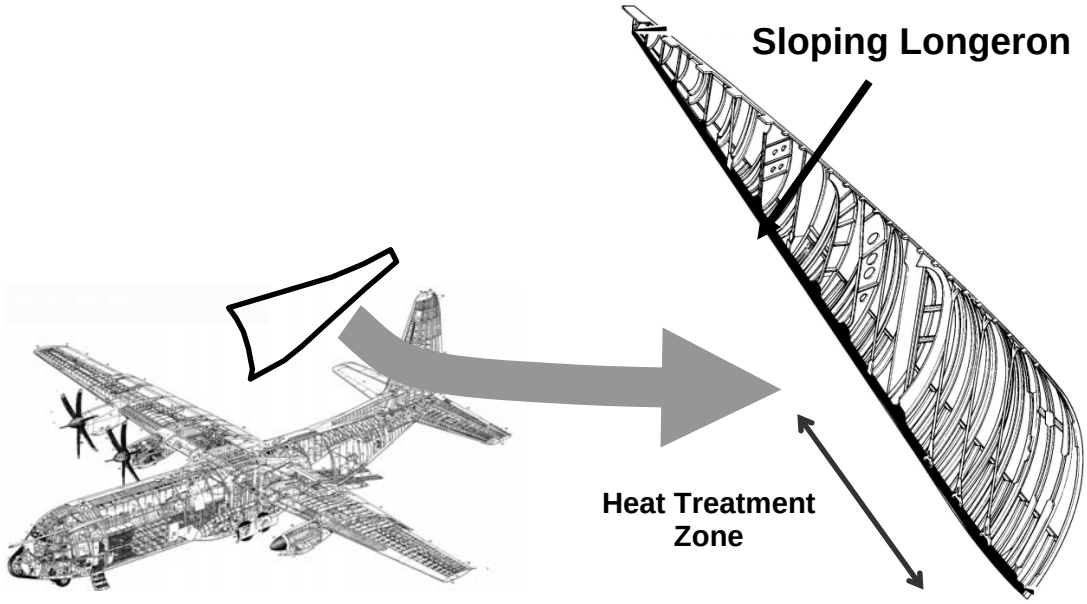
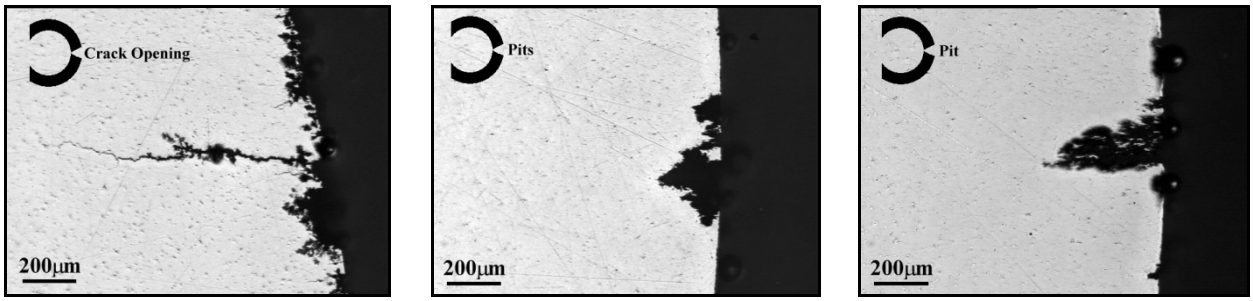


Figure 25-4: C-130 Showing Location of 26 ft Long Sloping Longeron and Heat Treatment Zone.

25.6.1 Sloping Longeron Corrosion Results

Exfoliation corrosion tests were conducted in accordance with ASTM Standard G 34. The RRA treated specimens improved the exfoliation corrosion resistance of as-received Al 7075-T6511 sloping longeron specimens from an EXCO rating of ED (severe separation of metal into layers) to a rating of P (discrete pits).

Stress corrosion cracking tests were conducted in accordance with ASTM Standards G 38 and G 44. The C-ring specimen was chosen because of the limited material thickness available in the S-T orientation. The specimens were pre-stressed to 45 and 54 ksi and placed in an alternate immersion tank where they were exposed to a 10 minute immersion in a 3.5% NaCl solution followed by 50 minutes of drying time. According to Federal Specification Sheet QQ-A-200/11E [21], the test duration is to be 20 days and specimens lasting until the end of 20 day period are considered to have SCC resistance equivalent to the T73 temper. The RRA treated specimens successfully completed the 20 day exposure period without developing evidence of intergranular cracks. Figure 25-5 shows polished x-sections of typical T6511, T73511 and T6RRA C-ring specimens.



T6 Specimen SK29 (16 days to failure)	T73 Specimen SL03 (20+ days)	T6RRA Specimen SK01 (20+ days)

Figure 25-5: C-Ring SCC Comparison with 54 ksi (372 MPa) Pre-Stress Level.

A more extensive series of stress corrosion tests were conducted with four inch thick new Al 7075-T6511 and Al7075-T73511 extrusions. For this work ASTM Standard G-139 was used. Figure 25-6 is a comparison of residual strength averages in Box-Cox metric shows comparable performance between the T73 temper and the T6RRA processed material.

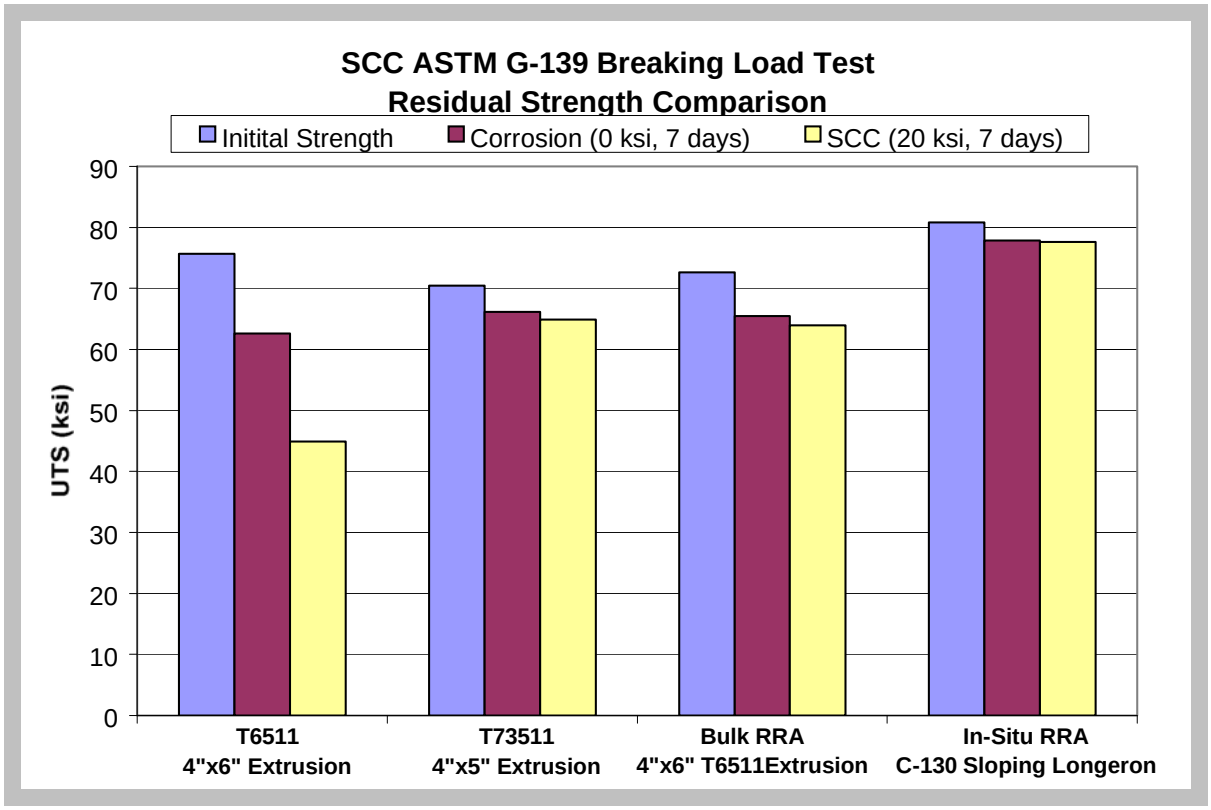


Figure 25-6: Breaking Load SCC Test Results Using Box-Cox Metric (ASTM G 139).

25.6.2 Sloping Longeron Tensile Test Results

Tensile testing was conducted in accordance with ASTM Standard B 557. Tensile specimens in the L direction were cut from as-received and RRA treated material. The average results for 84 RRA treated specimens (Table 25-1) show that the mean strength values exceeded the MIL-HDBK-5H A-Basis tensile strength allowable (the target value for minimum strength reduction). If a normal distribution for the tensile data is assumed, the mean value minus three standard deviations for the optimized RRA treatments also exceeds the MIL-HDBK-5H A-Basis allowable.

Table 25-1: C-130 Sloping Longeron Tensile Strength Results.

	Yield (ksi)	Ultimate (ksi)	Elongation (%)
As-received Al 7075-T6511	83.6 (576 MPa)	90.7 (625 MPa)	10.6
RRA Treated Material	77 (530 MPa)	84.5 (583 MPa)	9.6
MIL-HDBK-5H A-Basis	72 (496 MPa)	81 (558 MPa)	7

25.6.3 Sloping Longeron Electrical Conductivity Results

For Al 7075-T6, there is a correlation between electrical conductivity and corrosion resistance. If the electrical conductivity of RRA treated Al 7075-T6 material is increased beyond 38% IACS, the exfoliation corrosion rating will improve to EXCO A or greater and the SCC resistance will improve to that of the T73 temper. There is not an established correlation between electrical conductivity and static strength. However, for the C-130 material test program, electrical conductivity measurements were taken on each tensile specimen. The results for the L-direction tensile data are shown in Figure 25-7. The results indicate that electrical conductivity values between 38 and 40% IACS will consistently produce tensile strength results above the MIL-HDBK-5H A-Basis allowable. This finding is important because electrical conductivity measurements are non-intrusive, accurate and repeatable. For quality control purposes, electrical conductivity can be used in a highly reliable fashion to evaluate the as-received and the RRA condition (corrosion resistance and static strength) of a sloping longeron.

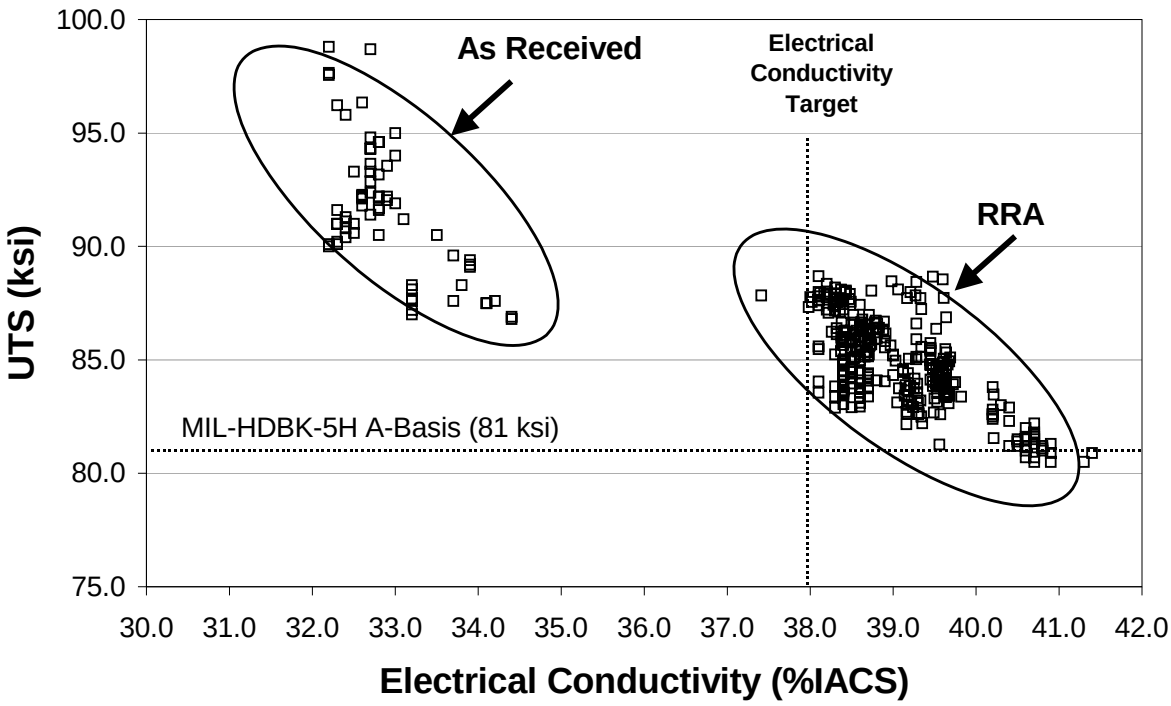


Figure 25-7: UTS vs. Conductivity Data for Sloping Longeron Material.

25.7 C-130 PARATROOPER DOOR LONGERON

The forged Al 7075-T6 Paratrooper Door Longerons (PDL) of the C-130 have been affected by pitting and exfoliation corrosion damage. The lower longitudinal attachment of the fitting on the outboard side is an area on the PDL that is particularly susceptible to exfoliation and pitting corrosion. The current off-the-aircraft repair method permits grinding away the corrosion to specific limits based on where the corrosion is found on the longeron. Once the grind-out limits have been reached, replacement of the longeron is mandated. For the PDL the RRA process was developed as a shop process using an oil bath. This is an easier heat treatment process to carry out than the in-situ RRA process developed for the C-130 sloping longeron and the C-141 landing gear mainframe. The retrogression phase is carried out for a specific period of time with no monitoring of part temperature or use of ‘real-time’ dissolution kinetics.

25.7.1 PDL Test Results

The results for stress corrosion cracking and static strength tests are presented in Table 25-2 and Table 25-3. The alternate immersion stress corrosion cracking testing was carried out using C-ring specimens in accordance with ASTM Standard G 38. All C-ring specimens were pre-stressed to 45 ksi (310 MPa). Tensile strength testing was carried out in accordance with ASTM Standard E 8.

Table 25-2: C-130 PDL Tensile Strength Results.

	Ultimate Strength (ksi)
Service Exposed Al 7075-T6	85 (586 MPa)
Service Exposed RRA	81 (558 MPa)
New Al 7075-T6	94.5 (650 MPa)
New Material RRA	87 to 93 (600 to 640 MPa)

Table 25-3: C-130 PDL Stress Corrosion Cracking Results (ASTM G 38).

	Electrical Conductivity (%IACS)	Duration (days)
Service Exposed Al 7075-T6	32.9	5 to 9
New Material RRA	38.5	20+

The corrosion and tensile results for the forged Al 7075 PDL material are similar to those presented for the C-130 sloping longeron extrusions. The RRA process can therefore be applied to either extruded or forged Al 7075 T6 aircraft components.

25.8 CONCLUSIONS

- Retrogression and re-aging of Al 7075 in the T6 temper significantly improves the material's resistance to corrosion without compromising static strength properties.
- RRA tensile data in the L-direction indicated that tensile strength, yield strength and percent elongation were consistent with the A-Basis allowables from MIL-HDBK-5H.
- The exfoliation test results indicate that the RRA process raises the resistance to exfoliation corrosion from AD (severe exfoliation) to EA (superficial exfoliation) / P (pitting).
- Stress corrosion tests showed no evidence of intergranular cracking after 20 days of alternate immersion exposure. This corrosion resistance is equivalent to the T73 temper.
- The retrogression phase has been optimized using computer controlled 'real-time' dissolution kinetics to ensure that for an RRA treated component:
  1) The electrical conductivity is raised to at least 38% IACS; and
  2) The static strength properties remain above MIL-HDBK-5H A-Basis allowables.

- The RRA process can be carried out using two heat transfer mediums. The first is an oil bath, ideally suited for small components such as the C-130 paratrooper door longeron. The second is an in-situ conduction process that is applied to selected areas of larger components such as the C-130 sloping longeron and the C-141 landing gear mainframe.

- For C-130 Al 7075-T6511 extrusions no greater than 0.75 inches thick (20 mm), a consistent trend between electrical conductivity and static strength was established. This correlation will be useful as a tool to assess the as-received static strength and the post RRA static strength.

25.9 ACKNOWLEDGEMENTS

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25.10 REFERENCES

[1] Cina, B. and Gan, R., “Reducing the Susceptibility of Alloys, Particularly Aluminum Alloys, to Stress Corrosion Cracking”, United States Patent 3856584, 24 December 1974.

[2] Cina, B. and Zeidess, F., “Advances in the Heat Treatment of 7000 Type Aluminum Alloys by Retrogression and Re-Aging”, Materials Science Forum, Vol. 102-104, 1992.

[3] Wallace, W., Beddoes, J.C. and de Malherbe, M.C., “A New Approach to the Problem of Stress Corrosion Cracking in 7075-T6 Aluminum”, Canadian Aeronautics and Space Journal, Vol. 27, No. 3, 1981.

[4] Rajan, K., Wallace, W. and Beddoes, J.C., “Microstructural Study of a High Strength Stress Corrosion Resistant 7075 Aluminum Alloy”, Journal of Material Science and Technology, Vol. 17, 1982.

[5] Wallace, W. and Beddoes, J.C., “Microstructural Study of a High Strength Stress Corrosion Resistant 7075 Aluminum Alloy,” Journal of Material Science, Vol. 17, 1982.

[6] Danh, N.C., Rajan, K. and Wallace, W., “A TEM Study of Microstructural Changes During Retrogression and Re-Aging in 7075 Aluminum”, Metallurgical Transactions, Vol. 14A, 1983.

[7] Islam, M.U. and Wallace, W., “Retrogression and Re-Aging Response of 7475 Aluminum Alloy”, Metals Technology, Vol. 10, 1983.

[8] Holt, R.T., Parameswaran, V.R. and Wallace, W., “RRA Treatment of 7075-T6 Aluminum Components”, Canadian Aeronautics and Space Journal, Vol. 42, No. 2, 1996.

[9] Wallace, W., Holt, R.T., Butler, C. and DuQuesnay, D.L., “Retrogression and Re-Aging Revisited”, Key Engineering Materials, Vols. 145-149, Part 2, 1998.

[10] Holt, R.T., Raizenne, M.D. and Wallace, W., “RRA Heat Treatment of Large Al 7075-T6 Components”, NATO/RTA AVT, Corfu, Greece, 1999.

[11] Wu, X.J., Raizenne, D.M., Poon, C., Chen, W. and Wallace, W., “Thirty Years of Retrogression and Re-Aging (RRA)”, Presented at 23rd Congress of International Council of Aeronautical Science, Toronto, Ontario, Canada, 2002.

[12] Raizenne, D.M., Wu, X.J., Whitehouse, M. and Poon, C., "In-Situ Retrogression and Re-Aging of a C-130 Al 7075-T6511 Sloping Longeron", USAF Aircraft Structural Integrity Program Conference, San Antonio, TX, USA, December 2000.

[13] Raizenne, D.M., Sjöblom, P., Snide, J., Peeler, D.T. and Hunter, C., "In-Situ Retrogression and Re-Aging of C-130 Al 7075 T6511 Sloping Longerons", Presented at AEROMAT 2001 Conference, Long Beach, CA, USA, June 2001.

[14] Raizenne, D.M., Wu, X. and Poon, C., "In-Situ Retrogression and Re-Aging of Al 7075-T6 Parts", 5th FAA/DoD/NASA Conference on Aging Aircraft, Orlando, FL, USA, September 2001.

[15] Raizenne, D., Sjöblom, P., Rondeau, R. and Peeler, D.T., "Transitioning of the Retrogression and Re-Aging (RRA) Process to USAF Hardware", USAF Aircraft Structural Integrity Program Conference, Williamsburg, VA, USA, December 2001.

[16] Raizenne, D.M., Sjöblom, P., Rondeau, R., Snide, J. and Peeler, D.T., "Retrogression and Re-Aging of New and Old Aircraft Parts", 6th FAA/DoD/NASA Conference on Aging Aircraft, San Francisco, CA, USA, September 2002.

[17] Rondeau, R., "Retrogression and Re-Aging (RRA) of 7075-T6 Corrosion Prone Parts", USAF Report ASC-TR-2005-0001, January 2005.

[18] Peeler, D.T., Raizenne, D., Sjöblom, P., Rondeau, R., Kuhlman, S. and Snide, J., "Retrogression and Re-Aging (RRA) of Aluminum 7075-T6 Aircraft Components", Tri-Service Corrosion Conference, Las Vegas, NV, USA, November 2003.

[19] Wu, X.J., Kou, A.K. and Zhao, L., "A New Approach to Heat Damage Evaluation for 7XXX Aluminum Alloy", Canadian Aeronautics and Space Journal, Vol. 42, 1996.

[20] Hunt, W.H., Stiffler, R.C. and Green, J., "The Use of Electrical Conductivity for Heat Treatable Aluminum Alloys", 18th ASM Heat Treating Society Conference, Rosemont, IL, USA, 1998.

[21] Federal Specification Sheet QQ-A-200/11E, November 1993.