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Cryogenic Fracture Toughness Improvement for the Super Lightweight Tank's Main Structural Alloy

P.S. Chen
IIT Research Institute, Huntsville, Alabama

W.P. Stanton
Marshall Space Flight Center, Marshall Space Flight Center, Alabama

February 2002

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Space Administration

Marshall Space Flight Center • MSFC, Alabama 35812

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LIST OF ABBREVIATIONS, ACRONYMS, AND SYMBOLS

%El	percent elongation (ductility)
$\sqrt{in}$	square root inch
θ'	precursor-phase of Al_2Cu precipitate compound
θ''	stable-phase of Al_2Cu precipitate compound
Ag	silver
Al	aluminum
ASTM	American Society for Testing Materials
CFT	cryogenic fracture toughness
Cu	copper
HR_B	Rockwell hardness B scale
ksi	thousand pounds per square inch
K	stress intensity factor
K_{Ic}	plane-strain fracture toughness
L orientation	longitudinal direction
Li	lithium
LN_2	liquid nitrogen
LT orientation	long transverse direction
Mg	magnesium
MSRC	multistep heating rate-controlled aging treatment
NaCl	sodium chloride

LIST OF ABBREVIATIONS, ACRONYMS, AND SYMBOLS (Continued)

SCC	stress corrosion cracking
SLWT	super lightweight tank
SS	simulated service testing
ST orientation	short transverse direction
T_1	strengthening precipitate
TEM	transmission electron microscopy
T-*L* orientation	notch parallel to the rolling direction
T-*S* orientation	notch perpendicular to the rolling direction
TS	two-step
UTS	ultimate tensile strength
YS	yield strength
Zr	zirconium

UNUSUAL TERMS

Simulated service testing	A test method developed by NASA and Lockheed Martin to simulate SLWT launch conditions

TECHNICAL MEMORANDUM

CRYOGENIC FRACTURE TOUGHNESS IMPROVEMENT FOR THE SUPER LIGHTWEIGHT TANK'S MAIN STRUCTURAL ALLOY

1. INTRODUCTION

NASA has selected Al-Li alloy 2195 as the main structural alloy for the super lightweight tank (SLWT) of the Space Shuttle. Cryogenic strength and toughness are critical to this application, since the SLWT houses liquid oxygen and hydrogen. To ensure proper quality control, NASA has imposed lot acceptance testing on alloy 2195 plate before it can be used in the SLWT program. Some commercial 2195 plates were rejected for the SLWT program, mostly due to low cryogenic fracture toughness (CFT) that was found to be related to the density, size, and location of a precipitate labeled T_1[1,2] in this alloy. CFT decreases considerably as T_1 increases in density at the subgrain boundaries. Therefore, attempts to improve fracture toughness were directed toward reducing the density of T_1 at subgrain boundaries and enhancing the nucleation of T_1 in the matrix.

A new two-step (TS) artificial aging treatment has been developed that can greatly improve CFT by controlling the location and size of T_1. Such aging improves fracture toughness, without sacrificing tensile and yield strength (YS). This Technical Memorandum details improvements in CFT and resistance to stress corrosion cracking (SCC) that resulted from the use of TS aging.

2. TECHNICAL APPROACH

In Al-Cu-Li alloys, fracture toughness ratio correlates well with the size and density of T_1 in the subgrain boundaries[2] (fig. 1). High CFT can be achieved by suppressing T_1 precipitation at subgrain boundaries and enhancing T_1 nucleation in the matrix, thus eliminating premature fractures along precipitate-rich subgrain boundaries.

Based on this finding, a series of step-aging treatments was conducted in order to promote T_1 nucleation and growth in the matrix rather than at subgrain boundaries (table 1). This approach began with an initial holding at low temperature (with high undercooling) to enhance formation of T_1 nuclei in the matrix. Then the furnace temperature was raised to permit each precipitate nucleus to grow and become stable. These nuclei continued to grow during aging, with negligible dissolution into solid solution. Long-term aging at low temperatures also allowed T_1 to grow in the matrix before they could nucleate and grow at the subgrain boundaries. The most promising two-step aging treatment was selected for evaluation of the resulting tensile and cryogenic properties.

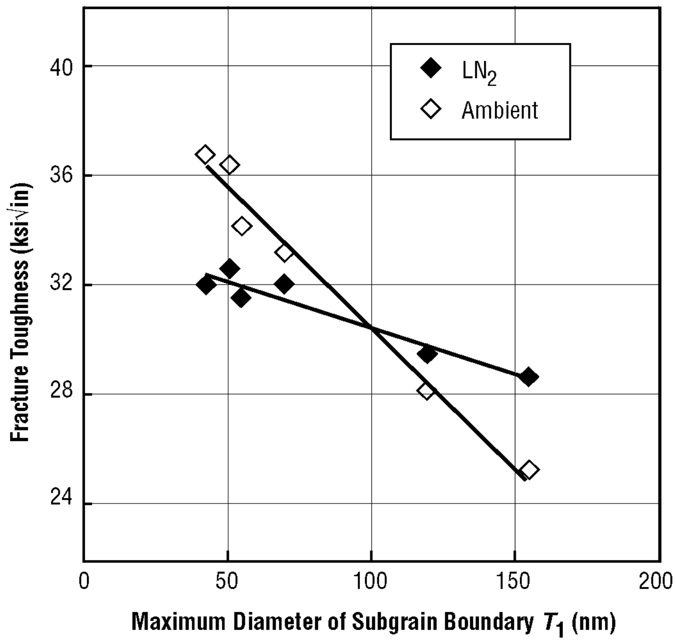


Figure 1. Fracture toughness versus maximum size of T_1 at subgrain boundaries with fracture toughness decreasing as T_1 size increases.[1,2]

Table 1. Aging treatment matrix.

Aging Treatment	129 °C (270 °F) (hr)	132 °C (270 °F) (hr)	135 °C (275 °F) (hr)	138 °C (280 °F) (hr)	141 °C (285 °F) (hr)	143 °C (290 °F) (hr)
1		20		20		15
2		20		20		10
3		20		20		5
4		20		15		10
5		15		20		10
6		15		15		10
7	20		20		20	
8		20		40		
9			20		40	
10		20		42		
Conventional						32

3. EXPERIMENTAL PROCEDURES

Alloy 2195 (nominal composition: Al-4.0Cu-1.0Li-0.52Mg-0.42Ag-0.12Zr) was received in the form of 1.7-in thick rolled plates, which had been solutionized and stretched for 3 percent at ambient temperature. The experiments began with a series of step-aging treatments (table 1). Based on hardness test results and microstructural characterization, the most promising step-aging treatments were selected for evaluation of their resultant tensile properties, CFT, and SCC resistance. The following procedures were performed:

- Tensile tests were carried out at ambient temperature, using flat tensile specimens to evaluate the effects of microstructural variation. Uniaxial tensile properties were evaluated in the longitudinal (*L*), longitudinal transverse (*LT*), and short transverse (*ST*) orientations, with at least two tests performed in each orientation. Fracture toughness tests were performed at ambient temperature and –196 °C (–320 °F). The plates were evaluated in the *T-L* orientation (notch parallel to the rolling direction) per American Society for Testing Materials (ASTM) E740. The specimens were fatigue precracked at 20 Hz, then tensile tested to failure at a crosshead speed of 0.13 cm/min. Precrack length and maximum load to failure were factored into the standard equation. Simulated service tests were performed at –196 °C (–320 °F). The plates were evaluated in the *T-S* orientation (notch perpendicular to the rolling direction) per ASTM E740. The specimens were fatigue precracked at 20 Hz, then tensile tested at a crosshead speed of 0.05 in/min to failure. Precrack length and maximum load to failure were factored into the standard equation. Microstructural characterization was performed using a JEOL, Ltd. 2000F transmission electron microscope operated at 200 kV. Samples were jet polished in an electrolyte (70-percent methanol and 30-percent nitric acid) at –20 °C (–4 °F) with an applied potential of 12 V.

- Stress corrosion evaluation was performed on four lots of Al-Li 2195 (960M030F, 30J, 30K, and 30L) to determine whether these materials would have met minimum requirements if they had been step-aged. Two sets of specimens were tested for each lot. One set was stressed to 45 ksi (≈60 percent of the 0.2-percent offset YS). The other was stressed to ≈56 ksi (75 percent of the 0.2-percent offset YS), which was well above the lot acceptance requirement for stress corrosion. The test environment was 3.5-percent sodium chloride (NaCl) alternate immersion per ASTM G44. Some specimens were exposed unstressed and then removed from testing after 60 and 90 days, so that they could be tensile tested to failure to determine how much degradation had taken place (based on reduction in load-carrying ability). All specimens passed the minimum requirement of 10 days in 3.5-percent NaCl alternate immersion at a stress of 45 ksi, per Lockheed Martin Specification STM 11A1–4.

4. RESULTS AND DISCUSSION

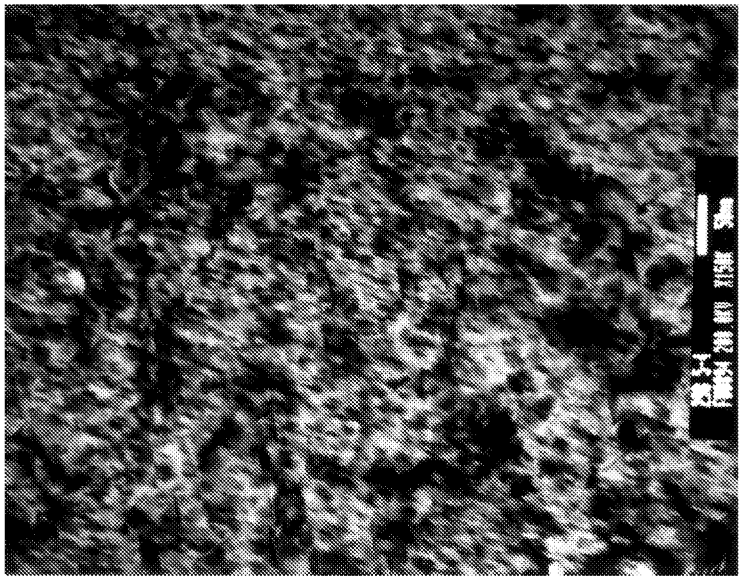
4.1 Hardness and Microstructure

TS aging caused hardness variations, as shown in table 2. The alloy was underaged at 132 °C (270 °F) to enhance precipitate nucleation in the matrix. Then it was heated for various times at higher temperatures in an effort to obtain peak or near-peak aged conditions, while preventing preferential nucleation and growth of T_1 at subgrain boundaries. For alloy 2195, a Rockwell hardness B scale (HR_B) of 90 is roughly equivalent to 73-ksi YS in the *L* and *LT* orientation, the minimum strength requirement for the SLWT program. A three-step aging treatment (No. 1) reached a hardness of HR_B 91.7 after 55 hr, while a two-step aging treatment (No. 8) reached a hardness of more than HR_B 90 after 60 hr. These results proved that the use of proper parameters would permit the aging treatment to be reduced to only two steps.

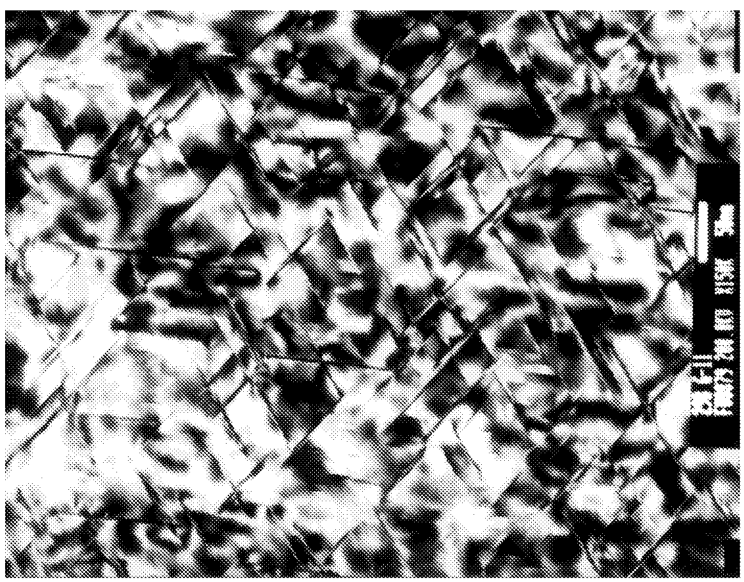
Table 2. Hardness values as a function of time and temperature for two- and three-step aging treatments (lot 950M029B).

Aging Treatment	129 °C (265 °F) (hr)	HR_B	132 °C (270 °F) (hr)	HR_B	135 °C (275 °F) (hr)	HR_B	138 °C (280 °F) (hr)	HR_B	141 °C (285 °F) (hr)	HR_B	143 °C (290 °F) (hr)	HR_B
1			20	76.2			20	86.2			15	91.7
2			20				20				10	88.9
3			20				20				5	87.1
4			20				15	84.1			10	88.0
5			15				20	84.4			10	88.5
6			15				15				10	87.6
7	20	75.8			20	83.2			20	89.2		
8			20	76.2			40	90.7				
9					20	79.1			40	89.8		
10			20	76.2			42	91.2				

Step-aged microstructures were examined using transmission electron microscopy (TEM). After aging at 132 °C (270 °F)/20 hr, the matrix consisted of numerous fine precursor-phase Al_2Cu precipitate compounds (θ'') with scattered T_1. The large number of very fine θ'' precipitates indicated that an early stage of nucleation took place at 132 °C (270 °F), as shown in figure 2(a). Hardness increased rapidly at 138 °C (280 °F)/40 hr, indicating near-peak precipitation of the strengthening phases. T_1 grew considerably to become the majority phase in the matrix. It was also present at subgrain boundaries in sizes no coarser than those found in the matrix, as shown in figure 2(b). Similar microstructural evolution was observed for the other aging treatments.



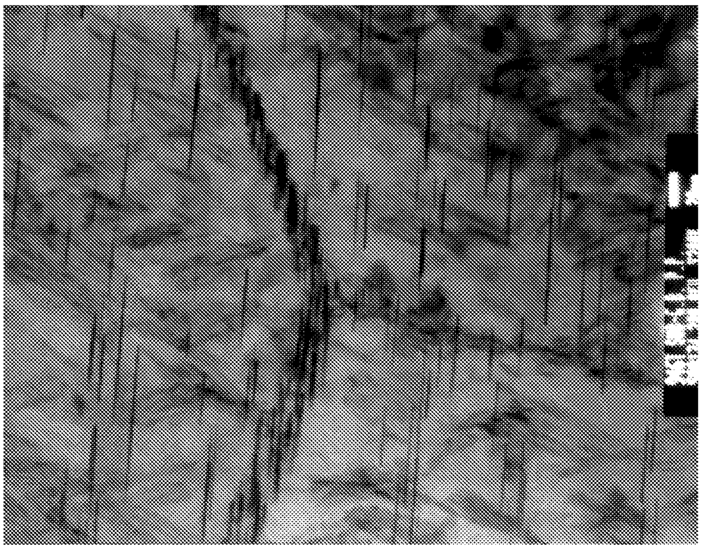
(a) After aging at 132 °C (270 °F)/20 hr (aging treatments Nos. 1–4 and 8).



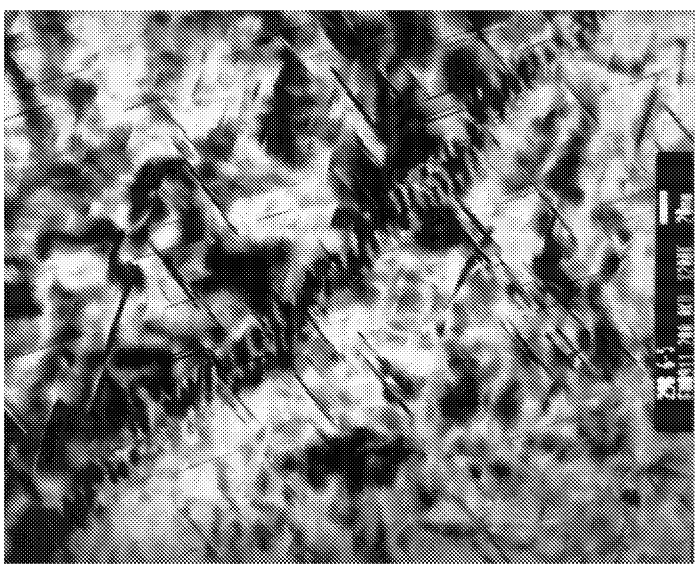
(b) After aging at 132 °C (270 °F)/20 hr + 138 °C (280 °F)/40 hr (aging treatment No. 8).

Figure 2. Precipitate morphology at early stage of nucleation and growth.

Substantial microstructural differences were found between conventionally aged alloy (with coarser T_1 that was much denser in subgrain boundaries than in the matrix[1,2]) and step-aged alloy (where T_1 occasionally existed at subgrain boundaries, but not as densely as in the matrix), as seen in figure 3. Size and density also differed for precipitates in the matrix, where conventionally aged alloy contained more T_1 than stable-phase Al_2Cu precipitate compounds (θ') and θ'' while TS-aged material contained more θ' and θ'' than T_1. TS aging achieved the same strength levels as conventional aging by precipitating more θ' and θ'' in the matrix while preventing preferential T_1 precipitation at subgrain boundaries.



(a) After aging at 143 °C (290 °F)/32 hr (conventional aging).



(b) After aging at 132 °C (270 °F)/20 hr + 138 °C (280 °F)/40 hr (aging treatment No. 8).

Figure 3. TEM micrographs comparing subgrain boundary microstructures.

4.2 Mechanical Properties

TS aging treatment No. 8 was selected for tensile strength and fracture toughness evaluation. Results indicated that TS aging can achieve ductility and YS levels that are acceptable for the SLWT program, as seen in table 3.

Table 3. Tensile properties (aging treatment Nos. 8 and 10).

Aging Treatment	Lot	Orientation	YS (ksi)	UTS (ksi)	%El
8	950M029B	*L*	79.4	84.5	9.5
8	950M029B	*LT*	76.8	84.6	9.9
8	950M029B	*ST*	69.6	82.2	4.5
10	950M020F	*L*	79.1	85.7	10.6
10	950M020F	*LT*	71.8	81.2	10.1
10	950M020F	*ST*	67.2	80.5	7.6
10	960M030F	*L*	79.8	87.4	8.9
10	960M030F	*LT*	72.0	83.8	9.2
10	960M030F	45°	71.6	83.5	10.6
10	960M030F	*ST*	75.0	87.9	4.0
10	960M030J	*L*	82.1	88.2	9.6
10	960M030J	*LT*	74.1	84.6	9.0
10	960M030J	45°	72.3	82.6	11.0
10	960M030J	*ST*	74.9	87.5	4.0
10	960M030K	*L*	77.1	85.7	9.9
10	960M030K	*LT*	70.3	81.5	7.4
10	960M030K	45°	69.9	81.0	10.3
10	960M030K	*ST*	73.3	85.2	2.1
10	960M030L	*L*	83.4	92.0	8.8
10	960M030L	*LT*	74.9	84.6	9.4
10	960M030L	45°	71.9	83.3	10.4
10	960M030L	*ST*	75.2	88.1	4.8

Note: The SLWT engineering material specification imposes a YS requirement of 73 ksi only in the *L* and *LT* orientations. Here, all values presented in the *L* and *LT* orientations are considered representative of normal production materials.

The most noticeable change was that CFT (which must be at least 30 ksi√in for the SLWT program) significantly improved in all five lots of material subjected to TS aging, as shown in table 4 and figure 3. TS aging improved the CFT of conventionally aged lot 950M029B (a bad lot) by more than 30 percent (from as low as 25.4 ksi√in to 34 ksi√in). TS aging even improved the CFT of conventionally aged lot 950M020F (a good lot, with data provided here as a baseline) by ≈10 percent, as shown in figure 4. (Note: CFT data have not been made available for conventionally aged lots 960M030F, 960M030J, 960M030K, and 960M030L.)

Table 4. Tensile and mechanical properties (conventional aging and aging treatment Nos. 8 and 10).

Lot (3% stretch)	Aging Treatment	*L* Orientation			*T-L* Orientation	
		YS (ksi)	UTS (ksi)	%El	K at *a*/2 (LN_2)	K at *a*/2 (ambient)
950M029B	TS aging No. 8	79.4	84.5	9.5	33.74	30.44
950M029B	Conventional	74.0	83.1	7.0	25.40	30.04
950M020F	TS aging No. 10	79.1	85.7	10.6	37.64	34.79
950M020F	Conventional	76.1	83.4	8.0	34.91	32.90
960M030F	TS aging No. 10	79.8	87.4	8.9	30.18	29.34
960M030F	Conventional	78.9	85.0	6.5	–	27.50
960M030J	TS aging No. 10	82.1	88.2	9.6	30.10	28.66
960M030J	Conventional	77.4	84.5	6.5	–	28.25
960M030K	TS aging No. 10	77.1	85.7	9.9	29.49	29.41
960M030K	Conventional	74.6	82.4	7.8	–	27.30
960M030L	TS aging No. 10	83.4	92.0	8.8	31.53	29.66
960M030L	Conventional	77.9	84.3	8.3	–	27.00

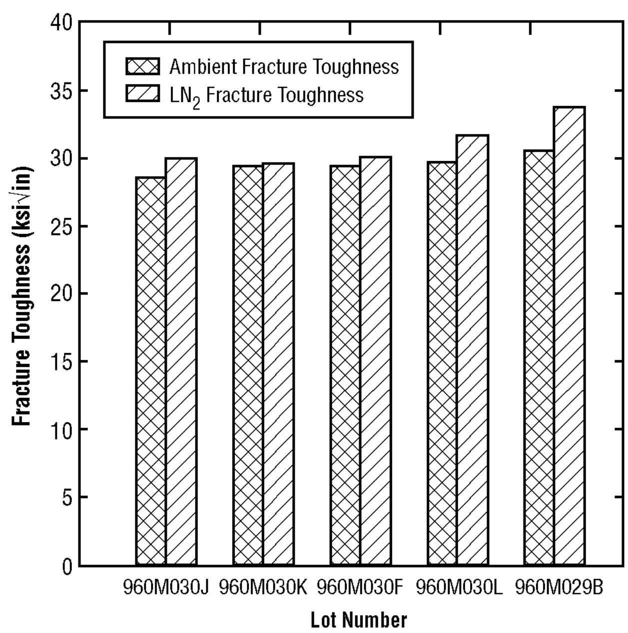


Figure 4. Fracture toughness data (aging treatment Nos. 8 and 10).

Simulated service testing was conducted on five lots of material that had failed to pass such tests after conventional aging. After TS aging, all five lots passed by a very comfortable margin (see table 5). TS-aged material also exhibited much higher fracture toughness at cryogenic temperature than at ambient temperature.

Table 5. Simulated service results (conventional aging and aging treatment No. 10).

Lot	Aging Treatment	Test Type, Temperature	Induced Flaw (Before Testing) Height a (in)	Induced Flaw (Before Testing) Width 2C (in)	Offset (in)	Net Section Fracture Stress (ksi)	K at a/2 (ksi√in)
950M029B	MSRC aging[1]	Proof, ambient	–	–	74.10	76.25	30.57
950M029B	MSRC aging[1]	SS, –323 °F	–	–	–	85.07	34.11
960M030F	Conventional[2]	Proof, ambient	0.072	0.173	0.0010	71.50	27.50
960M030F	TS aging No. 10	Proof, ambient	0.072	0.175	0.0027	75.56	29.34
960M030F	TS aging No. 10	SS, –323 °F	0.073	0.179	–	90.10	33.84
960M030J	Conventional	Proof, ambient	0.074	0.174	0.0013	73.70	28.70
960M030J	Conventional[2]	Proof, ambient	0.080	0.179	0.0005	69.20	27.80
960M030J	TS aging No. 10	Proof, ambient	0.072	0.178	0.0024	74.56	29.07
960M030J	TS aging No. 10	Proof, ambient	0.072	0.182	–	72.77	28.66
960M030J	TS aging No. 10	SS, –323 °F	0.071	0.184	–	76.47	30.11
960M030K	Conventional	Proof, ambient	0.076	0.179	0.0009	71.90	28.40
960M030K	Conventional[3]	Proof, ambient	0.071	0.174	0.0004	68.40	26.20
960M030K	TS aging No. 10	Proof, ambient	0.072	0.176	0.0025	75.47	29.41
960M030K	TS aging No. 10	SS, –323 °F	0.071	0.175	–	84.47	32.72
960M030L	Conventional[2]	Proof, ambient	0.072	0.176	0.0009	69.80	27.00
960M030L	TS aging No. 10	Proof, ambient	0.068	0.175	0.0021	77.41	29.66
960M030L	TS aging No. 10	SS, –323 °F	0.072	0.178	–	87.91	34.43

[1]MSRC aging consists of a 3% stretch with solution heat treatment + 127 °C (260 °F)/5 hr
+ temperature ramp to 135 °C (275 °F) at a rate of 0.56 °C (1 °F)/hr + 135 °C (275 °F)/5 hr
+ temperature ramp to 143 °C (290 °F) at a rate of 0.56 °C (1 °F)/hr + 143 °C (290 °F)/25 hr.

[2]Tests failed because specification requires net section stress to be ≥71.9 ksi.

[3]Test failed because specification requires critical offsets to be ≥0.0007 in/in.

Table 6 shows the results of SCC testing (3.5-percent NaCl alternate immersion) for TS-aged specimens. At a stress level of 45 ksi (≈60-percent YS), all specimens passed the minimum 10-day requirement, with an average SCC life of 70 days. At a higher stress level of ≈55 ksi (75-percent YS), all specimens passed the minimum 10-day requirement, with an average SCC life of 52 days.

The matrix of a TS-aged specimen has a significantly different microstructure than that of a conventionally aged specimen, with the former producing much higher CFT and nearly the same YS. Similar YS levels observed for TS-aged and conventionally aged materials can be qualitatively correlated to microstructural characteristics; e.g., type, size, distribution, and density of strengthening phases T_1 and θ''. Initial holding at low temperature with high undercooling increases the number of precipitate embryos. Subsequent aging at 138 °C (280 °F) enables precipitate particles to coarsen slowly without dissolving, thereby increasing the total number of precipitates. Additional strengthening is provided by the much higher number of θ′ and θ″ precipitates present in TS-aged materials, making the YS comparable to conventionally aged materials. As aging continues, T_1 will eventually nucleate at subgrain boundaries and start to grow. However, this treatment allows matrix T_1 to precipitate and grow before subgrain boundary T_1 does. In addition, early coarsening of matrix T_1 greatly reduces the concentration of matrix Cu and Li, hindering the growth of subgrain boundary T_1 in a diluted Al-Cu-Li solid solution. Therefore, it is believed that reduced T_1 precipitation at subgrain boundaries leads to much improved CFT and SCC resistance in TS-aged material.

Table 6. Stress corrosion results (aging treatment No. 10).

Lot	Stress Level		Failure Ratio	Days to Failure
	YS (%)	ksi		
960M030F	60.0	45.0	3/3	60, 75, 77
960M030F	75.0	56.2	2/2	40, 44
960M030J	60.4	45.0	3/3	75, 75, 83
960M030J	75.0	55.9	2/2	47, 56
960M030K	61.4	45.0	2/3	63, 77, 90
960M030K	75.0	55.0	2/2	23, 62
960M030L	59.9	45.0	3/3	57, 60, 75
960M030L	75.0	56.3	2/2	56, 90

5. CONCLUSIONS

Marshall Space Flight Center has developed a new TS aging treatment that consists of exposures at 132 °C (270 °F)/20 hr + 138 °C (280 °F)/42 hr. TS aging can achieve the same YS levels as those produced by conventional aging, while providing much improved CFT, SCC resistance, and %El ductility in the *ST* direction. After TS aging, several rejected lots of alloy 2195 (which had previously failed to pass simulated service testing) were found acceptable for use in the SLWT program.

These material properties were improved by constraining T_1 nucleation and growth at subgrain boundaries, which permitted more uniform distribution of T_1 throughout the alloy.

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13. ABSTRACT *(Maximum 200 words)*

Marshall Space Flight Center has developed a two-step (TS) artificial aging technique that can significantly enhance cryogenic fracture toughness and resistance to stress corrosion cracking (SCC) in aluminum-copper-lithium alloy 2195. The new TS aging treatment consists of exposures at 132 °C (270 °F)/20 hr + 138 °C (280 °F)/42 hr, which can be readily applied to flight hardware production. TS aging achieves the same yield strength levels as conventional aging, while providing much improved ductility in the short transverse direction. After TS aging, five previously rejected lots of alloy 2195 (lots 950M029B, 960M030F, 960M030J, 960M030K, and 960M030L) passed simulated service testing for use in the super lightweight tank program. Each lot exhibited higher fracture toughness at cryogenic temperature than at ambient temperature. Their SCC resistance was also enhanced. All SCC specimens passed the minimum 10-day requirement in 3.5-percent sodium chloride alternate immersion at a stress of 45 ksi. The SCC lives ranged from 57 to 83 days, with an average of 70 days.

14. SUBJECT TERMS
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George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama
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