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THE STRESS-CORROSION CRACKING BEHAVIOR
OF THE U-4.2 wt% Nb ALLOY AGED
80 HOURS AT 260°C

James M. Macki

Robert L. Kochen

DOW CHEMICAL U.S.A.
ROCKY FLATS DIVISION
P. O. BOX 888
GOLDEN, COLORADO 80401

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ROCKY FLATS DIVISION
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THE STRESS-CORROSION CRACKING BEHAVIOR OF THE U-4.2 wt% Nb ALLOY AGED 80 HOURS AT 260°C

James M. Macki and Robert L. Kochen

Abstract. The stress-corrosion cracking (SCC) behavior of the U-4.2 wt% Nb alloy aged 80 hours at 260°C has been investigated. Smooth four-point bend SCC tests and smooth static tensile SCC tests were used. The results of this study show that the aged U-4.2 wt% Nb alloy is susceptible to SCC when stressed and exposed to water containing oxygen and chloride ions. The SC cracks which nucleate in the aqueous chloride environments proceed by an intergranular SCC fracture mode. No failures were observed in dry air containing less than 200-ppm moisture or in 100% relative humidity air. The surface tensile stress required for SCC is less than 20,000 psi in oxygen-saturated water containing 50-ppm chloride ions.

INTRODUCTION

This report is a summary of stress-corrosion cracking (SCC) experimental work on the aged U-4.2 wt% Nb binary alloy.

For this investigation, the U-4.2 wt% Nb alloy is quenched from 850°C and then aged 80 hours at 260°C. This produces equiaxed grains with the α'' crystal structure. The α'' structure is a monoclinic transition phase which can contain 3.7 to 6.9 wt% Nb in a super-saturated solid solution. The aged material exhibits an average 0.2% yield strength of 180,000 psi, an average ultimate tensile strength of 200,000 psi, an average elongation of 6%, and an average elastic modulus of 18.3×10^6 psi.¹ Typical specifications for the gamma-quenched and the age-hardened alloy are listed in Table 1.²

Table 1. Typical Specifications for the U-4.2 wt% Nb Alloy.²

Property	Gamma-Quenched	Aged 80 hours at 260°C
Yield Strength (0.2% offset)	45,000 psi	175,000 psi
Tensile Strength	150,000 psi	190,000 psi
Elastic Modulus	12×10^6 psi	18×10^6 psi
Reduction of Area	25%	4%
Elongation	23%	4%
Hardness	R _A 65	R _A 75
Impact Strength	—	4.5 ft-lb
Density	17.9 ± 0.2 g/cm ³	18.0 ± 0.2 g/cm ³
Grain Size	60 microns	60 microns

The U-Mo and U-Nb binary alloys and the ternary U-7.5 wt% Nb-2.5 wt% Zr (Mulberry) alloy are known to be susceptible to stress-corrosion cracking.³⁻¹⁰ Two modes of

stress-corrosion crack propagation, intergranular and transgranular, have been observed in U-Nb alloys.^{7,10} Mixed transgranular and intergranular SCC modes have also been reported.^{7,9,10}

This investigation was conducted to determine some of the environmental parameters which influence SCC in the aged U-4.2 wt% Nb alloy. Smooth, rather than precracked, SCC specimens were used for the tests.

Smooth SCC tests involve loading a specimen to some stress level, exposing the stressed specimen to a test environment, and recording the time-to-failure (TTF). This TTF is the sum of the time to nucleate an SCC crack and the time to propagate the crack through the specimen.

These TTF results are quantitative only for the specific specimens used for the tests; however, these TTF data do provide qualitative information on the SCC behavior of the alloy in the test environments.

EXPERIMENTAL

For this investigation, two types of smooth SCC tests were used: constant deflection four-point bend tests, and static tensile tests. The four-point bend test simulates a residual stress condition; whereas, the static tensile test simulates an applied load condition.¹¹ The bend and tensile specimens were obtained from flat plates.

Figure 1 shows a bend specimen mounted in the stressing device. The rectangular bend specimen is 5.5-inches long by 0.75-inch wide by 0.10-inch thick. The distance between the two middle contact cylinders is 2 inches so that the uniformly stressed region is about 1.5 square inches. The bend specimen is loaded to the required stress level by advancing the bolt and measuring the deflection of the specimen with the deflection meter. The applied stress is calculated from the specimen dimensions and the geometry of the stressing device using the expression:¹¹

$$\sigma = \frac{12Et\delta}{[3L^2 - 4A^2]}$$

Figure 1. A Four-Point Bend Specimen Mounted in the Stressing Device.

where,

- σ = maximum applied tensile stress (psi)
- E = elastic modulus (psi)
- t = thickness of the specimen (inches)
- δ = deflection of the mid point of the specimen (inches)
- L = distance between the two outer contact points (4 inches)
- A = distance between each outer contact point and the adjacent inner contact point (1 inch).

The applied tensile stress, σ , is constant over the convex surface of the bent specimen between the two inner contact points.

Four-point bend specimens were stressed to 75,000 psi and 130,000 psi and exposed to four test environments; dry air, humid air, salt fog, and oxygen-saturated water containing 50 ppm chloride ions. The dry air environment was room temperature ($26 \pm 2^\circ\text{C}$) air with 10 to 200 ppm moisture in a desiccator containing silica gel. The moisture in the desiccator was continuously monitored using a Panametrics humidity probe. The humidity probe was calibrated by both Panametrics and the Rocky Flats Mass Spectroscopy Laboratory. The dry air environment contained 10 ppm H_2O according to the Panametrics calibration and 200 ppm H_2O according to the Rocky Flats calibration.

The humid air environment was 100% relative humidity (condensing) air at $32 \pm 1^\circ\text{C}$. The salt fog environment was an aqueous 3.5 wt% NaCl solution dispersed as a fog in a salt fog chamber maintained at $50 \pm 2^\circ\text{C}$.

The oxygen-saturated water containing 50 ppm chloride ions was distilled water with NaCl added to provide a 50 ± 1 ppm chloride ion concentration. Oxygen saturation was obtained by continuously bubbling oxygen through the solution. The aqueous solution was at room temperature ($26 \pm 2^\circ\text{C}$).

The static tensile test specimen and test chamber are shown in Figure 2. The static tensile test specimens are pin-loaded tensile test type specimens with an elongated mid section. The mid section is 3-inches long and has a rectangular cross section 0.200-inch wide by 0.080-inch thick. The tensile specimen is mounted into a glass environmental chamber with nylon end caps. All joints are sealed with silicone rubber (Dow Corning 3145 RTV® adhesive/sealant). Chloride ions are not leached from this silicone rubber by aqueous solutions.

Figure 2. Static-Tensile Stress-Corrosion Cracking Specimen and Environmental Chamber.

Two inches of the 3-inch mid section of each tensile specimen are exposed to the test environment inside the glass chamber. The specimen and chamber are mounted onto a stressing device which works by means of lead bricks loaded onto the end of a lever beam as shown in Figure 3. The load is applied after the test solution has been squirted into the chamber through the fill tube. Oxygen or nitrogen is continuously bubbled through the solution during the test.

Figure 3. Stress-Corrosion Cracking Specimen and Environmental Chamber Mounted in Stressing Device.

The static tensile SCC specimens were loaded to stress levels between 20,000 psi and 100,000 psi, and exposed to three aqueous test environments at room temperature ($26 \pm 2^\circ\text{C}$). The specimens were exposed to the test environment for at least one hour before being stressed. The three test environments were oxygen-saturated distilled water, oxygen-saturated water containing 50-ppm chloride ions, and nitrogen-saturated (deoxygenated) water containing 50 ppm chloride ions. The chloride ion concentration in the distilled water was less than 0.5 ppm. Oxygen-saturation and nitrogen-saturation were achieved by continuously bubbling oxygen or nitrogen through the water in the environmental chamber. The dissolved oxygen concentration in the nitrogen-saturated water was not measured.

The 50-ppm chloride ion concentration was achieved by adding NaCl to distilled water. The chloride ion concentration was determined using an Orion specific ion electrode with an Orion Model 801 analyzer. The distilled water-NaCl solutions all had initial chloride ion concentrations of 50 ± 1 ppm.

The SCC tests were continued for at least one year or until fracture occurred. The time-to-failure (TTF) of each static tensile SCC specimen was recorded to within ± 8 minutes by a multichannel recorder. The TTF of each four-point bend specimen was measured with varying precision depending on the TTF. For TTF less than 6 hours, the precision was

± 15 minutes; for TTF between 6 and 294 hours, the precision was ± 12 hours. After 294 hours, the precision decreased to ± 32 hours.

Fractured specimens were examined metallographically and the fracture surfaces were examined using a scanning electron microscope (SEM). The metallographic and SEM examinations were conducted to determine the microstructure and the fracture modes.

Pieces of some fractured specimens were sent to the Analytical Laboratory for niobium assays and impurity analyses.

The pH and chloride ion concentration of the used test solution were determined whenever possible.

RESULTS

The results of the four-point bend SCC tests are summarized in Table 2. The results of the static tensile SCC tests are summarized in Table 3 and in Figure 4. Representative photomicrographs from the metallographic and SEM examinations are reproduced in Figures 5 through 16. The chemical and spectrographic analyses results obtained from fractured specimens are shown in Table 4. The hydrogen content of ten static tensile specimens was 5 ± 3 ppm.

Table 2. The Results of the Four-Point Bend SCC Tests on U-4.2 wt% Nb Aged 80 Hours at 260°C .

Stress ^a (ksi)	Environment	Time-to-Failure (hours)	Test Time on Remaining Specimens ^f (number/hours)
75	Dry Air ^b	None	3/9890, 3/10348
130	Dry Air	None	4/9890
75	Humid Air ^c	None	2/9888, 2/10299
100	Humid Air	None	2/10299
130	Humid Air	None	5/9888, 2/10348
75	Salt Fog ^d	0.3, 0.3, 0.3	
130	Salt Fog	0.1, 0.3, 2.1, 2.5	
130	O ₂ -H ₂ O-Cl ^e	2, 3, 3.5, 5	

^aThe applied stress is calculated using 18×10^6 psi for the modulus.

^bThe dry air environment is air containing 10 to 200 ppm moisture at $26 \pm 2^\circ\text{C}$.

^cThe humid air environment is 100% relative humidity air at $32 \pm 2^\circ\text{C}$.

^dThe salt fog environment consists of air with an aqueous 3.5 wt% NaCl solution dispersed as a fog at $50 \pm 2^\circ\text{C}$.

^eO₂-H₂O-Cl⁻ represents oxygen-saturated water containing 50 ppm chloride ions at $26 \pm 2^\circ\text{C}$.

^fTest times are as of noon, November 1, 1971.

Table 3. The Results of the Static Tensile SCC Tests on U-4.2 wt% Nb Aged 80 Hours at 260°C.

Stress (ksi)	Environment ^a	Time-to-Failure (hours)
50 ^b	O ₂ -H ₂ O ^c	2390, 4301, 4222
50 ^b	O ₂ -H ₂ O-Cl ^{-d}	1, 2, 7, 8, 28
50 ^e	N ₂ -H ₂ O-Cl ^{-f}	7, 73, 108, 274, 1068
100 ^b	O ₂ -H ₂ O-Cl ⁻	0.2, 0.2, 0.5
100 ^e	N ₂ -H ₂ O-Cl ⁻	0.2, 0.2, 0.5, 1, 18

^aAll test temperatures were 26 ± 2°C.^bThe cross-sectional area of the specimens was 0.014 in² (0.200 ± 0.002 inch by 0.070 ± 0.001 inch).^cOxygen-saturated distilled water containing <0.5 ppm chloride ion.^dOxygen-saturated distilled water containing 50 ppm chloride ions.^eThe cross-sectional area of the specimens was 0.012 in² (0.200 ± 0.002 inch by 0.060 ± 0.001 inch) except for the 73 hour specimen which was 0.013 in² (0.200 inch by 0.067 inch).^fNitrogen-saturated distilled water containing 50-ppm chloride ions.**Table 4. The Results of the Laboratory Analyses on U-4.2 wt% Nb SCC Specimens in ppm Unless Otherwise Noted.^{a,b}**

Sample	C	O	Fe	Si	wt% Nb	Al	Be	Cu	Mn	Ni
Tensile 1	90	80	60	40	4.38-4.33	30	5	10	5	5
Tensile 2	90	30	50	40	4.39-4.39	40	1	10	5	5
Tensile 3	100	40	40	40	4.31-4.29	30	5	10	5	5
Tensile 4	100	70	30	40	5.22-5.19	40	5	10	5	5
Tensile 5	30	30	30	30	4.27-4.27	20	5	5	5	<1
Tensile 6	20	30	20	40	4.41-4.40	20	1	10	5	<1
Bend 1	70	58	48	80	4.32	10	<1	10	<10	10
Bend 2	90	47	49	60	4.40	20	<1	10	<10	10

^aC and O by a Leico total combustion analyzer.

Si and Fe by atomic absorption.

Nb by gravimetric analyses.

Remainder by emission spectrographic analyses.

^bEmission spectroscopic analyses also showed all the U-4.2 wt% Nb specimens contained:

< 1 ppm of B, Co, Li, Mg, Pb, Sb, and Sn.

<10 ppm of Ba, Bi, Ca, Cd, Cr, K, Mo, Na, Ti, and V.

<25 ppm of Zn.

The U-4.2 wt% Nb alloy corroded, in the oxygen-saturated water containing chloride ions, to produce an adherent black oxide and a loose, bright-yellow oxide. X-ray diffraction identified the black oxide as UO₂ and the yellow oxide as UO₃·2H₂O. In the salt fog environment, a loose black oxide was observed and was identified by X-ray diffraction to be UO_{2,3}.

In the nitrogen-saturated water containing 50 ppm chloride ions and in the distilled water, only UO₂ was observed. No chloride compounds were detected after any of the SCC tests.

Figure 4. The SCC of Aged U-4.2 wt% Nb in Oxygen-Saturated Water Containing 50-ppm Chloride Ions. The cross-sectional area of the specimens was 0.025 in.² (0.123 ± 0.003 in. by 0.202 ± 0.003 in.).**Figure 5. Fracture Edge of Four-Point Bend Specimen Showing Intergranular Failure after 3½ Hours at 130 ksi Applied Stress in Oxygen-Saturated Water Containing 50-ppm Chloride Ions.** (1) Edge of fracture surface, and (2) secondary crack. 100X.

Figure 6. Intergranular Secondary Crack Parallel to and $\frac{1}{8}$ Inch from Fracture Surface of Four-Point Bend Specimen after 2 Hours at 130 ksi Applied Stress in Oxygen-Saturated Water Containing 50-ppm Chloride Ions. (1) Top surface, (2) secondary crack. 150X.

Figure 7. Mosaic of Static Tensile Specimen Fracture Edge after 28 Hours at 50 ksi in Oxygen-Saturated Water Containing 50-ppm Chloride Ions. (1) Intergranular SCC, (2) secondary intergranular SC crack, (3) transgranular mechanical fracture, and (4) shear lip. 100X.

Figure 8. Stress-Corrosion-Crack Fracture Edge and Intergranular SCC Secondary Cracks of Static Tensile Specimen Shown in Figure 7. (1) Fracture edge, (2) intergranular secondary SC cracks. 400X.

Figure 9. Fracture Edge of Static Tensile Specimen after 1677 Hours at 20 ksi in Oxygen-Saturated Water Containing 50 ppm Chloride Ions. Note heavy precipitation along grain boundaries indicating improper heat treatment. 100X.

Figure 10. Scanning Electron Microscope (SEM) Fractograph of Static Tensile Stress-Corrosion Cracked Specimen after 7 Hours at 50 ksi in Oxygen-Saturated Water Containing 50-ppm Chloride Ions. (1) Edge of Specimen, (2) Intergranular SCC region, and (3) mechanical fracture region. 100X.

Figure 11. An SEM Fractograph of Specimen in Figure 10 Showing Fracture Inclusion and Microvoid Coalescence Fracture Surface in Mechanical Failure Region. 4000X.

Figure 12. An SEM Fractograph of Specimen in Figure 10 Showing Fracture Surface in Intergranular-to-Transgranular Transition Region. Note striations on transgranular fracture surface. 2000X.

Figure 14. An SEM Fractograph of Specimen in Figure 13 Showing Intergranular SCC Fracture Surface. 500X.

Figure 13. An SEM Fractograph of Static Tensile SCC Specimen after 274 Hours at 50 ksi in Nitrogen-Saturated Water Containing 50-ppm Chloride Ions. (1) Edge of specimen, (2) intergranular SCC region, and (3) mechanical fracture region. 100X.

Figure 15. An SEM Fractograph of Specimen in Figure 13 Showing Fracture Surface in Intergranular-to-Transgranular Transition Region. Note striations on transgranular fracture surface. 1000X.

Figure 16. An SEM Fractograph of Specimen in Figure 13 Showing Microvoid Coalescence Fracture Surface in Mechanical Failure Region. 5000X.

DISCUSSION

The results summarized in Tables 2 and 3 and in Figures 4 through 16 show that the aged (80 hours at 260°C) U-4.2 wt% Nb alloy will stress-corrosion crack when stressed and exposed to oxygen-saturated water containing 50-ppm chloride ions and that the stress-corrosion cracks propagate by an intergranular SCC mode.

The TTF's in nitrogen-saturated water containing 50-ppm chloride ions vary from 7 to 1068 hours (Table 3). This suggests that oxygen may be required for SCC and the Times-to-Failure represent the times required for the necessary oxygen to get into the environmental chamber.

Similarly, the extremely long lives of the SCC specimens in oxygen-saturated distilled water (Table 3) show that chloride ions are required for, or at least accelerate, the nucleation and/or propagation of SCC cracks. The chloride ion concentration in the oxygen-saturated distilled water was initially less than 0.5 ppm; however, additional water was added during the tests to replace that lost by evaporation into the oxygen bubbles. This would add some chloride ions to the environment. The long TTF in the distilled water environments would also permit chloride ions to concentrate at local regions of anodic activity.

Therefore, the SCC which eventually occurred in the oxygen-saturated distilled water environment does not necessarily conflict with the chloride ion requirement conjecture.

The absence of failures in the four-point bend specimens in the 100% relative humidity air (Table 2) supports the chloride ion requirement conjecture, since the specimen surfaces in this environment are subjected to condensing (distilled) water. The SCC apparently will not nucleate and/or propagate on stressed specimens in humid air in the absence of chloride ions.

The relationship between applied stress and the TTF of the static tensile specimens in oxygen-saturated water containing 50-ppm chloride ions is shown in Figure 4. In Figure 4, the log of the TTF is plotted as a function of the applied stress. A threshold stress appears to exist at around 20,000 psi below which these particular specimens may not be susceptible to SCC in the test environment. One specimen loaded to 20,000 psi failed after 1677 hours and its duplicate failed after 5590 hours. Metallographic examination of the specimen that failed after 1677 hours (Figure 9) showed α -precipitation along the grain boundaries, indicating that this specimen had not been properly heat-treated (the other specimens exhibited the normal equiaxed, single phase microstructure). The apparent SCC threshold shown in Figure 4, if it exists, only applies to the specimens that were tested; specimens with different geometries would probably exhibit different threshold values. The significance of Figure 4 is that it shows an abrupt change in slope at about 20,000 psi which corresponds to a practical SCC threshold for these specimens.

The SEM fractographs and the metallographs demonstrate that the SCC cracks which nucleate in the aqueous environments propagate by an intergranular SCC mode. The photomicrographs in Figures 5 and 6 show that the SCC mode is intergranular and that more than one intergranular crack nucleated before the bend specimen fractured. Figures 7 and 8 show similar behavior for the static tensile test specimens. The SEM fractographs in Figures 10 through 16 also show that the SC cracks propagate by means of an intergranular fracture mode. Figures 10 and 13 indicate that the SC crack nucleated near a corner of the specimen; similar features were observed on all the static tensile SCC specimen fracture surfaces that were examined.

CONCLUSIONS

The U-4.2 wt% Nb alloy is susceptible to SCC when stressed and exposed to water containing oxygen and chloride ions.

The SCC proceeds by an intergranular fracture mode.

Chloride ions, oxygen, and moisture may all three be required to nucleate the intergranular SCC fracture.

The tensile stress required for SCC is less than 20,000 psi in oxygen-saturated water containing 50-ppm chloride ions.

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