NASA/TM—2016–218230

Stress Corrosion Evaluation of Nitinol 60 for the International Space Station Water Recycling System

P.D. Torres
Marshall Space Flight Center, Huntsville, Alabama

November 2016

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Acknowledgments

The author wishes to acknowledge the following supporters:

Marshall Space Flight Center

AS10 (Environmental Engineering & Occupational Health Office): J. Troy, S. Adkins, P. Buckelew, and W. Carter
AS42 (Transportation & Logistics Engineering Office): A. Richardson
EE04 (Flight Programs & Partnerships Chief Engineers Office): R. Mize and B. Mitchell
EM01 (Materials and Processes Laboratory): S. Singhal and M. Prince
EM02 (Resource Management Team): D. Mahieux, J. Smith, T. Bili, and C. Cunningham
EM10 (Materials Test Branch): A. Girgis, L. Sharff, W. Tilson, and C. Harris
EM30 (Metals Engineering Division): T. Vaughn and F. Lowery
EM31 (Failure Analysis and Metallurgy Branch): B. Panda, G. Jerman, J. Hastings, and J. Honeycutt
EM32 (Metal Joining & Processes Branch): C. Russell, R. Hickman, M. Thames, and R. Lee (ESSSA)
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LIST OF ABBREVIATIONS, ACRONYMS, AND SYMBOLS

√	surface finish (arithmetic average roughness value)
AI	alternate immersion
Al	aluminum
ASTM	American Society for Testing and Materials
CP	commercially pure
ECLSS	Environmental Control and Life Support System
ELI	extra-low interstitial
HCl	hydrochloric acid
HNO_3	nitric acid
ksi	kilopounds per square inch
NaCl	sodium chloride
Ni	nickel
Nitinol	Nickel Titanium Naval Ordinance Laboratory
PABB	phosphoric acid-based brine
PABP	phosphoric acid-based pretreat
pretreat	pretreatment
SABB	sulfuric acid-based brine
SABP	sulfuric acid-based pretreat
SCC	stress corrosion cracking
Ti	titanium

LIST OF ABBREVIATIONS, ACRONYMS, AND SYMBOLS (Continued)

UNC	Unified National Course
UTS	ultimate tensile strength
UTS_f	final ultimate tensile strength (after exposure)
UTS_i	initial ultimate tensile strength (average for nonexposed specimens)

TECHNICAL MEMORANDUM

STRESS CORROSION EVALUATION OF NITINOL 60 FOR THE INTERNATIONAL SPACE STATION WATER RECYCLING SYSTEM

1. INTRODUCTION

A stress corrosion cracking (SCC) evaluation of Nitinol 60 was performed because this alloy is considered a candidate bearing material for the Environmental Control and Life Support System (ECLSS), specifically in the Urine Processing Assembly of the International Space Station. An SCC evaluation that preceded this one during the 2013–2014 timeframe included various alloys: Inconel 625, Hastelloy® C-276, titanium (Ti) commercially pure (CP), Ti 6Al-4V, extra-low interstitial (ELI) Ti 6Al-4V, and Cronidur 30. In that evaluation, most specimens were exposed for a year. The results of that evaluation were published in NASA/TM—2015–218206, entitled "Stress Corrosion Evaluation of Various Metallic Materials for the International Space Station Water Recycling System,"[1] available at the NASA Scientific and Technical Information program web page: http://www.sti.nasa.gov. Nitinol 60 was added to the test program in 2014.

2. DEFINITION

2.1 Stress Corrosion

Stress corrosion may be defined as the combined action of sustained tensile stress and corrosion to cause premature failure of a susceptible material. Certain metallic materials are more susceptible than others. If a susceptible material is placed in service in a corrosive environment under tension of sufficient magnitude, and the duration of service is sufficient to permit the initiation and growth of cracks, failures can occur at a stress lower than the material would normally be expected to withstand.

3. EXPERIMENTAL PROCEDURE

3.1 Test Matrix

A stress corrosion test matrix showing the number of specimens and how they were allocated to various types of tests is presented in table 1. As seen in table 1, different types of ECLSS solutions were used in this evaluation. The 3.5% NaCl alternate immersion tests per ASTM G44[2] are for comparison. Five specimens were used for tensile tests.

Table 1. Stress corrosion test matrix for Nitinol 60.

Environment	Stress Level (ksi)	Number of Samples
Complete immersion in phosphoric acid-based pretreat	15 25 50	2 3 3
Complete immersion in phosphoric acid-based brine	15 25 50	2 3 3
Complete immersion in baseline sulfuric acid-based pretreat	15 25 20	2 3 3
Complete immersion in baseline sulfuric acid-based brine	15 25 50	2 3 3
Alternate immersion in 3.5% NaCl per ASTM G44	25	3
Tensile tests	N/A	5
Total		40

3.2 Material

Nitinol 60 is an isostatically pressed (at 1,800 °F (982 °C)) bearing material containing approximately 60% nickel and approximately 40% titanium. This material is a shape memory alloy discovered in the late 1950s by the U.S. Naval Ordnance Laboratory. Because of difficulties in producing this material, it was abandoned; however, the composition and processing parameters were recently revived by Summit Materials, LLC under the trademarked name SM-100. Additional information on this alloy is presented in reference 3. MSFC-SPEC-3706[4] establishes the chemical compostion, heat treatment, hardness, and tensile requirements of this alloy.

For traceability purposes, the material certification—obtained from NASA Glenn Research Center—is shown in figure 1. Information included in figure 1 includes specific lot, chemical composition, and form of material.

Material Certification

SUMMIT MATERIALS LLC
1274 Oakridge Road, McDonald, PA 15057

Date: 9/27/13

Customer: NASA/Glenn Research Center

Purchase Order No.: NNC13ME77P

Product: 60NiTiNOL (Ni-40Ti) Billets (with carbon steel container)
Nominal size with container: 114mm Dia x 322mm
Nominal 60NiTiNOL size: 108mm Dia x 326mm
Billet ID Nos: H13-144, H13-145, H13-146

H13-144 Powder Lot 522-023
H13-145 Powder Lot 522-022
H13-146 50% Lot 522-022, 50% Lot 522-023

Product Condition: Hot Isostatically Pressed 1800F (982C)

Chemical Analysis:

Powder Lot Number	522-022, -80 mesh	522-023, -80 mesh
Nickel wt%	60.0	59.9
Carbon wt%	0.020	0.023
Oxygen wt%	0.081	0.075
Nitrogen wt%	0.004	0.008
Hydrogen wt%	0.0010	0.0008
Titanium	Balance	Balance

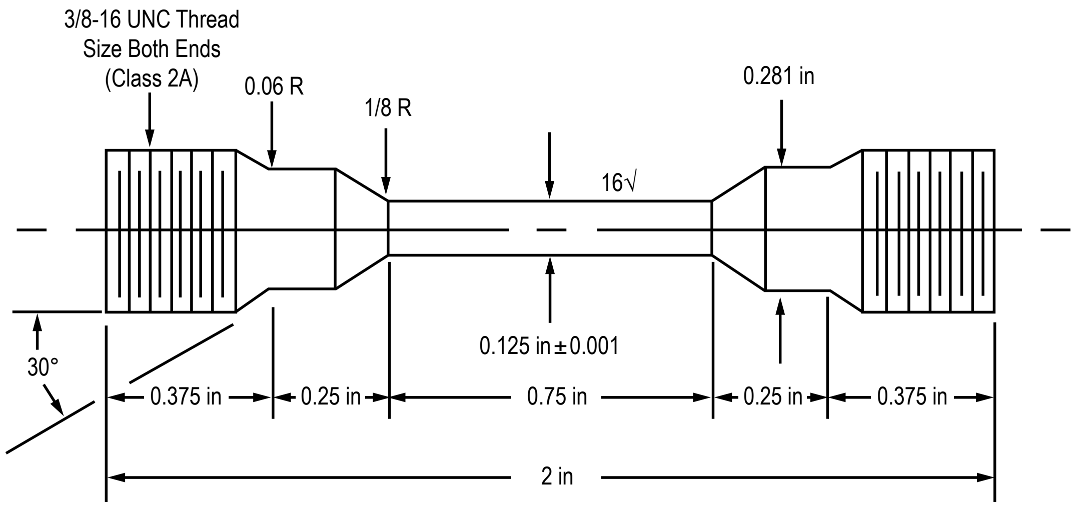
BY: C.F. Yolton

Figure 1. Nitinol 60 test material certification.

Nitinol 60 is a high-strength, low-modulus material, and in this program, it was tested in the hard condition with a Rockwell C hardness of at least 58.

3.3 Specimen Configuration

The drawing for the stress corrosion sample is shown in figure 2. Forty specimens were fabricated per this drawing. A representative machined Nitinol 60 specimen is shown in figure 3.



Notes:

(1) Tolerances: ±0.005 in, except otherwise specified.
(2) Surface finish (arithmetic average roughness value): 16 μin for the reduced section.
(3) Thread dimensions must be as specified. Measurement by fabricator is mandatory.
(4) No undercutting of radii permitted.
(5) Gauge section to be concentric with axis within 0.002 in total indicator reading (gauge section of the tensile specimen cannot have more than 0.002 in total run-out) and parallel.
(6) No file marks or nicks permitted within gauge section.
(7) Drawing not to scale.

Figure 2. Drawing of round tensile specimen.

Figure 3. Photograph of Nitinol 60 round tensile specimen.

3.4 Tensile Tests of Specimens As Received

Five Nitinol 60 specimens were tensile tested per ASTM E8[5] as received to obtain the mechanical properties and the results are presented in table 2. All the five specimens that were tensile tested failed before yielding, therefore, no yield strength values were obtained. The ultimate tensile strength (UTS) values are shown in increasing order in table 2. These values varied significantly, ranging from 146.1 ksi to 199.6 ksi, with an average value of 172.4 ksi. It is common for high hardness materials like this to behave in this manner. Tensile tests curves (load versus displacement) are presented in figure 4. It can be observed from these curves the very low ductility of this material when heat treated as for bearings. The modulus of elasticity (Young's modulus) was very consistent and the average value was 12.9 Msi.

Table 2. Tensile data for Nitinol 60.

Specimen ID	Ultimate Tensile Strength (ksi)	Modulus of Elasticity (Msi)
NiTi60-10	146.1 (minimum value)	12.8
NiTi60-21	148.6	13.2
NiTi60-1	175.6 (median)	12.9
NiTi60-41	192.3	–
NiTi60-31	199.6 (maximum value)	12.7
Average Values	172.4	12.9
UTS standard deviation: 24.52 UTS coefficient of variation: 14.22%		

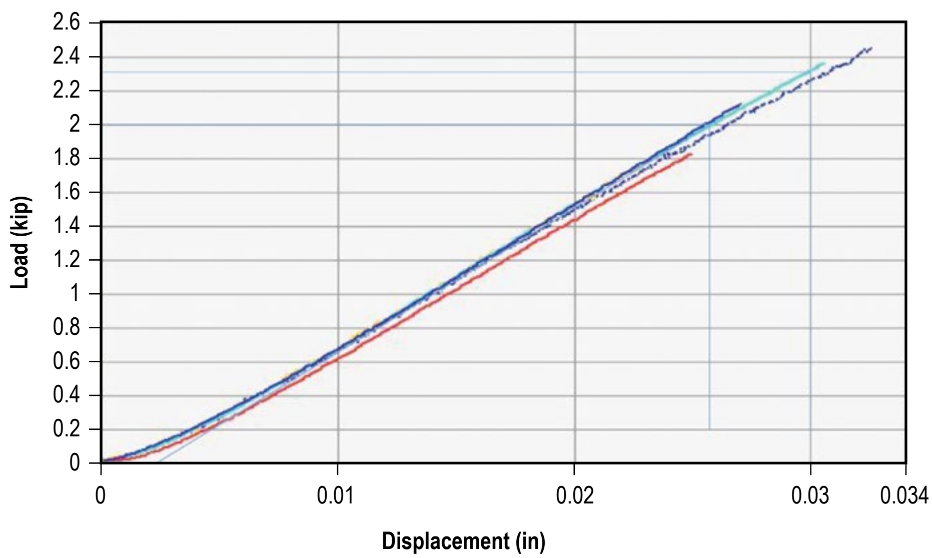


Figure 4. Tensile tests curves for Nitinol 60.

3.5 Stressing of the Specimens

The strain corresponding to the desired stress levels was obtained by using the averaged modulus of elasticity of 12.9 Msi (strain equals stress divided by modulus of elasticity). The specimens were stressed per ASTM G49[6] by using the stressing device shown in figure 5.

The various components of the stressing fixtures are identified in the schematic diagram shown in figure 6.

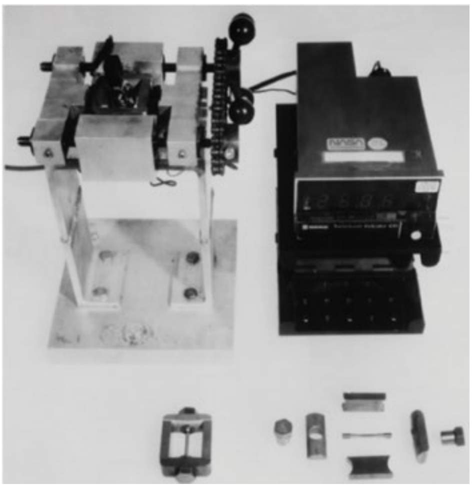


Figure 5. Device for stressing stress corrosion round tensile specimens.

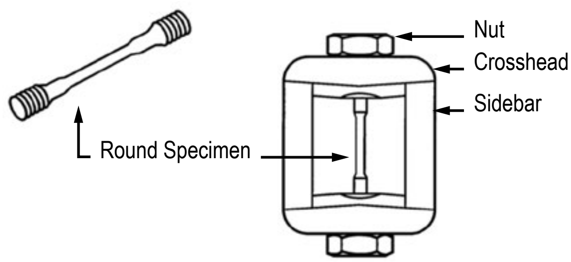


Figure 6. Schematic diagram of specimen and frame assembly.

In this method, the specimen is assembled into the stressing fixtures and an extensometer component is attached on the reduced section of the specimen. Two sidebars are then pushed toward the center by means of the device. The strain is measured by obtaining the difference between the initial and final readings. A representative assembly is shown in figure 7.

Figure 7. Stress corrosion assembly.

3.6 Test Environments

The majority of the specimens in this test program were exposed by total immersion in the four ECLSS solutions used: (1) Sulfuric acid-based pretreat, (2) sulfuric acid-based brine, (3) phosphoric acid-based pretreat, and (4) phosphoric acid-based brine.

These solutions also contain small amounts of chromic acid. The brine for each ECLSS solution was obtained by distillation of the pretreat, removing part of the water, and leaving a more concentrated solution. The pretreat and brine solutions had a pH of approximately 2. Exposure was carried out by completely immersing the specimens in the liquids, which were contained in plastic containers. The specimens were exposed for 1 year. A representative specimen being immersed is presented in figure 8.

Figure 8. Representative Nitinol 60 specimen being immersed.

Several samples were also tested in alternate immersion in 3.5% NaCl per ASTM G44 for comparison. Tests in alternate immersion are performed in a neutral pH. In the alternate immersion method the specimens are exposed to 1-hour cycles. Each cycle includes a 10-minute immersion period in a 3.5% NaCl solution followed by a 50-minute drying period. The cycles continued 24 hours per day for the duration of the test. The alternate immersion apparatus is presented in figure 9.

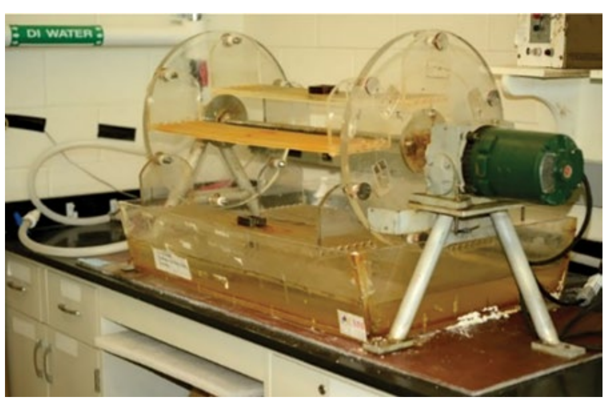


Figure 9. Alternate immersion apparatus.

4. RESULTS AND DISCUSSION

The stress corrosion results presented in table 3 show that no failures occurred in any environment after a year of exposure, which is a significant positive result. Appearance of the specimens after this year-long exposure can be observed in figures 10 through 14.

Table 3. Stress corrosion test results of Nitinol 60.

Test Environment	Stress Level (ksi)	Failure Ratio	Remarks
Sulfuric acid-based pretreat	15 25 50	0/2 0/3 0/3	No failures in a year-long exposure
Sulfuric acid-based brine	15 25 50	0/2 0/3 0/3	No failures in a year-long exposure
Phosphoric acid-based pretreat	15 25 50	0/2 0/3 0/3	No failures in a year-long exposure
Phosphoric acid-based brine	15 25 50	0/2 0/3 0/3	No failures in a year-long exposure
3.5% NaCl alternate immersion per ASTM G44	25	0/3	No failures in a year-long exposure

Notes:
- Forty specimens were received for this evaluation. Five of them were used for tensile data and the rest were used for the SCC evaluation as shown above.
- Failure ratio is the number of failures over number of specimens tested at the same condition.

Figure 10. Nitinol 60 specimens after a year-long exposure to sulfuric acid-based pretreat.

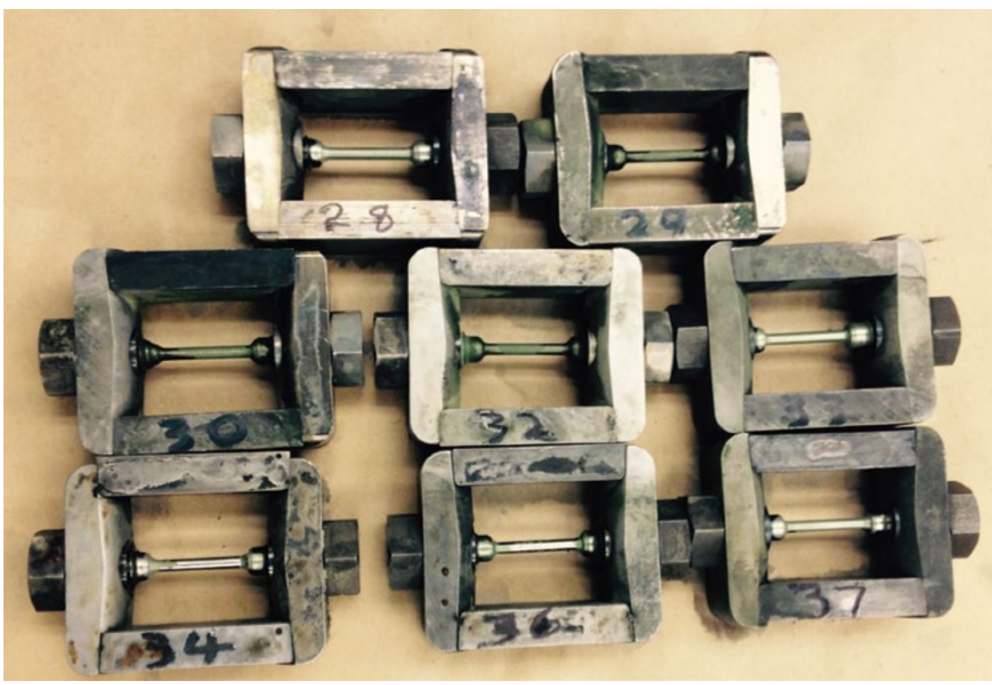


Figure 11. Nitinol 60 specimens after a year-long exposure to sulfuric acid-based brine.

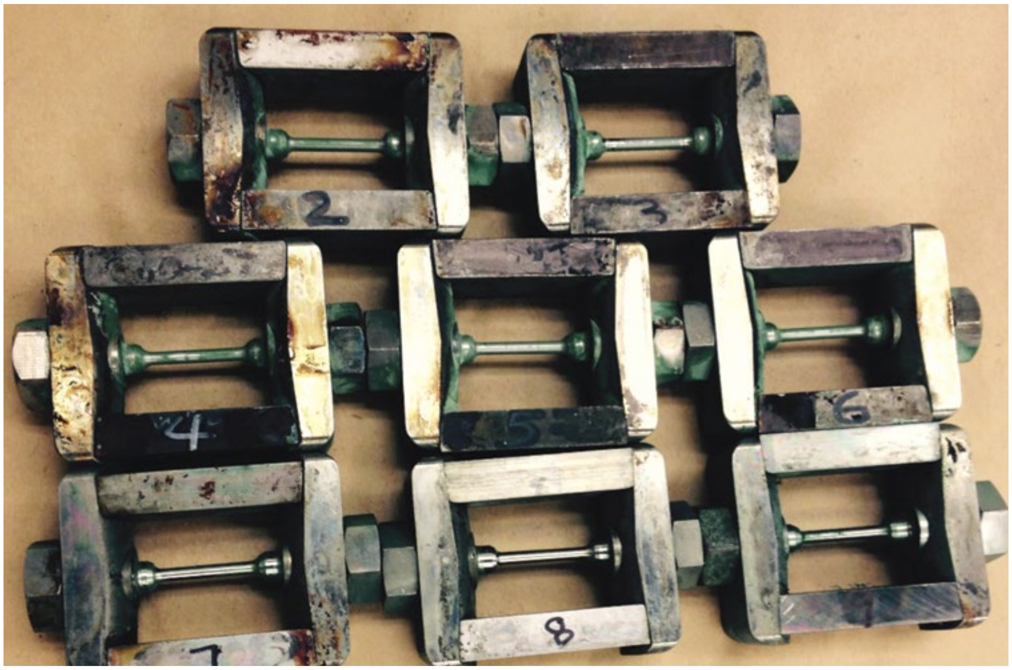


Figure 12. Nitinol 60 specimens after a year-long exposure to phosphoric acid-based pretreat.

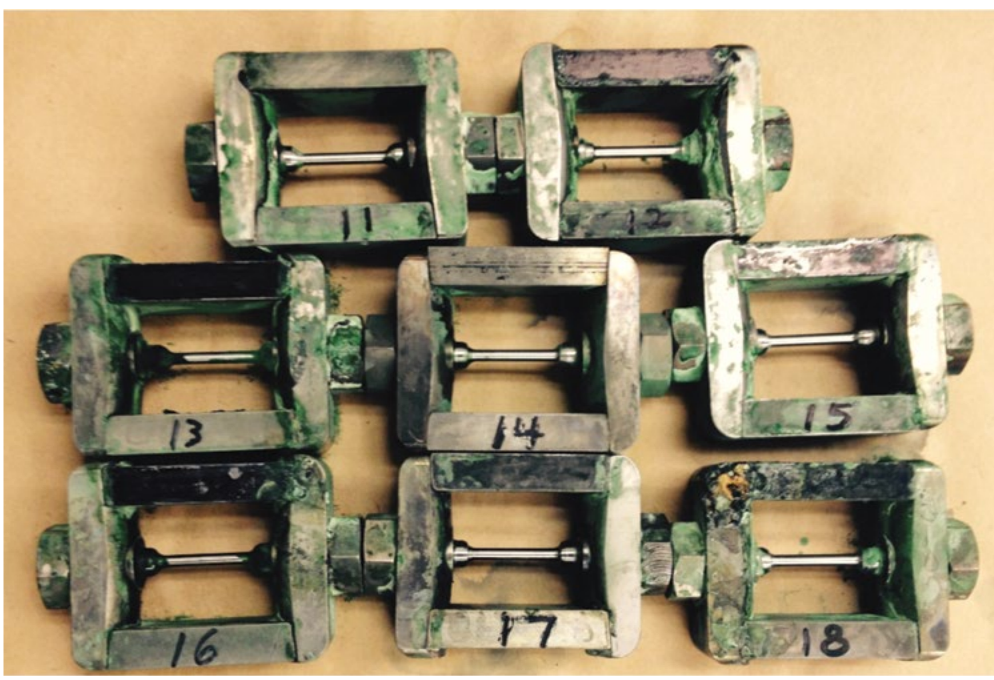


Figure 13. Nitinol 60 specimens after a year-long exposure to phosphoric acid-based brine.

Figure 14. Nitinol 60 specimens after a year-long exposure to 3.5% NaCl alternate immersion per ASTM G44.

The three specimens that were exposed for a year in alternate immersion were unloaded, then, one was reloaded to 75 ksi, one was reloaded to 100 ksi, and one was reloaded to 125 ksi. Afterwards all three samples were exposed for 32 additional days in alternate immersion. No failures occurred, as shown in table 4. Though this result is good, only one sample was tested at each stress level. More samples would be needed to better assess the stress corrosion resistance at these higher stress.

Table 4. Extended SCC evaluation in 3.5% NaCl alternate immersion.

Sample Number	Stress Level (ksi)	Failure Ratio	Days to Failure
38	75	0/1	No failures after the 32 additional days
39	100	0/1	
40	125	0/1	

At the end of all the final exposures, the specimens were cleaned and unloaded. Representative specimens were subjected to metallography and the results are presented in figures 15 through 19. Metallography did not reveal corrosion or stress corrosion attack on any of the specimens.

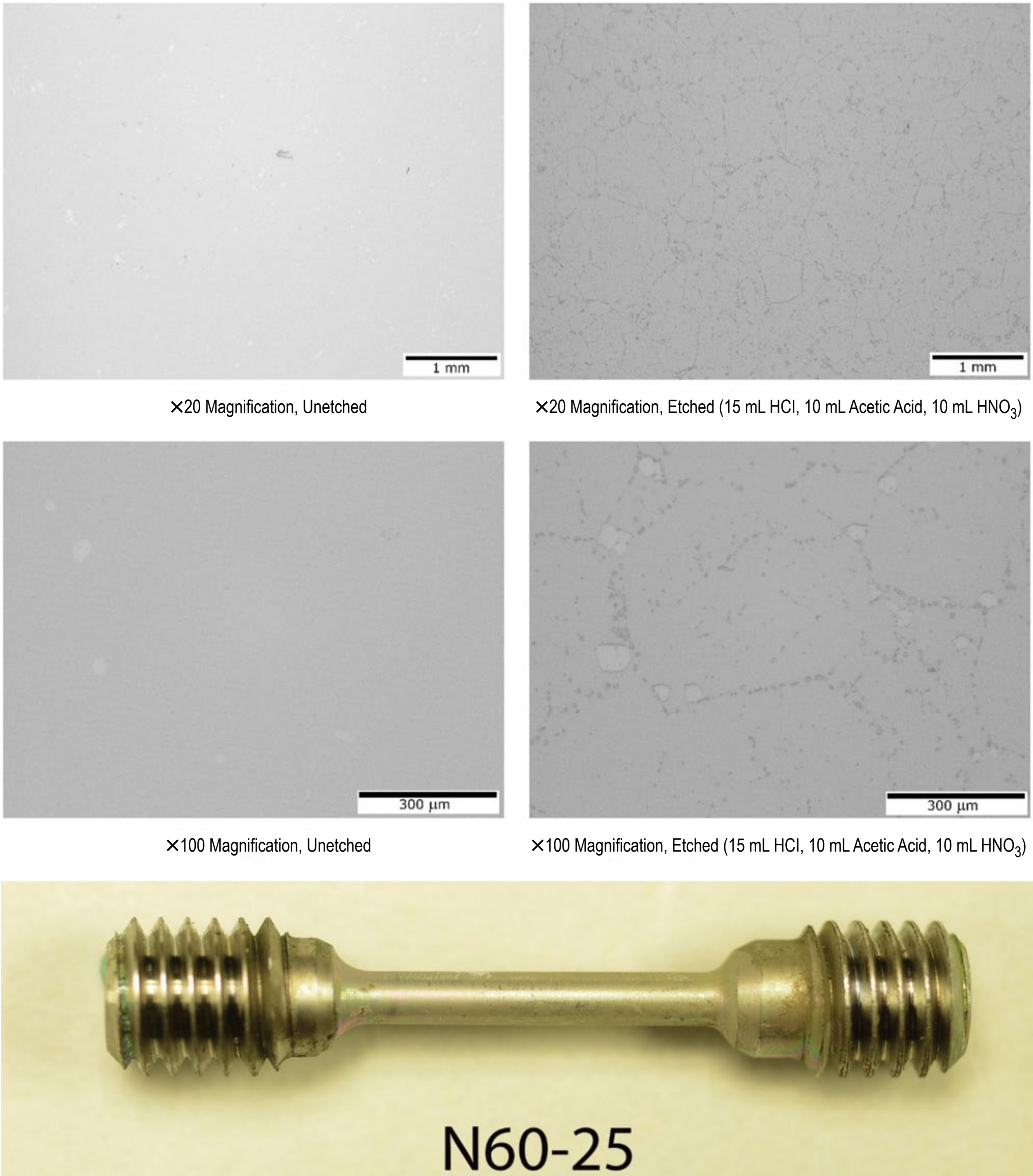
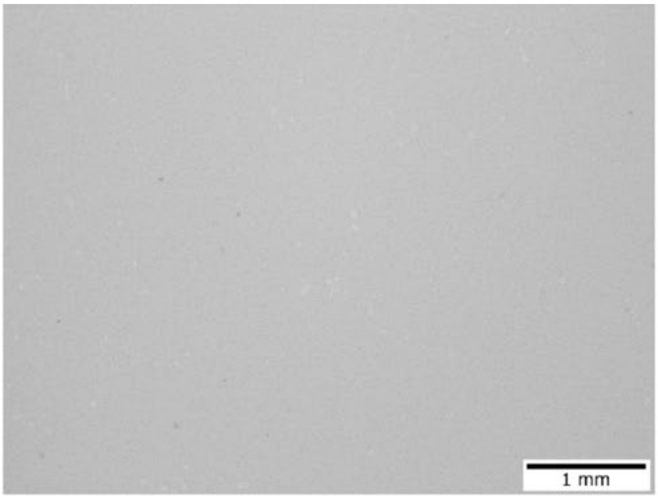


Figure 15. Metallographic and overall views of Nitinol 60 stressed to 50 ksi after a year-long exposure to sulfuric acid-based pretreat.



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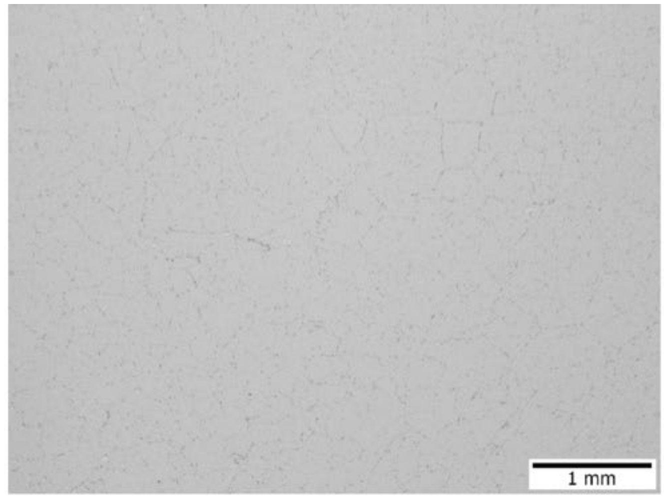

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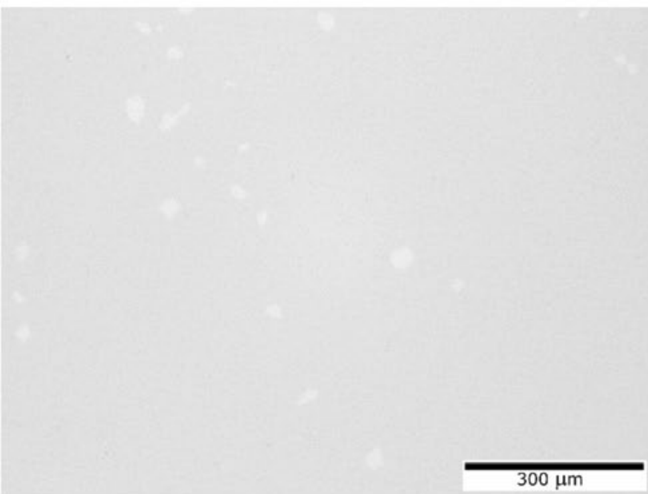

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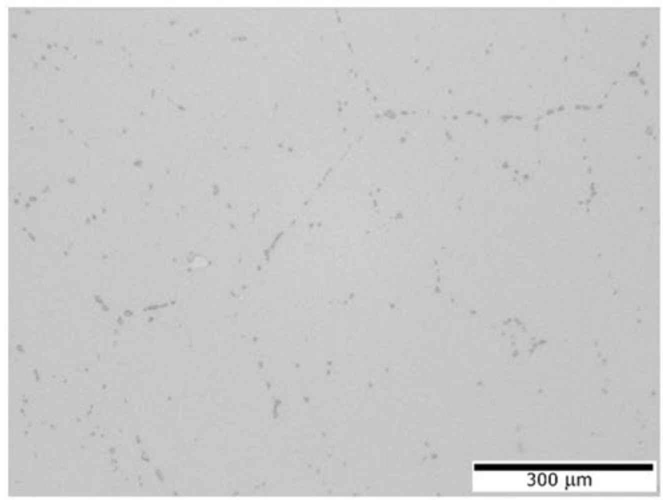

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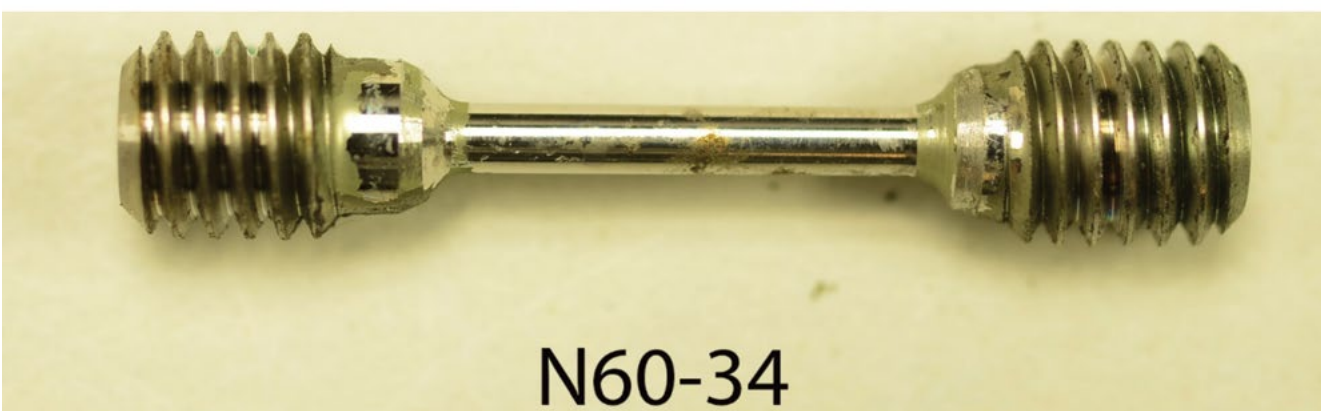
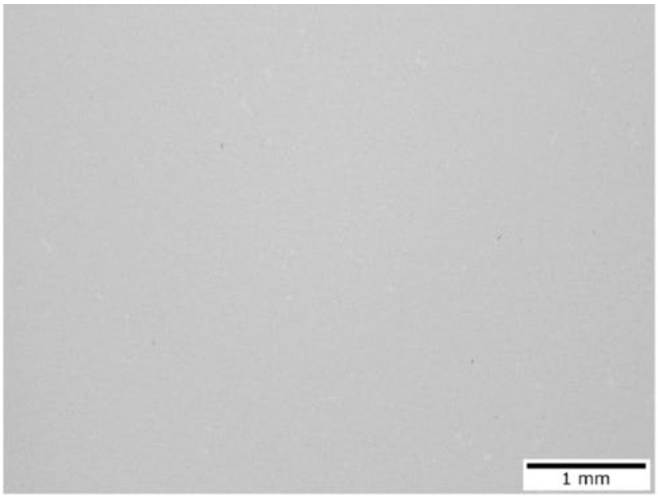
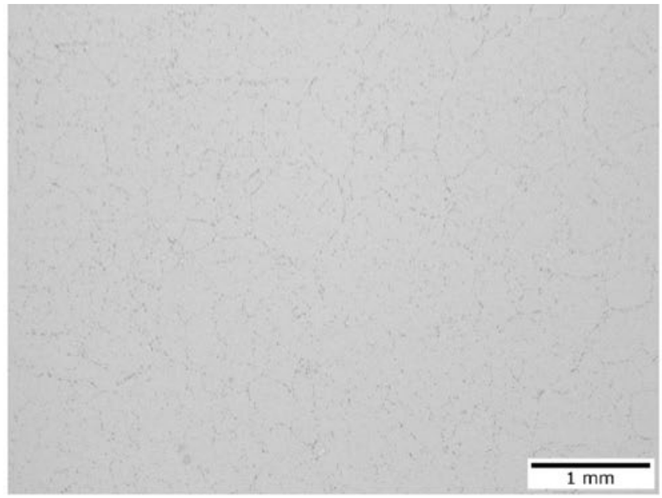


Figure 16. Metallographic and overall views of Nitinol 60 stressed to 50 ksi after a year-long exposure to sulfuric acid-based brine.





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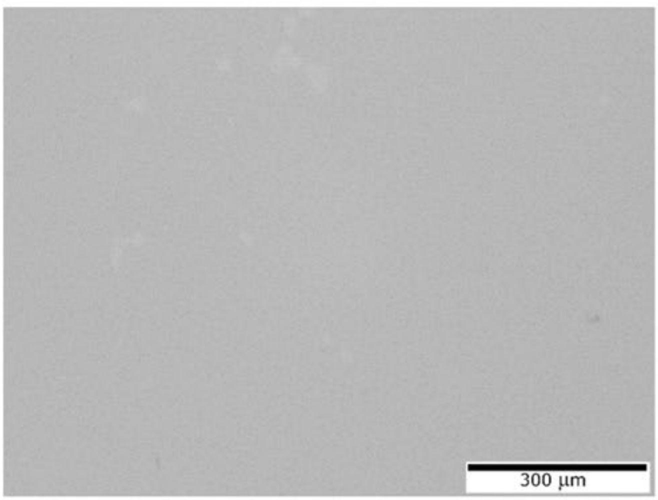

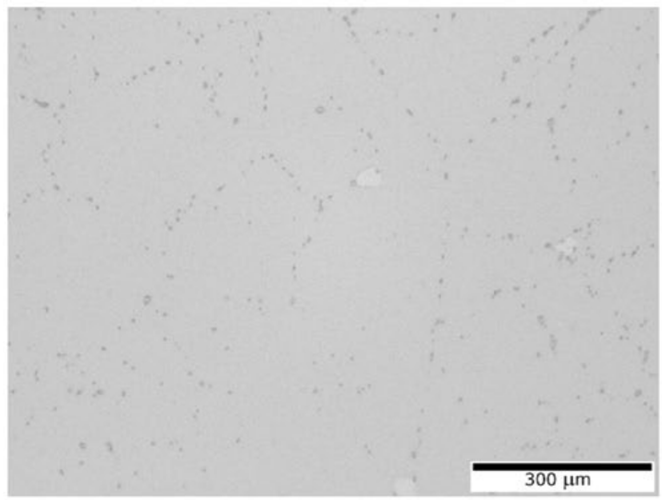

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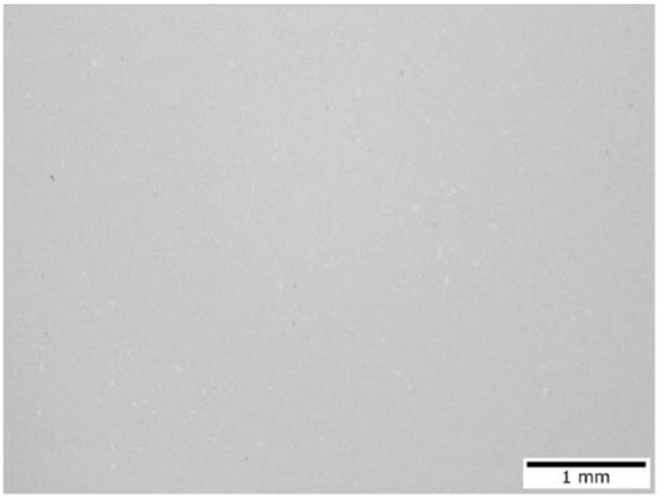
×100 Magnification, Etched (15 mL HCl, 10 mL Acetic Acid, 10 mL HNO_3)

Figure 17. Metallographic and overall views of Nitinol 60 stressed to 50 ksi after a year-long exposure to phosphoric acid-based pretreat.



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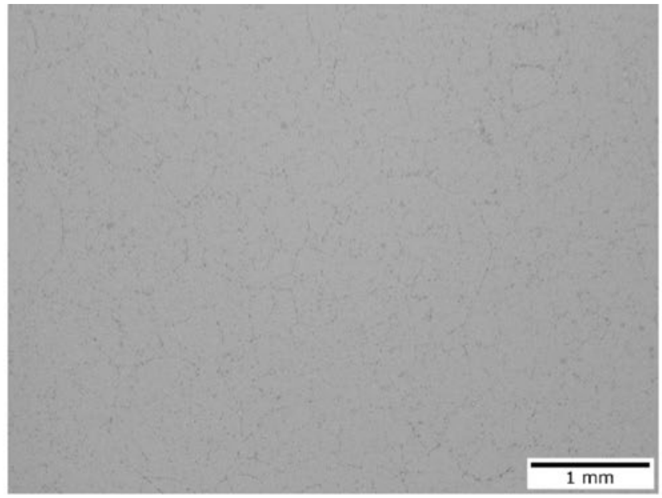

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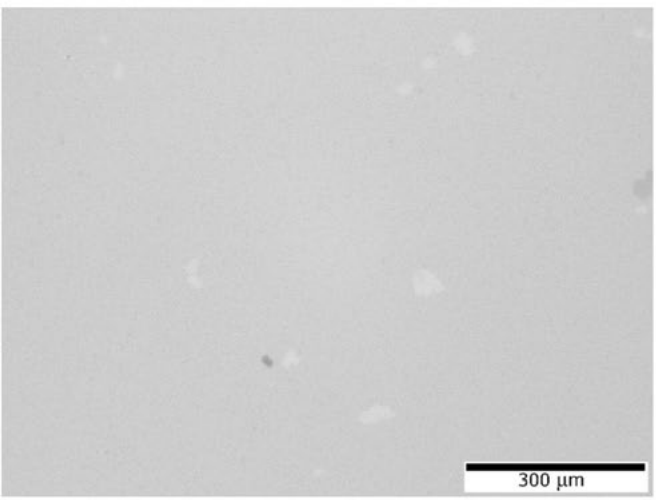

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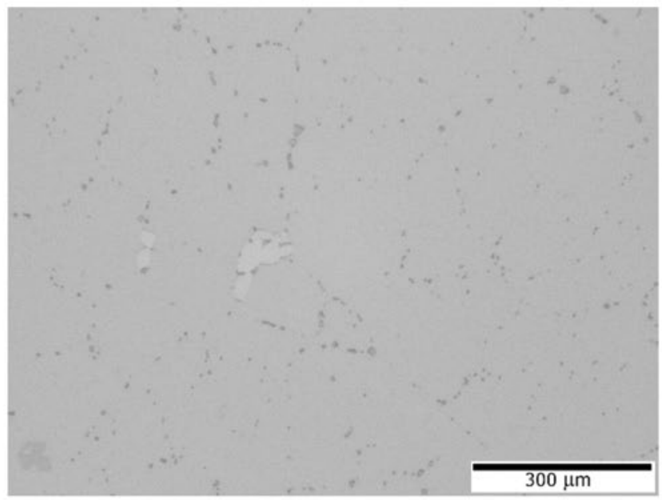

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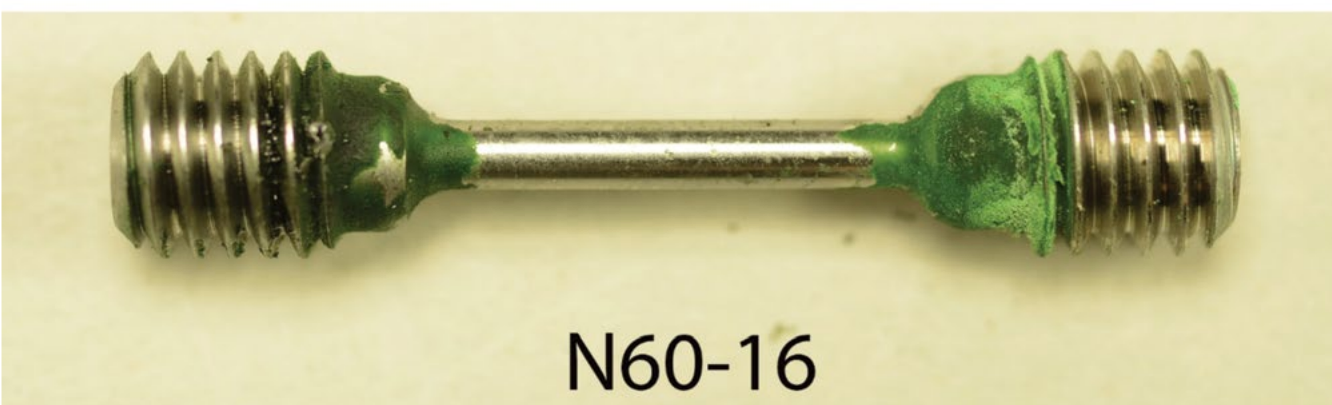
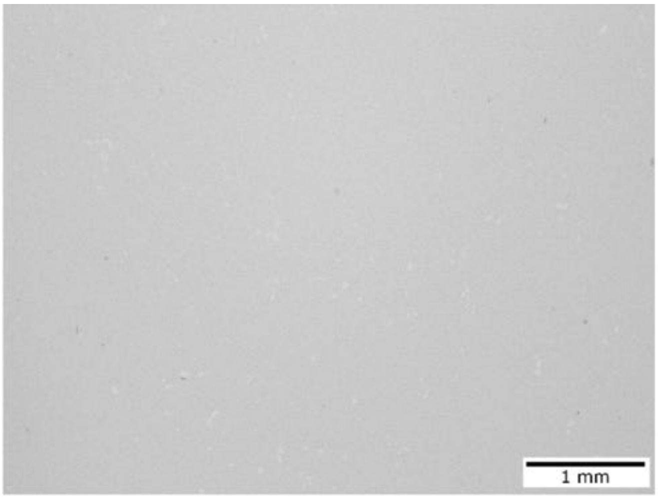


Figure 18. Metallographic and overall views of Nitinol 60 stressed to 50 ksi after a year-long exposure to phosphoric acid-based brine.



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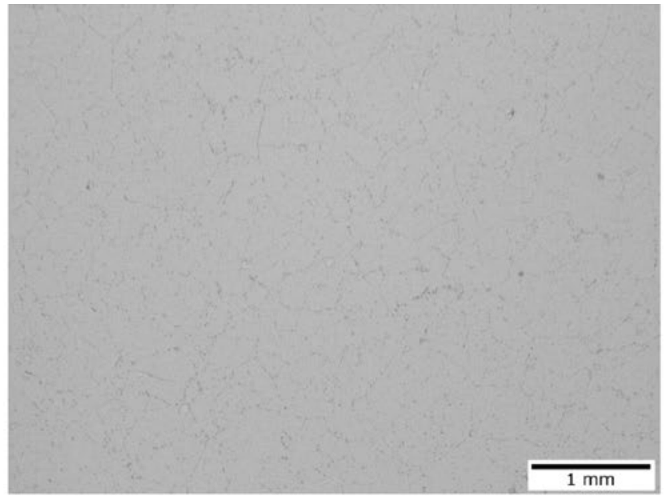

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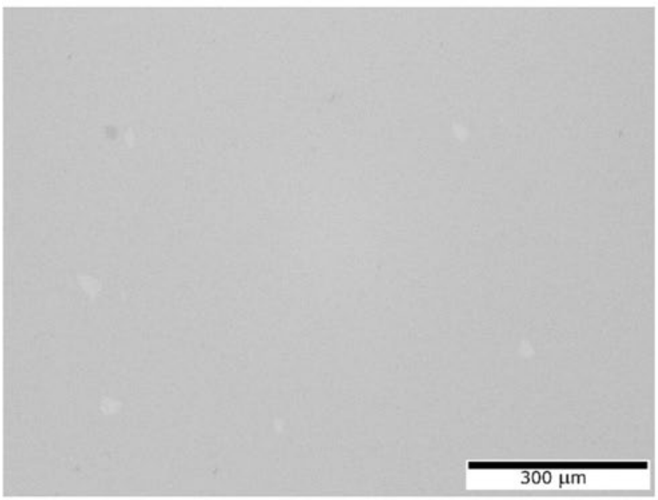

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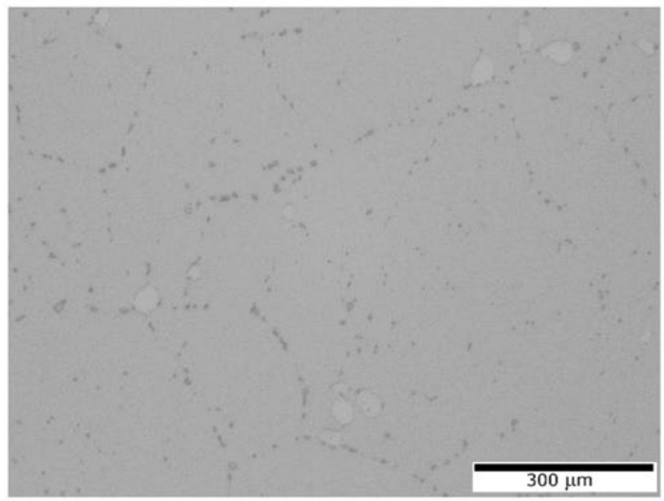


×100 Magnification, Etched (15 mL HCl, 10 mL Acetic Acid, 10 mL HNO_3)

Figure 19. Metallographic and overall views of a Nitinol 60 specimen after exposure to 3.5% NaCl alternate immersion (365 days at 25 ksi and 32 days at 75 ksi).

The remaining stress corrosion specimens were tensile tested to determine final ultimate tensile strength values. The results are presented in table 5 and plotted in figure 20. In addition to post-test ultimate tensile strength values, table 5 contains the ratio of each one of those values to the average tensile strength of nonexposed specimens (which was 172.4 ksi). The final ultimate tensile strength (UTS_f) values ranged from 126.1 ksi (for a specimen exposed to sulfuric acid-based brine) to 184.7 ksi (for a specimen exposed to sulfuric acid-based pretreat). Summarized values for each environment, shown in increasing order of average final ultimate tensile strength, are presented in table 6. The lower the ratio of final to initial tensile strength, the more the corrosive effect of the environment. Therefore, based on the average ratios shown in table 6 obtained after 365 days for the ECLSS environments, they can be listed from most corrosive to least corrosive as follows (the average ultimate tensile strength ratio is shown in parenthesis): (1) Sulfuric acid-based brine (0.85 average ratio), (2) phosphoric acid-based brine (0.87 ratio), (3) sulfuric acid-based pretreat (0.92 ratio), and (4) phosphoric acid-based pretreat (0.98 ratio).

Table 5. Tensile strength data for exposed Nitinol 60 specimens following exposure to various environments.

Specimen Number	Test Environment	Stress Level (ksi)	UTS_f (ksi)	UTS_f/ UTS_i	Evaluation Performed	Remarks
20	Sulfuric acid-based	15	158.4	0.92	Tensile test	Broke at end of gauge length
19	pretreat (365 days)	15	–	–	Metallography	
23		25	127.1	0.74	Tensile test	Broke at end of gauge length
24		25	166.5	0.97	Tensile test	Broke at end of gauge length
22		25	–	–	Metallography	
26		50	184.7	1.07	Tensile test	Broke in two places
27		50	153.2	0.89	Tensile test	Broke at end of gauge length
25		50	–	–	Metallography	
		Averages	158.0	0.92		
29	Sulfuric acid-based	15	143.2	0.83	Tensile test	Broke at end of gauge length
28	brine (365 days)	15	–	–	Metallography	
32		25	150.1	0.87	Tensile test	Broke at end of gauge length
33		25	126.1	0.73	Tensile test	Broke at end of gauge length
30		25	–	–	Metallography	
36		50	152.0	0.88	Tensile test	Broke at end of gauge length
37		50	159.1	0.92	Tensile test	Broke in two places
34		50	–	–	Metallography	
		Averages	146.1	0.85		
3	Phosphoric	15	148.4	0.86	Tensile test	Broke in two places
2	acid-based pretreat	15	–	–	Metallography	
	(365 days)					
5		25	158.0	0.92	Tensile test	Broke in two places
6		25	177.2	1.03	Tensile test	Broke at gauge length
4		25	–	–	Metallography	
8		50	176.1	1.02	Tensile test	Broke at gauge length
9		50	181.1	1.05	Tensile test	Broke at end of gauge length
7		50	–	–	Metallography	
		Averages	168.2	0.98		
12	Phosphoric	15	166.3	0.96	Tensile test	Broke at end of gauge length
11	acid-based brine	15	–	–	Metallography	
	(365 days)					
14		25	133.7	0.78	Tensile test	Broke at end of gauge length
15		25	130.8	0.76	Tensile test	Broke in two places
13		25	–	–	Metallography	
17		50	152.4	0.88	Tensile test	Broke at end of gauge length
18		50	168.9	0.98	Tensile test	Broke at end of gauge length
16		50	–	–	Metallography	
		Averages	150.4	0.87		
39	3.5% NaCl	1 yr at 25 ksi + 32 days at 100 ksi	145.2	0.84	Tensile test	Broke at gauge length
40	alternate immersion	1 yr at 25 ksi + 32 days at 125 ksi	150.0	0.87	Tensile test	Broke near end of gauge length
38	per ASTM G44	1 yr at 25 ksi + 32 days at 75 ksi	–	–	Metallography	
	(397 days)	Averages	147.6	0.86		

Notes:

- UTS_i = Initial average ultimate tensile strength of nonexposed specimens = 172.4 ksi. Values range from 146.1 to 199.6 ksi.
- UTS_f = Final ultimate tensile strength of exposed specimens.

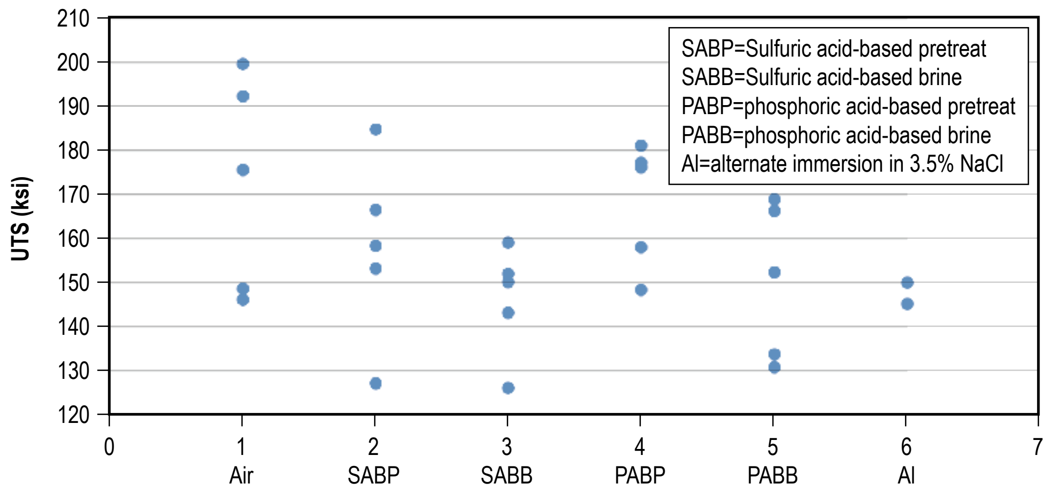


Figure 20. Graphic showing variation of ultimate tensile strength for Nitinol 60 exposed to various environments.

Table 6. Summarized tensile strength data for exposed Nitinol 60 specimens following exposure to various environments.

Test Environment	Average UTS_f (ksi)	Range of UTS_f (ksi) (Maximum minus minimum in parentheses)	Median UTS_f (ksi)	Average UTS_f/UTS_i	Median UTS_f/UTS_i	Range of UTS_f/UTS_i
Sulfuric acid-based brine (365 days)	146.1	126.1–159.1 (33 ksi)	150.1	0.85	0.87	0.73–0.92
3.5% NaCl AI (397 days)	147.6	145.2–150 (4.8 ksi)	147.6	0.86	0.86	0.84–0.87
Phosphoric acid-based brine (365 days)	150.4	130.8–168.9 (38.1 ksi)	152.4	0.87	0.88	0.76–0.98
Sulfuric acid-based pretreat (365 days)	158	127.1–184.7 (57.6 ksi)	158.4	0.92	0.92	0.74–1.07
Phosphoric acid-based pretreat (365 days)	168.2	148.4–181.1 (32.7 ksi)	176.1	0.98	1.02	0.86–1.05

Notes:
- UTS_i = Initial average ultimate tensile strength of nonexposed specimens = 172.4 ksi. Values range from 146.1 to 199.6 ksi.
- UTS_f = Final ultimate tensile strength of exposed specimens.

The average ratio of final to initial tensile strength for the specimens exposed for 397 days in 3.5% NaCl alternate immersion was 0.86.

The ultimate tensile strength ratios shown provide an indication of the effect each environment had on Nitinol 60 in particular, and that effect may not necessarily be the same for other materials. Also, because of the scatter in the data, these values should be considered approximations. Though these results suggest some reduction in tensile properties took place, they are in contrast with the metallography results, which did not show any corrosion. Table 7 shows modulus of elasticity, fracture elongation, and reduction of area values after exposure. The modulus of elasticity values, in particular, were very consistent with an average value of 13 Msi. Average fracture elongation and reduction of area were 1.29% and 1.30%, respectively.

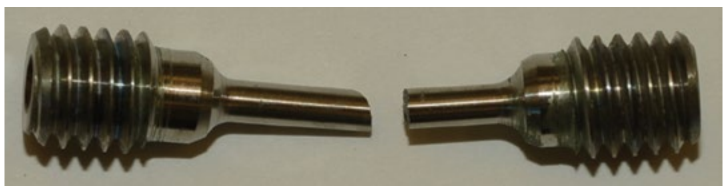
Table 7. Modulus of elasticity, fracture elongation, and reduction of area for Nitinol 60 after exposure.

Test Result	Maximum	Minimum	Median	Mean	Standard Deviation	Coefficient of Variation (%)
Modulus of Elasticity (Msi)	13.6	12.3	13	13	0.39	2.97
Fracture Elongation (%)	1.59	1.04	1.26	1.29	0.15	11.46
Reduction of Area (%)	3.18	–	1.60	1.30	1.12	86.45

Representative samples that were tensile tested are shown in figure 21. The samples did not experience any necking-down effect (localized area reduction of the specimen during plastic deformation) because of the low ductility of this material when it is in the hard condition. Also notice the tendency of these samples to break close to the end of the reduced section. Of the 22 specimens that were tensile tested after exposure, 14 broke at or near the end of the reduced section, 5 broke in two places, and only 3 broke within the gauge length. Stress-strain curves for these samples are shown in figure 22.

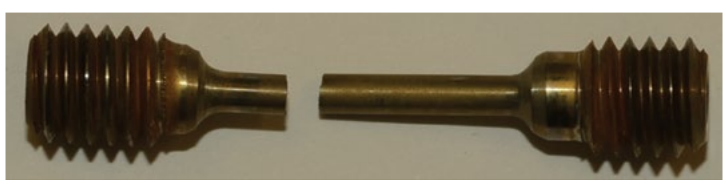
Sample N60-27-SABP-50: Stressed to 50 ksi, exposed for 1 year in sulfuric acid-based pretreat, and tensile tested to failure (153.2 ksi UTS)

Sample N60-36-SABB-50: Stressed to 50 ksi, exposed for 1 year in sulfuric acid-based brine, and tensile tested to failure (152 ksi UTS)



Sample N60-8-PABP-50: Stressed to 50 ksi, exposed for 1 year in phosphoric acid-based pretreat, and tensile tested to failure (176.1 ksi UTS)

Sample N60-17-PABB-50: Stressed to 50 ksi, exposed for 1 year in phosphoric acid-based brine, and tensile tested to failure (152.4 ksi UTS)



Sample N60-39-AI-25: Stressed to 25 ksi, exposed for 1 year in NaCl alternate immersion, reloaded to 100 ksi and exposed for 32 additional days, and then tensile tested to failure (145.2 ksi UTS)

Figure 21. Stress corrosion specimens exposed to various environments and tensile tested.

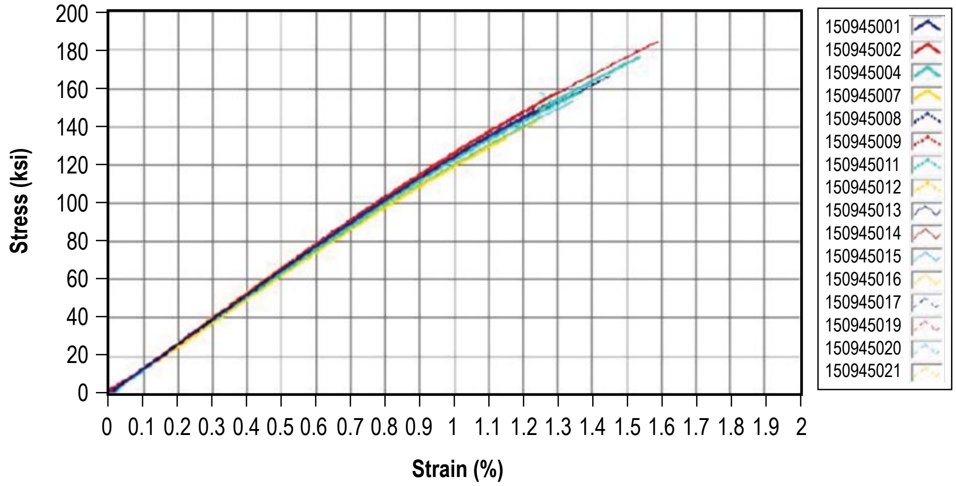


Figure 22. Stress-strain curves for stress corrosion specimens exposed to various environments

5. CONCLUSIONS

All the stress corrosion specimens exposed to the ECLSS solutions (sulfuric acid-based pretreat, sulfuric acid-based brine, phosphoric acid-based pretreat, and phosphoric acid-based brine) survived the year-long stress corrosion test at stress levels up to 50 ksi. The complementary specimens exposed to 3.5% NaCl alternate immersion per ASTM G44 also survived the year-long stress corrosion test at a stress level of 25 ksi, as well as the 32-day extended test at stress levels of up to 125 ksi. Metallographic examinations of representative specimens did not detect stress corrosion cracks on the specimens. However, the average ultimate tensile strength of exposed specimens were lower than the average for the initially tested specimens in air with no exposure. From the stress corrosion standpoint, that reduction is not expected to be a deterrent for the proposed use of this alloy as a bearing material, since for that type of applications, the stresses are in compression.

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*
01–11–2016

2. REPORT TYPE
Technical Memorandum

3. DATES COVERED *(From - To)*

4. TITLE AND SUBTITLE
Stress Corrosion Evaluation of Nitinol 60 for the International Space Station Water Recycling System

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
P.D. Torres

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
George C. Marshall Space Flight Center
Huntsville, AL 35812

8. PERFORMING ORGANIZATION REPORT NUMBER
M–1421

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
Washington, DC 20546–0001

10. SPONSORING/MONITOR'S ACRONYM(S)
NASA

11. SPONSORING/MONITORING REPORT NUMBER
NASA/TM—2016–218230

12. DISTRIBUTION/AVAILABILITY STATEMENT
Unclassified-Unlimited
Subject Category 26
Availability: NASA STI Information Desk (757–864–9658)

13. SUPPLEMENTARY NOTES
Prepared by the Materials and Processes Laboratory, Engineering Directorate

14. ABSTRACT
A stress corrosion evaluation was performed on Nitinol 60 because this alloy was proposed to be used as a bearing material for the Urine Processing Assembly of the Environmental Control and Life Support System (ECLSS) of the International Space Station. The specimens exposed to the ECLSS solutions (sulfuric acid-based pretreat, sulfuric acid-based brine, phosphoric acid-based pretreat, and phosphoric acid-based brine) survived the year-long stress corrosion test at stress levels up to 50 ksi. Metallography did not detect stress corrosion cracks, however, reduction in tensile strength took place. That is not expected to be a deterrent for the use of this material for bearings as stresses will be in compression.

15. SUBJECT TERMS
International Space Station, Environmental Control and Life Support System, Urine Processing Assembly, stress corrosion cracking, corrosion, Nitinol 60, urine pretreatment, urine brine, water recycling in the Space Station, water recovery in the Space Station

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
U	U	U	UU	40	STI Help Desk at email: help@sti.nasa.gov
					19b. TELEPHONE NUMBER *(Include area code)* STI Help Desk at: 757–864–9658

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39-18

National Aeronautics and
Space Administration
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