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Corrosion Fatigue Life Evaluation of 17-4PH in Support of Water Filled Turbine Hubs

Final Report

February 2025

Mychailo B. Toloczko Ryan A. Bouffioux Ferdinan Cintron Colin Ziqing Zhai



Prepared for the U.S. Army Corps of Engineers under Contract DE-AC05-76RL01830

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Pacific Northwest National Laboratory Richland, Washington 99354

Summary

High cycle corrosion fatigue (HCCF) life S-N tests were conducted on wrought 17-4PH in various water environments under accelerated corrosion fatigue conditions. Cyclic loading of tensile specimens was conducted at 2 Hz with an R value of 0.07. The method of corrosion and stress corrosion acceleration was to conduct the tests at a mildly elevated temperature. For tests conducted in aerated Columbia River water (CRW) and in aerated ASTM Type IV water, at ~10⁶ cycles, the HCCF stress amplitude was ~70% of the value in air. For deaerated water, the value was ~80%. These values are consistent with the limited literature data on HCCF life of 17-4PH in water environments.

The measured fatigue life both in air and water greatly exceed the ASME trend curve for high cycle fatigue life of moderate and high strength steels. Measured values also greatly exceed values found in a hydropower-relevant study in the literature on cast 17-4PH.

The HCCF life of 17-4PH exposed to CRW with added Amersite 61W was evaluated as part of this study and found to cause very low failure times compared to all the other water environments that were used. Amersite 61W is a commercial corrosion inhibitor that is typically marketed for use in boiler systems.

Post-test specimen examinations indicate that hydrogen embrittlement is likely to be the environmental degradation mechanism driving the reduced time to crack initiation and specimen failure during the HCCF tests. This is consistent with conclusions made by several other researchers who have investigated HCCF of 17-4PH and similar materials in water environments.

Other degradation mechanisms, in particular, crevice corrosion, may play a role during a 50+ year life of WFH BACH. The potential effects of crevice corrosion on HCCF life were reviewed as part of a literature study performed for the USACE, and crevice-affected HCCF life testing was a desired addition to the test matrix but could not be included due to project constraints. The primary concern for this degradation mode is high surface stress locations. It is recommended that additional consideration be given to crevice corrosion in defining optimal component corrosion fatigue life estimates. This could consist of finite element analysis to analyze the change in local stress based on hypothesized amounts of material loss due to corrosion along with simple, long term crevice testing.

Since tensile S-N testing is essentially a measure of the time to crack initiation, estimated WFH BACH life estimates may also be more fully informed by conducting corrosion fatigue crack growth rate testing of 17-4PH to assess the time needed for a crack to grow to a critical length.

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Acronyms and Abbreviations

A61W	Amersite 61W
ACRW	aerated Columbia River water
ASTM	American Society for Testing and Measurement
AT4W	ASTM Type 4 water
BACH	blade angle control hardware
BSE	backscattered electron
CA6NM	a cast, tempered martensitic steel containing ~12 wt% chromium
CGR	crack growth rate
CRW	Columbia River water
da/dN	crack advance per load or strain cycle
DCRW	deaerated Columbia River water
DCPD	direct current potential drop
DO	dissolved oxygen
EDS	energy-dispersive X-ray spectroscopy
EPA	Environmental Protection Agency
HCCF	high cycle corrosion fatigue
HCF	high cycle fatigue
ISO 9001	International Standards Organization standard for quality management
K	stress intensity factor
MIC	Microbial induced corrosion
MSS	martensitic stainless steel
NADCAP	National Aerospace and Defense Contractors Accreditation Program
PH	precipitation hardened
PNNL	Pacific Northwest National Laboratory
S-N curve	A curve on a plot that shows the number of cycles to crack initiation or
specimen failure for a	given applied stress amplitude
Sa	stress amplitude
SCC	stress corrosion cracking
SCCGR	stress corrosion crack growth rate
SCCI	stress corrosion crack initiation
SE	secondary electron
SEM	scanning electron microscope
UE	uniform elongation
UNF	unfailed
USACE	US Army Corps of Engineers
UTS	ultimate tensile stress
WFH	water-filled hub
YS	yield stress

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1.0 Introduction

Kaplan turbines are one of several types of turbines used in hydropower systems. It is a turbine with an adjustable blade angle to optimize efficiency as a function of the river flow rate and power setting. Within the hollow turbine hub (Figure 1) are the blade angle control hardware (BACH). A servo motor, which is a hydraulically piston, moves a crosshead along the axis of the hub. For each blade, the BACH hardware consists of an eyelet attached to the crosshead, a lever that rotates the blade, two links that span between the eyelet and the lever, and two pins that transmit load to the links (Figure 2). Movement of the crosshead rotates the levers that are bolted to the blades. This approach to blade angle control is used by all current Kaplan turbines, including those used by the US Army Corps of Engineers (USACE).



Figure 1. Detail drawing of an example Kaplan runner hub internals used to control blade angle. Blades are not shown in this sketch.



Figure 2. Sketch of BACH provided by the USACE.

1.1 Mechanical Loading Conditions

The servomotor that controls blade angle is actuated by hydraulic fluid, and a position sensor is used as the feedback mechanism to control fluid pressure. The feedback loop between the crosshead position and pressure control hardware operates at a frequency of ~0.0055 Hz (a period of 3 minutes). As with any feedback-based control system, constant small back-and-forth adjustments to blade angle occur that cause alternating loads on the BACH. In addition to these feedback-driven load variations to the BACH, load changes also arise from directed changes in blade angle in response to river head and flow conditions coupled with the power setting that occurs on a longer time scale. Load changes associated with these two types of blade angle movement are roughly the same, and the overall frequency of the change in stress remains ~0.0055 Hz. The mean force applied by the crosshead is to push on the BACH at low blade angles and pull on the BACH at high blade angles. The most common operating angle for John Day Kaplan turbines is in the regime where the crosshead is exhibiting a mean pulling force on the BACH which resists the hydraulic thrust of the water passing the turbine blades.

While BACH are designed to withstand the fatigue loading induced by the blade angle control operations, failures of these parts have occurred at John Day Dam and other USACE hydro power installations [USACE 2009]. Failures have occurred in pins, links, eye ends, and stud bolts with the pins being the most frequent. The most common failure point is at the small radius of the pin diameter changed location (Figure 3). Every time the blade position is changed, that small radius is exposed to a changing stress. While the small radius reduces the magnitude of the stress riser in that region, the failures at John Day are nevertheless attributed to sufficiently high stresses that allowed for fatigue cracking and failure.



Figure 3. Diagram of a Kaplan runner hub link pin provided by the USACE.

1.2 Water-Filled Hubs

All Kaplan hydropower systems in operation in the USA utilize an oil-filled hub. The USACE is now considering whether to switch to a water filled hub (WFH) to reduce the possibility of oil leakage into river systems. The use of water rather than oil creates a significant concern for a reduction in BACH lifetime due to the combined effects of corrosion and fatigue. Water-filled Kaplan turbine hubs are now widely used in Europe with adoption beginning approximately 30 years ago. As a result, challenges associated with using water are better understood, including the corrosion fatigue life of BACH. However, the USACE desires to make its own independent assessment as part of its goal to achieve at least a 50-year BACH lifetime with a WFH.

1.3 Approach to Water Quality in Water-Filled Hubs

Corrosion processes take place even in ultra-high purity water without any dissolved gases. The presence of selected impurities such as chlorides can greatly accelerate corrosion. Chlorides and other accelerating species are often present, sometimes in large quantities, in a variety of common water sources, including municipal water. It is recommended that the USACE include water quality specifications in their WFH operations and maintenance procedures to avoid unintended usage of water that may accelerate corrosion and corrosion fatigue.

1.4 17-4PH Information

This material is classified as a martensitic stainless steel (MSS), and more specifically, a high strength MSS. It is part of a class of materials called precipitation hardened (PH) MSS. It derives its corrosion resistance primarily from its chromium, nickel, and copper content, and as with any steel, corrosion resistance is improved by minimizing sulfur content. Its high strength is due in part to it undergoing an austenitic to martensitic phase transformation upon cooling from the solutionizing temperature and due to the formation of copper precipitates during subsequent heat treating. The martensitic transformation causes a large amount of deformation that drives the instantaneous formation of a very fine grain size and a high dislocation density, both of

which contribute to alloy strength. The strength, ductility, and to some extent, the corrosion resistance are tuned by the heat treating process that controls the formation of precipitates and the amount of other microstructural phases such as martensite, ferrite, and (retained) austenite. 17-4 PH is solution annealed at 1040°C, air or oil cooled to below 30°C (a.k.a. Condition A) and peak hardened by aging at 482–510°C (900–950°F). In the solution annealed condition, its yield strength (YS) is typically 750 MPa. Its YS can be as high as 1200 MPa after aging at 482°C. Aging promotes precipitation of coherent copper-rich clusters. Aging above 482°C leads to a decrease in strength and an increase in toughness with increasing aging duration. The Nb/C ratio has a significant influence on the impact toughness of this steel and Nb/C ratio should be maintained to achieve good toughness [Das 2006].

2.0 Corrosion Fatigue Life Testing Approach

Corrosion fatigue S-N data are commonly obtained from tests conducted at cyclic loading frequencies much higher than the expected operating frequency of the component. This allows tests to be completed in a reasonable amount of time, but it is a compromise approach because corrosion and stress corrosion processes are time dependent. Ideally, both the mechanical loading frequency and the corrosion processes would be accelerated simultaneously by the same factors. The approach taken for this test effort was to attempt to conduct tests via simultaneously accelerated loading frequency and corrosion processes.

In many cases, stress corrosion has a strong temperature dependence. An example for 300series stainless steel crack growth in chloride salt solutions and in deliquescent humid air chloride salt conditions is provided in Figure 4 [Bryan 2016, Toloczko 2020]. These are not corrosion fatigue crack growth tests, but rather true stress corrosion crack growth rate (SCCGR) tests conducted at constant load to show only the stress corrosion contribution to crack growth. In this example, a test temperature of 90°C produces an ~200x higher SCCGR than tests conducted at 22°C.

Another example of the dependence of SCCGR on temperature is shown in Figure 5 for Alloy 600 (a Ni-base alloy) measurements in high temperature water [Zhai 2020]. A complementary plot for Alloy 600 stress corrosion crack initiation (SCCI) in high temperature water is provided in Figure 6 [Etien 2011]. A strong temperature dependence on stress corrosion can again be seen. Note that the temperature dependence of Alloy 600 SCCI differs from the SCCGR temperature dependence, but the values are not substantially different, showing that, at least for this combination of material and environment, the accelerating effect of temperature is approximately the same for SCCI and SCCGR.



Figure 4. Literature compilation of 300-series stainless steel CISCC growth rate data [Bryan 2016]. Data points in gray are from air evaluations with deliquescent salts while immersed salt brine evaluations are colored.



Figure 5. PNNL-measured stress corrosion crack growth rates of Alloy 600 in high temperature water showing an activation energy of 117-160 kJ/mol [Zhai 2020].





If corrosion fatigue S-N curves are to be generated from higher frequency tests to reduce the time needed to obtain data, the test temperature should be increased by an amount that produces an increase in stress corrosion response that is proportional to the cyclic loading frequency increase. Thus, if the cyclic loading frequency is increased by 10x, then the test temperature should be increased such that the stress corrosion contribution is increased by 10x.

Research published by Thomas and Wei [Thomas 1992] contains data that allow a cursory evaluation of this approach. In this study, Thomas and Wei measured the corrosion fatigue crack growth rate (CGR) and da/dN of HY-130 steel in a sodium chloride solution. HY-130 is a high strength steel with 0.4Cr, 4.7Ni, 0.4Mo, 0.7Mn, and 0.2Si. da/dN data were obtained over a range of temperature, cyclic loading frequency, and stress intensity factor amplitude (ΔK) values. Plots of the da/dN as a function of cyclic loading frequency in Figure 7 (ΔK = 30.6 MPa \sqrt{m}) and Figure 8 (ΔK = 20.9 MPa \sqrt{m}) show that at any given temperature, the crack advance per load cycle (da/dN) decreases with increasing cyclic loading frequency because the time-dependent stress corrosion contribution to crack advance becomes smaller as the cycle period decreases. The plots suggest that for a limited range of frequencies, da/dN can be maintained at increasing frequencies by increasing the temperature. This is represented by horizontal lines of constant da/dN across the data sets. Using the data in Figure 7, if a plantrelevant corrosion fatigue environment is 0.05 Hz at 277K (5°C) where da/dN is ~0.45 µm/cycle, then an accelerated test that maintains that same crack advance per cycle for the same ΔK could be conducted at ~1 Hz (20x higher frequency) when using a test temperature of 321K (48°C). A similar accelerating approach is implied by the Thomas and Wei measurements conducted at $\Delta K = 20.9 \text{ MPa}\sqrt{\text{m}}$ as shown in Figure 8, and in particular, the same ~10x accelerating factor appears to exist between the 277K (5°C) and 321K (48°C) data traces.



Figure 7. Corrosion fatigue da/dN as a function of frequency for HY-130 steel obtained at $\Delta K = 30.6 \text{ MPa}\sqrt{m}$ [Thomas 1992].



Figure 8. Corrosion fatigue da/dN as a function of frequency for HY-130 steel obtained at $\Delta K = 20.9 \text{ MPa}\sqrt{m}$ [Thomas 1992].

While the information provided here is insufficient to yield a conclusive assessment about whether viable corrosion fatigue S-N data can be produced following this approach, data from the literature are promising, and the concept is compelling. A key limitation of this approach is that very limited data have been provided to suggest that corrosion fatigue crack initiation data produced at higher cyclic loading frequency and temperature would follow this behavior. And this accelerating approach may not work for all combinations of materials and environments.

The idea of using this approach was considered and explored by Micone [Micone 2016] for corrosion fatigue of a high strength low alloy steel in seawater. The corrosion fatigue life was observed to decrease by performing tests at mildly elevated temperatures while maintaining all other aspects of the environment and testing conditions, showing that there was an acceleration of corrosion processes due to increased temperature.

3.0 Experimental

3.1 Corrosion Fatigue Testing Details

High cycle fatigue (HCF) and high cycle corrosion fatigue (HCCF) life testing was conducted using tensile specimens in a tension-tension loading condition. A round tensile specimen with a nominal 0.150 inch (3.8 mm) gauge diameter was designed based on ASTM E466 guidelines [ASTM 2021]. As shown in Figure 9, this specimen has a 0.5 inch (12.7 mm) long gauge, a fillet radius of 0.933 inch (23.7 mm), and a shoulder diameter of ~0.5 inch (12.7 mm). A surface finish of RA32 (root mean square surface height variability ≤32 µinches or ≤0.8 µm) or better was specified for specimen fabrication, and spot checks were performed to verify surface roughness.



Figure 9. Tensile specimen design used for this project.

The environmental chamber is ~300 mm long, and replicate data for each test condition was obtained by testing three specimens simultaneously in serial loading. When a specimen fails, it is removed, and the test is resumed. The load cell calibration was checked between each test.

The environmental chamber is attached to a continuously recirculating water loop that provides control over dissolved gas content by sparging the desired gas through a water mixing column at a controlled pressure. Dissolved gas content was estimated from mixing column gas pressure and mixing column water temperature using Henry's law. As needed, dissolved gasses are forced to remain in solution in the test chamber by pressurizing the entire test system. The effect of test chamber water pressure on the specimen load (via water pressure applied to the pullrod) was included in the specimen load.

All tests were conducted using a sinusoidal waveform at a frequency of 2 Hz which is ~350x higher than the primary cyclic loading frequency of the Kaplan turbine BACH currently in use at the first USACE candidate location for adopting WFH. Tests were conducted at two

temperatures. One temperature was ~15°C which is the closest readily attainable temperature to the operating temperature of the Kaplan turbines in the Columbia River. This temperature also provides relevance to literature data on HCCF life of MSS that are typically obtained at room temperature. The other temperature was selected to be 90°C in an attempt to accelerate corrosion and corrosion processes to match the increase in mechanical fatigue rate due to higher frequency load cycling.

Since there are no data on the activation energy of stress corrosion of 17-4PH at river-water temperatures, the chloride-induced SCC activation energy of ~73 kJ/mol [Bryan 2016] for 300-series stainless steel was selected as a guide. For this activation energy, a 350x increase in stress corrosion rate occurs for a Δ T of 65°C. This would give a test temperature of 80°C. However, 90°C was selected to further favor an accelerated stress corrosion response.

Tests were conducted in either ASTM D1193 Type IV high purity water or in 1-µm particulatefiltered Columbia River water (CRW). The former represents idealized water conditions while the latter represents a potential off-normal BACH water chemistry. During testing, the water was actively maintained in either a deaerated condition or at 11 wppm dissolved oxygen (DO). The latter represents the DO content of air-saturated water at ~10°C and 1 atmosphere of pressure. Since WFHs are either sealed or have limited contact with outside air, the more representative condition in a hub is deaerated water.

Load cycling was achieved using a servo-electric loading system operating in load control with R = 0.07. Three stress amplitude (S_a) values were selected for each environmental condition except for the 90°C air tests where specimens were not cracking at the highest possible S_a. The desired range of fatigue life cycles to be characterized was 10^4 to $2x10^6$ cycles. The first S_a evaluated for each environmental condition was intended to be in the middle of this range. Subsequent S_a values were selected to meet the target fatigue life range. For a given environmental condition, the first S_a value was based on the measured tensile properties of the material, the fatigue life of 17-4PH found in the literature, and the 17-4PH fatigue life data that had already been obtained during this testing campaign.

3.2 Equipment

Test systems designed for stress corrosion and corrosion fatigue testing in very high temperature water (up to 360°C) [Toloczko 2016] were selected for the 17-4PH corrosion fatigue life testing. These systems have all the necessary features needed to accomplish the work. These include:

- Ability to conduct tests in water at temperatures ranging from 15°C to 90°C+. In order to run tests at 15°C, it was necessary to attach a set of cooling coils to the test chamber and then apply insulation over the cooling coils.
- Ability to control DO content in the water. This is achieved using a recirculating water chemistry control loop that continuously flows test system water through a pressurized water column. Air is bubbled through the water to produce aerated water while an inert gas such as nitrogen is bubbled through the water to produce deaerated water. Henry's Law relates the dissolved gas concentration in the water to the water temperature, column pressure, and gas composition. A side stream of this water is continuously fed through the exposure chamber that is pressurized to prevent the dissolved gas from coming out of solution. Oxygen is kept in solution during elevated temperature testing by maintaining a sufficiently high water pressure in the heated test loop. In the case of 90°C water testing, 100 psi pressure in the elevated temperature portion of the test loop is sufficient to maintain the DO

content evaluated in this study. The pullrod tare pressure due to the test system pressure was factored into the calculated specimen stress.

- The test systems can operate at cyclic loading frequencies up to 3 Hz in tension-tension loading conditions typical of high cycle fatigue testing. Cyclic loading can and was sometimes maintained for >1 month to run specimens to failure.
- These systems have a high degree of wetted component cleanliness, ensuring no undesired water contamination.
- All the wetted components are resistant to corrosion when exposed to Columbia River water.

These systems are also capable of measuring the time to crack initiation during the test. This is not a necessary measurement for corrosion fatigue life testing, but as will be discussed later, insitu measurement of time to crack initiation did reveal useful insight about the corrosion fatigue life behavior.

Because the corrosion fatigue tests were conducted at 2 Hz which is much lower than is typically used for fatigue testing, the time to specimen failure was a relatively long 1000 hours (~1 month) for the lowest stress amplitude testing condition where $\sim 2x10^6$ cycles to failure was desired. In order to obtain sufficient data in a reasonable period of time, three replicate specimens were tested at the same time using a serial loading approach as depicted in Figure 10. Using this approach, three specimens are exposed to the exact same environment and loading conditions. When a specimen fails, load cycling automatically stops, the failed specimen is replaced with a solid rod, and testing is resumed.



Figure 10. Sketch of multi-specimen corrosion fatigue life testing load train.

An example of a set of specimens installed in a test system is provided in Figure 10. Notable items in this photo are polytetrafluorethylene used for pivot joint lubrication and wiring attached to the specimens that is used to detect the formation of a crack.





3.3 Material

A single heat of 17-4PH (Heat number E210833) in the form of 1.5" diameter rod was used for all the tests. The bar material was formed into billets in the United Kingdom and converted to a finished bar in the United States. The composition is provided in Table 1.

		Elemental Composition (wt%)									
Heat/ID	С	Cr	Ni	Cu	Mn	Si	Р	S	Nb	Cb+Ta	Fe
ASTM	≤0.07	15.0-	3.0-	3.0-	≤1.0	≤1.0	≤0.04	≤0.03	0.15-		bal.
A693/A564		17.5	5.0	5.0					0.45		
Specification											
E210833	0.012	15.1	4.6	3.1	0.74	0.40	0.027	0.024		0.262	bal.

Table 1	Composition	of the	17-4PH	used for	this study	/
	Composition		17-4111	u3cu 101	this study	٠ /

The bar material was provided in "Condition A" which is a solution annealed condition. 2.8"-long pieces bar for specimen fabrication were heat treated to the H1075 condition [ASTM 2019]. This length was used to ensure uniform temperature throughout the bar. During the heat treating process, the exterior temperature of the bar pieces was monitored by a contacting thermocouple and used to define the hold times at each heat treatment step.

To evaluate the tensile properties needed to select the upper limit of the maximum stress for corrosion fatigue testing, a single tensile test was conducted at room temperature (24°C) and 90°C. Tensile curves and tensile properties consisting of YS, ultimate tensile strength (UTS), and uniform elongation (UE) are provided in Figures 12 and 13. As expected, only minor differences in tensile properties were observed between the room temperature and 90°C tests. The differences could be due to the difference in test temperature or material variability.



Figure 12. Room temperature tensile test of the 17-4PH used for this research.



Figure 13. 90°C tensile properties of the 17-4PH used for this research.

4.0 Experimental – Water Chemistry Information

As part of the preparation for the experimental work to be conducted under this project, possible water sources for use in WFHs were evaluated. This evaluation includes Columbia River water (CRW) because while the hubs internals will be sealed from the Columbia River, it is expected that over the target 50-year life span of a WFH, some river water leakage into the hub may take place, possibly resulting in a situation where the hub eventually becomes filled with CRW.

4.1 Water Composition

lonic content and pH can have a substantial impact on corrosion and corrosion fatigue behavior, so these parameters are important when considering what water to use in a WFH.

4.1.1 Municipal Tap Water

Municipal tap water could be considered a baseline choice because its potability and use in metal water supply lines suggest that it will have a minimal impact on corrosion and corrosion fatigue behavior. It is also readily available and inexpensive. However, the U.S. Environmental Protection Agency (EPA) "National Secondary Drinking Water Regulation" standards state that municipal tap water aims to contain no more than 250 ppm of chloride (CI⁻) [U.S. EPA 2024]. This is only a recommended limit and can be exceeded. Another important factor affecting corrosivity is pH. The recommended pH range is 6.5 to 8.5 and can be exceeded on either side of the limit. Values outside this pH range can substantially affect the corrosivity of water. Without strict limits, there is a great deal of uncertainty in these two key factors that affect the corrosivity of water. Therefore, using municipal tap water was not considered for this testing.

4.1.2 Controlled Purity Water

ASTM [ASTM 2018] and ISO [ISO 1995] provide standards for various levels of water purity with an aim towards higher purity water. Even the lowest purity standards (ASTM Type IV and ISO Grade 3), shown in Tables 2 and 3 respectively, represent many orders of magnitude higher purity water than what can be reasonably expected from municipal tap water, but just as important is the presence of a standard that ensures the water will meet defined specifications. The ASTM standard is more commonly used in the USA, and 275 quantities of ASTM Type IV cost ~\$900 (\$3.25/gallon). Larger quantities can undoubtedly be purchased at an even lower cost per gallon.

Measurement (unit)	Type I	Type II	Type III	Type IV
Conductivity (µS/cm)	<0.056	<1	<0.25	<5.0
pH at 25°C	N/A	N/A	N/A	5.0 - 8.0
Total Organic Carbon (TOC) ppb or µg/L	<50	<50	<200	N/A
Sodium (ppb or µg/L)	<1	<5	<10	<50
Chloride (ppb or µg/L)	<1	<5	<10	<50
Silica (ppb or µg/L)	<3	<3	<500	N/A

Table 2 Summary of ASTM D1193 high purity water quality standards [ASTM 2018].

Table 3 Summary of ISO 3696 high purity water quality standards [ISO 1995].

Parameter	Grade 1	Grade 2	Grade 3
Conductivity µS/cm (Temperature Corrected)	<0.1	<0.1	<5.0
pH at25°C	N/A	N/A	5.0 - 7.0
Oxidizable matter Oxygen (O2) content mg/L	N/A	<0.08	<0.4
Absorbance at 254 nm and 1 cm optical path length, absorbance units	<0.001	<0.01	N/A
Residue after evaporation on heating at 110°C mg/kg	N/A	<1	<2
Silica (SiO ₂) mg/L	<0.01	<0.02	N/A

PNNL purchased ASTM Type IV water from two vendors for the experimental work and had the water content analyzed. Measured pH for one of the batches of water was found to be ~7.3 (air saturated, room temperature). For both waters, chloride content was below the detectable limit of 0.004 ppb, and sodium was also observed to be below the detectable limit of 35 ppb. The chloride level meets the ASTM Type I specification while the sodium level is significantly below the ASTM Type IV limit. These measurements suggest that high purity water vendors are selling ASTM Type I water as ASTM Type IV. Regardless of the approach to providing ASTM Type IV water, these data show that vendors are adhering to the ASTM standard.

4.1.3 Columbia River Water

The CRW used for the experimental work was sourced from two locations in the Richland, WA area. The first batch was obtained from a dock in north Richland in September 2022. The subsequent batches, obtained between February 2023 and April 2023, were sourced from the PNNL 331 Building where fish studies are performed. All batches were filtered with a 1 μ m particulate filter prior to water quality measurements and use in the test systems. pH was found to be ~8.2 (air saturated, room temperature) for the two batches that were measured. Ion concentrations provided in Table 4 for the first three batches show a chloride content of ~1.5-2 ppm that is approximately two orders of magnitude less than the US municipal tap water limit and is approximately two magnitude greater than the ASTM Type IV water quality limit. Where

other ion comparisons can be made (fluoride, nitrate, sulfate), these batches of CRW have ion concentrations much lower than US municipal water limits. Thus, based on ion content values, CRW is clearly preferable to US municipal water.

Analyte	Analysis Method	Units	EQL†	CRW1 ¹	CRW2 ²	CRW3 ³
Calcium	ICP-OES Vadose-NP	ppm*	0.080	16.4	20.8	19.9
Chloride	Anions by IC-NP	ppm	0.040	1.53	2.16	2.03
Chromium	ICP-OES Vadose-NP	ppb**	9.00	ND	ND	9.25
Cobalt	ICP-OES Vadose-NP	ppb	18.0	ND	ND	ND
Copper	ICP-OES Vadose-NP	ppb	33.0	ND	ND	ND
Fluoride	Anions by IC-NP	ppm	0.030	0.067	0.160	0.150
Iron	ICP-OES Vadose-NP	ppb	30.0	ND	ND	ND
Magnesium	ICP-OES Vadose-NP	ppm	0.024	3.85	5.88	5.86
Manganese	ICP-OES Vadose-NP	ppb	9.00	ND	ND	ND
Molybdenum	ICP-OES Vadose-NP	ppb	45.0	ND	ND	56.2
Nickel	ICP-OES Vadose-NP	ppb	45.0	ND	ND	ND
Nitrate	Anions by IC-NP	ppm	0.080	0.36	1.11	1.15
Phosphate	Anions by IC-NP	ppb	0.160	ND	ND	ND
Phosphorus	ICP-OES Vadose-NP	ppb	252	ND	ND	ND
Potassium	ICP-OES Vadose-NP	ppm	0.079	0.67	0.88	ND
Silicon	ICP-OES Vadose-NP	ppm	0.019	3.76	2.70	2.56
Sodium	ICP-OES Vadose-NP	ppm	0.011	2.30	2.64	2.73
Sulfate	Anions by IC-NP	ppm	0.120	7.52	11.9	11.4
Sulfur	ICP-OES Vadose-NP	ppm	0.041	2.74	4.18	4.21

Table 4	Measured ion concentrations from three batches of water collected from the Columbia
	River in Richland, WA.

†EQL: Estimated Quantitation Limit

¹CRW1: CRW batch taken from north Richland dock.

²CRW2: First batch of CRW taken from the PNNL 331 Building.

³CRW3: Second batch of CRW taken from the PNNL 331 Building. Measurements were performed after using the water for corrosion fatigue testing.

**ppm equivalent unit is mg/L.

*ppb equivalent unit is µg/L.

4.2 Columbia River Water Microbial Analysis

To assess whether MIC could be a concern in a leaking WFH of a Columbia River hydropower system, microbial analysis was performed on Columbia River water samples. To ensure that the water quality represents what flows through John Day Dam, the water samples were collected ~7 miles downstream of John Day Dam at Maryhill State Park on November 3rd, 2022 at 8:00 AM. Two types of analyses were performed at Microbial Insights (<u>https://microbe.com/</u>). The first analysis, Next Generation Sequencing, identifies microorganisms present in the water sample down to the genus level. Such information can be used to assess differences or shifts in the microbial community by location, over time, or in response to changes in process operations and O&M activities. The second analysis, QuantArray, is a hybrid technology

combining the highly parallel detection of DNA microarrays with the accurate and precise quantification of qPCR into a single platform. It identifies the microorganisms by their functional genes involved in MIC and quantifies their corresponding concentration in the water sample. A result summary of the QuantArray analysis is provided in Figure 14. Four functional groups are observed to have a higher concentration than the others: metal oxidizers, sulfur oxidizers, nitrogen fixers, and slime producers. If the growth conditions are right, they can breakdown passivated metal surface and create an anaerobic microenvironment favored by sulfate-reducing microorganisms, leading to or accelerating pitting and crevice corrosion [Zhai 2023]. Since most of these are aerobic microbes, reducing the DO level in the WFH could be considered a measure to control MIC if it is a concern. The complete test result reports of these two analyses can be found in Reference [Zhai 2023].



Figure 14. Microbial population summary on water sample retrieved from Columbia River downstream of John Day Dam in November 2022 to aid in evaluating potential MIC mechanisms. The dots in the same category represents the concentration of different microbe species that belong to the same functional group.

4.3 Amersite 61W Information

The USACE originally requested that tests be conducted using Amersite 65, however it is not available in the USA. Amersite 61W, which is the North America version of Amersite 65 and available in the USA, was used because its key active ingredient is the same as that in Amersite 65 (erythorbate) used by Andritz and chemically related to Amersite 60 (erythorbic acid) used by GE. To better understand its effect and determine the appropriate dosage, PNNL had further discussions with Solenis and surveyed additional literature on sodium erythorbate which is the key ingredient in Amersite 61W. The characteristics of this substance are described in detail in the literature review provided to the USACE [Zhai 2023].

For S-N tests that were conducted using Amersite 61W, DO content was controlled differently than for tests conducted in aerated conditions and deaerated conditions. Amersite 61W is meant to be used in closed water systems with passive or active hardware to limit oxygen ingress. Water used for testing was deaerated prior to adding Amersite 61W. During test system warm-up and during testing, a very low rate of nitrogen was bubbled through the water mixing column while the water column was vented via a 3 mm diameter hole. Because of the low nitrogen bubbling rate, some air is able to diffuse into the space above the water in the water column. This was meant to represent a semi-sealed WFH environment.

5.0 Results

The test matrix is summarized in Table 5. Baseline testing was conducted in air to provide a point of connection to 17-4PH air data. Factors evaluated in the matrix included:

- The effect of water temperature. Most tests were conducted at 90°C as a test accelerant.
 - 16°C for tests in water and 21°C for tests in air
 - 90°C
- The effect of water type. Water composition can affect corrosion fatigue behavior, especially the presence of some anion species such as chloride. T4W is a potential water type to be used in water filled hubs. Anionic chloride content is limited to no more than 0.05 wppm. CRW was evaluated on the scenario where the trunnion seals eventually leak and allow CRW to infiltrate into the hub internals. Anionic chloride content of CRW was found to be <2.5 wppm across multiple water samples. Most tests were conducted using CRW.
 - CRW
 - T4W
- The effect of DO content. DO typically accelerates corrosion and stress corrosion. A WFH is expected to be sealed or semi-sealed which prevents or limits intrusion of hub water containing DO. A WFH will likely have <0.1 ppm DO. However, leaking trunnion seals will allow river water to enter the hub. River water is air saturated meaning that the DO content is in equilibrium with air. 10°C water at 1 atm pressure has 11 ppm DO.
 - Deaerated water with <0.05 wppm DO.
 - Aerated water with ~11 wppm DO.
- The effect of Amersite 61W addition. Some hydropower facilities utilizing WFH employ the use of corrosion inhibitors in the hub water. Amersite 61W is an example product.
 0.25 vol% was used. Details of this and other corrosion inhibitors are provided in the literature review [Zhai 2023].

Test Condition Name	Number of Specimens Tested	Test Temp. (°C)	Water Type	Dissolved Oxygen Content (wppm)	Chloride Content (ppm§)	Water Resistivity (Mohm-cm)
Air	12	21	NA*	NA	NA	NA
90C Air	6	90	NA	NA	NA	NA
ACRW	16	16	CRW	11.6	<2.5	0.02
90C ACRW	11	90	CRW	10.6	<2.5	0.01
90C DCRW	11	90	CRW	<0.05	<2.5	0.01
90C A61W	9	90	CRW+A61W†	NE**	<2.5	NM***
90C AT4W	13	90	ASTM T4	10.6	<0.001	0.67

Table 5. List of test conditions. The completed matrix consists of 78 specimens.

* NA: not applicable

** NE: not estimated

*** NM: not measured

§ ppm here is expressed in mg/l

† 0.25 vol% Amersite 61W added

5.1 Baseline Air and Water Response

As a point of comparison to HCF data in the literature, the response of 17-4PH in air and water is presented in Figure 15 for tests conducted near room temperature and at 90°C. The UTS of the material is provided in this plot to show that the highest S_a was just below the upper limit of acceptable values for HCF testing. Looking first at the RT HCF life, it can be seen that all specimens ran at the highest S_a failed. For all lower S_a values, no specimens failed within the available time limit for testing (>10⁶ cycles). The RT HCF trendline is estimated based on the performance of the 16°C HCCF tests conducted in aerated CRW (ACRW).

In the 10⁶ cycles to failure range, the RT HCF S_a is approximately 3x higher than the ASME BPVC Section VIII Division 2, Annex 3-F HCF curve. The ASME BPVC Section VIII Division 2 fatigue life curve is designed to provide a safe design limit for various moderate- and high-strength boiler steels over a wide range of compositions [ASME 2023]. As discussed in detail in the literature review report [Zhai 2023], the ASME curve has several conservatisms based on alloy composition, alloy strength, and loading conditions. The curve is extremely conservative compared to 17-4PH tested under more realistic conditions. The Sonsino results on cast 17-4PH [Sonsino 1990] are also shown on all the S-N plots because it is considered a useful indicator of HCF life of cast 17-4PH in air and water.

The RT HCF performance of the 17-4PH material is consistent with the available literature data on wrought 17-4PH with similar heat treat conditions and similar loading conditions (Figure 16).



Figure 15. HCF and HCCF life in air and water for tests conducted near room temperature and at 90°C.



Figure 16. 17-4PH air HCF data from the literature [Zhai 2023].

The 16°C HCCF performance of the PNNL-tested 17-4PH is approximately 96% of the room temperature HCF performance. It is difficult to compare this aspect of the PNNL test results to the literature because virtually all literature HCCF data on 17-4PH was obtained in water with 3+ orders of magnitude higher chloride content [Zhai 2023]. For such tests, the ratio of HCCF S_a to HCF S_a near 10⁶ cycles-to-failure ranged from 80-90%. For material roughly similar to 17-4PH, comparisons of HCCF and HCF performance for water purity similar to what were used by PNNL are possible. In particular, 410 stainless steel was found to have an HCCF life only slightly below its HCF life when tested in similar purity water used for the PNNL tests [Zhai 2023].

The air HCF tests conducted at 90°C exhibited much longer fatigue life than at room temperature. Even for the higher S_a , no specimens failed for testing out to 2.3×10^6 cycles. For the lower S_a , one specimen failed at 1.4×10^6 cycles, but the other two specimens went out to 1×10^7 cycles without failure.

The HCCF performance of 17-4PH in 90°C ACRW and 90°C aerated T4W (AT4W) is significantly reduced compared to its 90°C air HCF behavior. As shown in Figure 15, at $2x10^6$ cycles, the HCCF S_a at failure is 64% of the HCF S_a with no failure. The 90°C HCCF data also fall well below the RT HCCF data, indicating a strong effect of test temperature on HCCF life.

There is very little useful data in the literature on testing at 90°C. One example is that Schönbauer conducted HCCF testing 17-4PH in the H1150 condition using tensile specimens at 90°C in air and 10,000 ppm Cl⁻ water [Schönbauer 2015]. For this study, HCCF was accelerated both by the increased test temperature and the high chloride content. The solution was made using NaCl, and during testing at 90°C, the water was actively air saturated,

producing a DO content of 1.9 wppm. Load cycling was conducted at a very high frequency of 20 kHz. At this load cycling frequency, 10⁷ cycles were attained in 8.3 minutes, nominally giving very little time for corrosion to contribute. Although not discussed in Schönbauer's paper, it is very likely that before load cycling began, a specimen would spend at least one hour in the test environment as the system is brought up to temperature and environmental conditions are established. HCF and HCCF life tests for R=0.4 are presented in Figure 17. Even with this incredibly high load cycling frequency, a strong effect of the environment can be seen, but is not possible to isolate the contribution to the corrosion fatigue life from just the water or just the chloride given the limited data.



Figure 17. 17-4PH smooth specimens tested in 90°C air and water with 10,000 ppm Cl⁻ from NaCl. Plots taken from [Schönbauer 2015].

Schönbauer also conducted HCF and HCCF testing on 403/410 SS [Schönbauer 2014] in a manner similar to his research on other materials. Tensile fatigue load cycling was conducted at 20 kHz at various load ratios in air and a solution of NaCl. For these steels, the chloride content was set to only 6 ppm, most likely because of the significantly reduced corrosion resistance of 403/410 SS. The results of the fatigue life tests are presented in Figure 18, showing the response of this material in air and 6 ppm Cl⁻ water for various load ratios. For R=0.05, the HCCF life of this material in a 6 ppm Cl⁻ solution is 85-90% of the HCF life. This result shows a clear effect of the water environment, but it is not nearly as strong as that observed in the PNNL tests.



Figure 18. 403/410 SS life in air and chloride water at 90°C at various R values. Plot taken from [Schönbauer 2014].

5.2 Effect of Water Type, Dissolved Oxygen, and Amersite 61W

The HCCF life results for 90°C tests in ACRW, AT4W, and deaerated CRW (DCRW) are compared in Figure 19. The performance of 17-4PH in ACRW and AT4W are virtually identical. A single runout data point for ACRW at the lowest S_a is the only deviation in performance between the two different waters. Considering that CRW has a low concentration of ions, this is not a surprising result. The performance of 17-4PH in DCRW is identical to ACRW and AT4W at the higher S_a values, but is significantly improved at the lowest S_a value.



Figure 19. 17-4 PH fatigue life in 90°C CRW and T4W.

The effect of Amersite 61W corrosion inhibitor on 90°C HCCF life is presented in Figure 20. Surprisingly, the HCCF life is significantly reduced compared to the other tested water conditions without corrosion inhibitors. Post-test examinations shown in the next section confirm a much higher corrosion fatigue susceptibility of 17-4PH with Amersite 61W added to the water.

Only a single example of corrosion inhibitor evaluation for similar materials and water conditions was found in the literature. This was a study performed by Freitag [Freitag 2019] to evaluate F6NM as a candidate material for WFH. F6NM is the wrought version of CA6NM. Its composition range overlaps with CA6NM in both the target chromium and nickel contents of 13 wt% and 4 wt%, respectively. However, this material has no copper, and its strength is derived from a fine grain size and high dislocation density. In this study, F6NM was exposed at room temperature to air and to water with two different corrosion inhibitors, neither of which are known to be compositionally similar to Amersite 61W. All water tests were conducted with a corrosion inhibitor. Tests were conducted on tensile specimens at 20 Hz with R=0.1, and the water was actively deaerated. The HCCF results, which are reproduced in Figure 20, show that the HCCF performance of F6NM with the Isosynth corrosion inhibitor tends to run slightly below that of the HCF tests. This is consistent with the room temperature PNNL tests where the HCCF life of 17-4PH in ACRW was only slightly less than its HCF life in air (Figure 15). The other corrosion inhibitor produced significantly lower HCCF performance. Without control tests conducted in water with no corrosion inhibitor, it is difficult to make any conclusions about the effect of the two corrosion inhibitors, but since the Isosynth results mirror the PNNL results with ACRW, the Freitag study appears to indicate that the sodium sulfate corrosion inhibitor may also cause reduced performance compared to water without any corrosion inhibitor.



Figure 20. Fatigue life of 17-4PH in aerated CRW with Amersite 61W added.





5.3 Post-Test Examinations

Post-test scanning electron microscopy (SEM) examinations were performed on selected specimens with a focus on identifying whether corrosion and/or metallurgical factors contributed

to fatigue crack initiation of the 17-4PH specimens in the different environments evaluated for this study. Table 6 summarizes the specimens that were examined in detail with their testing conditions. At least two, and usually three specimens from a given test condition were examined to ensure data collected from SEM represent the general behavior of the material under each specific test condition.

Three types of imaging conditions were used during the SEM examination. Secondary electron (SE) imaging, usually performed at high electron accelerating voltage (~20 – 30 keV) to penetrate thin surface deposits and oxides (if applicable), was used to document the topological morphology of the gauge surface or crack surface. Backscattered electron (BSE) imaging, usually performed at low accelerating voltage (~7.5 – 10 keV), was used to examine indications of corrosion buildup on the specimen gauge or crack surface, particularly around the crack initiation site, to help identify whether corrosion had played a role in crack initiation. Low-keV BSE imaging is sensitive to atomic weight of elements; lighter atomic weight elements appear as darker contrast in the images. For this research, the presence of oxygen in any oxide layers will contribute to darker contrast. Finally, energy-dispersive X-ray spectroscopy (EDS) was employed when an area or feature of interest was identified to collect qualitative information on the local elemental chemistry of the specimen. This was often used to identify any inclusions found on a crack surface or to confirm a prominent oxygen presence around an initiation site that was identified by BSE imaging.

Spec ID	Test	Samp	S _{max}	S _{min}	Smean	Cycles to	Time To
	env.					Failure	Failure (h)
FTS027	Air, 21°C	470	1011	71	541	1.8x10 ⁵	25.0
FTS028		470	1011	71	541	1.1x10 ⁵	15.3
FTS001	CRW,	295	634	44	339	8.4x10 ⁶	1169.4
FTS002	90°C	295	634	44	339	2.1x10 ⁶	287.5
FTS004		295	634	44	339	2.1x10 ⁶	297.2
FTS018		420	903	63	483	1.3x10⁵	17.6
FTS019		420	903	63	483	1.3x10⁵	18.6
FTS020		420	903	63	483	1.3x10⁵	18.8
FTS043	CRW+	405	871	61	466	5.7x10 ⁴	8.0
FTS044	A61W,	405	871	61	466	4.3x10 ⁴	6.0
FTS045	90°C	405	871	61	466	5.0x10 ⁴	6.9

Table 6.	Summary	v of the testir	g conditions	s of the s	pecimens	for post-tes	t examinations
		,	9 00				

The post-test examination started with two specimens tested in room-temperature air, FTS027 and FTS028, as control samples to the specimens tested in water environments. The FTS028 was stopped after crack initiation but prior to specimen failure. As shown in Figure 22, FTS028 had one large crack in the gauge section that was more than 2 mm long and normal to the tensile loading direction. Significant deformation bands can be seen around the end of the crack in the image. No other cracks were observed. The two lower images in the same figure show the representative morphology of the gauge surface. Radial grooves and micrometer-size patches of shallow flaking are common features of the surface finish produced by the lathe machining process used to make these specimens.



Figure 22. 30 keV SE montage image of the gauge surface of FTS028 with zoom in images of typical surface morphology found on the gauge of this specimen.

FTS027 fractured before the test system detected the presence of a crack. This provided an opportunity to conduct observations of the crack surface which is complimentary to the gauge surface observations of FTS028. Figure 23 shows the crack surface topology (upper images) and composition map (lower images) of the FTS027 crack surface around the initiation site. The initiation site was determined by tracing the striations to their convergence point on the crack surface. Note that the lower images in Figure 23 show that there are some dark stringers where the crack meets the gauge surface. These features are likely to be surface contaminants deposited near sharp edges on the specimen after the test ended. EDS indicates that the contamination is typically enriched with carbon, calcium, oxygen, and silicon, but the source of the contamination was not identified. This contamination was resistant to removal by sonication and soaking in alcohols. In a few of the other examined specimens, these features covered the initiation site completely.

A closer look at the initiation site revealed that the point of initiation occurred at a particle that intersected the surface of the specimen (Figure 24). EDS performed on this area suggests that the particle is an intermetallic material inclusion enriched in aluminum, calcium, silicon, oxygen, and sulfur (Figure 25). Most particles embedded in the matrix, whether second phase precipitates (formed during alloy cooling or heat treating) or inclusions (present before the alloy solidifies) are brittle and can act as a stress riser due to having a different modulus of elasticity and also if it decoheres from the matrix.



Figure 23. High-keV SE (upper) and low-keV BSE (lower) of the crack surface near the initiation site in FTS027.



Figure 24. Zoom-in BSE image of the region highlighted in orange in the lower right image in Figure 23 for EDS.



Figure 25. Elemental EDS mapping of the region shown in Figure 24 near the initiation site on the crack surface of FTS027.

The next set of specimens examined is FTS001, FTS002, and FTS004 that were tested in 90°C CRW at low stress amplitude. FTS001 and FTS002 failed prior to crack initiation detection, so gauge surface examinations focused on FTS004. As shown in Figure 26, only one large crack was found on the gauge section of FTS004. The rest of the gauge surface shows the typical lathe-machined appearance similar to the air-fatigued FTS028 specimen (Figure 22). There were no indications of pitting on the surface.

When examining the specimen in low-keV BSE mode, an interesting feature was observed near the middle point of the crack mouth. This feature consists of two half-moon-shaped darker patches of dense oxides hovering on both sides of the crack flank, suggesting that it was spherical in shape before it fractured (Figure 27). To better understand whether this feature was associated with crack initiation, FTS004 was broken in two by applying cyclic loading in air, and then one-half of the specimen was examined in SEM in a tilted position where both the crack surface and gauge surface can be viewed. As shown in Figure 28, one half-moon-shaped oxide patch is present at the crack flank on the gauge surface, while cleavage patterns emanate from the same region on the crack surface is indeed the crack initiation site.

To better document the initiation site, the crack surface was imaged in a top-down view in both SE and BSE modes, and an overview is provided in Figure 29. The SE montage image provides a better visualization of the fan-shaped cleavage patterns emanating from the initiation site at the bottom center, while the low-keV BSE montage image revealed a uniformly darker

contrast within ~150 – 200 μm radius from the initiation site. This finding suggests more prominent oxide build-up occurred at the initiation site and was later confirmed by EDS.



Figure 26. High-keV SE montage image of part of the gauge surface (left) and zoom-in images of the gauge surface morphology (right) of FTS004.



Figure 27. Low-kV BSE image showing the half-moon shaped dark oxides found on both sides near the crack center on the gauge surface of FTS027.



Figure 28. BSE image of a tilted view of one failed half of FTS027 where the half-moon shaped dark oxide on the gauge is found to correspond to the crack initiation site on the crack surface.



Figure 29. High-keV SE (upper) and low-keV BSE (lower) montage image of the top-down view of the crack surface on one failed half of FTS027.

As shown in Figure 30, the initiation site on the crack surface contained a smooth, roughly equiaxed-shape surface compared to the appearance surrounding it. This feature is approximately 20 μ m in size. Figure 31 shows that this region is compositionally identical to the matrix material around it, making it difficult to assess why this region exhibited a unique fracture

morphology. Interestingly, several secondary cracks (indicated by arrows) orthogonal to the orientation of the crack surface are intersect this smoother surface and appeared to grow into the specimen. These cracks appear to confirm that this region is uniquely brittle compared to the material around it.

EDS of this region also identified a 2 μ m-size particle rich in Al, Si, and Cl located on the fracture surface approximately 10 μ m away from the gauge surface (circled item in Figure 30 with composition mapping shown in Figure 31). As with the air fatigue tested specimen, this particle may have contributed to crack initiation.



Figure 30. Close-up SE (upper) and BSE (lower) images of the initiation site found on the crack surface of one failed half of FTS027.



Figure 31. EDS elemental mapping of the area around the crack initiation site as shown in Figure 30.

It should be noted that the initiation site morphology of FT004 is not unique. FTS001 tested in the same batch also revealed a similar smooth surface at the initiation site (Figure 32). Indications of more profound oxide presence were also found at this initiation site (Figure 33). It is assumed that this oxide build-up occurred at the initial stage of crack nucleation when the crack grew relatively slowly, allowing more time for the material to interact with the water environment to drive the formation of an oxide layer.



Figure 32. High-keV SE montage image showing the topological morphology around the initiation site on the crack surface of one failed half of FTS001.



Figure 33. Low-keV BSE montage image showing heavier oxides near the initiation site on the crack surface of one failed half of FTS001.

Another set of specimens (FTS018, FTS019, FTS020) tested in the same environment (90°C CRW) but at high stress amplitude was also examined. FTS018 and FTS019 failed during the test. Tenacious contaminants on both crack faces of FTS018 covered most of the initiation site on both halves, so only the examination results of FTS019 are presented. The SE images in Figure 34 show that an angled facet intersecting the gauge surface was found at the initiation site on the crack surface of FTS019. The maximum depth is approximately 30 μ m. These specimens were made to a specified surface roughness of 0.8 μ m, so this feature was likely a small scratch. While the post-test images suggest that this feature acted as the driver for crack initiation, it is worth noting that the specimen had virtually the same time to failure as the other two specimens tested with it. A low-keV BSE image of the same site (the bottom image in Figure 34) revealed beach mark fatigue striations. Unlike in FTS001 and FTS004, no smoother surface was found at the initiation site of the specimens tested at high stress amplitude.

The gauge surface morphology of FTS020, which was stopped before fracture, is presented in Figure 35. No other cracks were found in the gauge surface, suggesting that once crack initiation occurs, the crack propagates across the specimen very rapidly. A very small oxide patch was observed on the gauge surface on one side of the crack mouth (the right side crack flank as shown in Figure 35). This oxide patch is much smaller than the oxide build-up that was correlated to the point of initiation for FTS027. Almost opposite this feature on the left crack flank there is another slightly larger feature with darker contrast. This was found to be a piece of external junk adhered to the flank instead of a corrosion product formed on the surface.



Figure 34. High-keV SE (upper) and low-keV BSE (lower) montage image of the top-down view of the crack surface on one failed half of FTS019.



Figure 35. Low-kV BSE image showing the surface morphology near the crack center on the gauge surface of FTS020.

The last set of specimens examined in detail (FTS043, FTS044, FTS045) were tested at high stress amplitude in 90°C CRW with the addition of the corrosion inhibitor Amersite 61W. Fatigue cycling of FTS043 and FTS045 was stopped before the specimens failed, and thus the characterization focused on documenting the gauge surface morphology of these two specimens. As shown in Figures 36–40, these specimens distinguish themselves from previous specimens by containing multiple cracks on the gauge surface. As can be seen in Figures 36 and 38, besides a large crack that dominated the DCPD response for crack detection, multiple small cracks with a surface length of hundreds of micrometers also formed on the gauge surface, especially in FTS043. High-magnification images are provided in Figures 37 and 39.



Figure 36. BSE montage image of two rotations of FTS043 showing a high density of cracks observed on the gauge surface (highlighted in red besides the largest crack).



Figure 37. Zoom-in BSE and SE images of representative small cracks found in FTS043.



Figure 38. BSE montage image of one rotation of FTS045 showing multiple cracks found on the gauge surface (highlighted in red besides the largest crack).



Figure 39. Higher magnification BSE images of representative cracks found in FTS045.

SEM observations were also performed on the crack surface of FTS044, which failed before load cycling could be stopped. As shown in Figure 40, the SEM-SE image of the crack surface of FTS044 exhibited a different morphology than the specimens tested without the corrosion inhibitor. No beach marks were observed in the SEM-BSE images conducted at several different accelerating voltages. The appearance of the crack surface of the specimen tested with the corrosion inhibitor is more typical of a cleavage morphology while the specimens tested without corrosion inhibitor exhibited features consistent with ductile fatigue.



Figure 40. High-keV SE image (left) and BSE images taken at three different keV levels (right) on the crack initiation site (highlighted by arrows) on the surface of one failed half of FTS044.

6.0 Discussion

6.1 17-4PH Performance in Air

Some points presented in the Results section can be repeated here. First is that the room temperature HCF life of the PNNL-tested 17-4PH is consistent with the available literature data [Zhai 2023], establishing credibility for the testing equipment and approach used by PNNL.

Another point is that at load cycling conditions relevant to BACH in Kaplan turbine systems, 17-4PH performs much better than the BPVC Section VIII Division 2, Annex 3-F HCF curve. There are several reasons for this. A primary reason is that the ASME curve is intended to bound a wide range of steels and associated alloy strength. Another factor is that the ASME curve incorporates mean stress effects as noted in Section 5.5.1.4 of the Section VIII-2 document. This means that this curve is applicable to any R value, and the curve effectively represents conditions where the maximum load cycling stress is always at the UTS, e.g., the stress amplitude limit of 100 MPa at ~4x10⁸ cycles would be applicable to a cyclic loading condition where S_{max} is 892 MPa and S_{min} is 692, which gives an S_a of 100 MPa. In comparison, fatigue testing for the USACE centered around using the same R value of 0.07 for all loading conditions, so an S_a of 100 MPa will be produced by load cycling between S_{max} of 215 MPa and S_{min} of 15 MPa. These are substantially less aggressive stresses on a material/component than represented by the ASME design curve. In practice, no structure would be designed to operate at stresses reaching the UTS.

Another conservatism of the ASME curve is that it covers materials up to a UTS of 892 MPa, whereas 17-4PH in an H1075 condition can be expected to have a room temperature UTS of at least 1000 MPa which is ~12% higher than 892 MPa. Fatigue life generally improves as S_a becomes a smaller fraction of the UTS.

One further point is that the PNNL wrought 17-4PH HCF life results and the available literature data on wrought 17-4PH significantly exceed that of the Sonsino results on cast 17-4PH that have been a benchmark [Sonsino 1990]. Defects and microstructure associated with casting are known to reduce HCF life, so this result is not unexpected, and the PNNL tests help establish that wrought 17-4PH performs substantially better than cast 17-4PH.

6.2 17-4PH Performance in Water

HCCF tests conducted in 16°C ACRW had nearly the same performance as HCF tests conducted in 21°C air. This is a good indicator that the contribution of corrosion and stress corrosion is very low in 16°C ACRW. These results also indicate that some kind of acceleration of the corrosion and stress corrosion response is needed to obtain the most informative HCCF data for tests run at accelerated load cycling frequencies.

Ideally, a relevant assessment of HCCF performance is best accomplished by accelerating the corrosion processes in accordance with the fatigue load cycling frequency increase. Virtually all of the literature studies of HCCF performance of 17-4PH and similar materials have accelerated the corrosion processes by increasing the ionic content in the water, in particular the chloride content [Zhai 2023]. Typically, very high concentrations of NaCl relative to CRW composition were used. Significant reductions in fatigue performance were observed as summarized in Table 7.

Table 7. Ratio of fatigue strength for HCCF life compared to HCF life of relevant martensitic stainless steels.

Material, and Condition	Chloride Concentration (ppm)	Load Cycles	S _{a,HCCF} /S _{a,HCF}	Comments	Reference
Cast 17-4PH, Custom HT	6,000	10 ⁷	0.58		[Sonsino 1990]
17-4PH, H900	21,000	2x10 ⁶	0.83		[Lin 2002]
17-4PH, H1150	21,000	2x10 ⁶	0.88		[Lin 2002]
17-4PH, SA	21,300	3x10⁵	0.82		[McMurtrey 2019]
17-4PH, H1150	10,000	>5x10 ⁶	~0.75	90°C Test	[Schönbauer 2015]
Custom 450, H900	21,000	10 ⁶	0.84		[Lin 2000]
Custom 450, H1150	21,000	10 ⁶	0.79		[Lin 2000]

As described in the work proposal and in the literature review prepared for the USACE, PNNL took an alternative approach to accelerating the corrosion and stress corrosion response by conducting tests at a mildly elevated temperature. Based on available information, the test temperature was raised to a point where the estimated increase in corrosion and stress corrosion contribution would match the increase in the cyclic loading frequency relative to the BACH cyclic loading response. The test results revealed a substantial effect of temperature with the 90°C HCCF S_a at 2x10⁶ cycles in ACRW and AT4W being 68% of the S_a used in the 90°C HCF life results (Figure 15). This reduction in S_a is consistent with, but slightly more than the reduction in S_a reported for concentrated NaCl HCCF tests described in the literature. The 90°C HCF life tests conducted by PNNL revealed that testing in air at 90°C increases HCF performance relative to room temperature HCF life testing, clearly showing that the reduction in HCCF life during testing in 90°C water was due to the water environment and was not due to a change in material behavior due to the elevated temperature.

Four different water environments – ACRW, AT4W, DCRW, and ACRW with Amersite 61W – were evaluated at 90°C. The ACRW and AT4W results were essentially identical, suggesting that there would be minimal effect of BACH HCCF life due to CRW infiltration into a WFH. The DCRW HCCF life was significantly better than ACRW and AT4W at lower S_a values more characteristic of BACH loading conditions, showing the value of operating a WFH with deaerated water. Additions of Amersite 61W were found to substantially reduce HCCF life for all S_a levels.

6.3 Degradation Mechanisms

6.3.1 Hydrogen Embrittlement

The microstructure observations revealed that most of the investigated cracks from the 90°C ACRW specimens initiated at an off-normal microstructural defect intersecting the surface of the specimen. Typically, this feature was some kind of particle, likely an inclusion. This is a common cause of fatigue crack initiation and also can affect fracture toughness in more brittle

materials. Importantly, when specimens were removed from tests, the specimen surface was shiny and without any features suggestive of corrosion, and SEM observations confirmed that there was pitting corrosion on any of the specimens. Thus, pitting did not contribute to HCCF life behavior. Some HCCF studies from the literature also reported no pitting even in high chloride environments [Lin 2002, Schönbauer 2014, McMurtrey 2019].

As was discussed in the literature review, the potential environmentally-induced degradation mechanism for the PNNL tests is likely to be hydrogen induced embrittlement. It was noted that 17-4PH is known to be susceptible to hydrogen embrittlement [Hayes 1982, Yamabe 2022], and some researchers conducting HCCF life studies of 17-4PH have suggested that hydrogen embrittlement is a candidate [Schönbauer 2015, McMurtrey 2019]. This is discussed in greater detail in the literature review.

6.3.2 Loss of Material on Open Surfaces

For 17-4PH, pitting is the operant corrosion mechanism causing loss of material on open surfaces. Pitting can occur in pure water, but many factors greatly accelerate the formation of pits. This includes the presence of ions in the water, MIC, and dissimilar metal contact.

Among the literature studies on HCCF life of 17-4PH or similar materials, chlorides were assumed to be driving degradation, including pitting. For studies that reported post-test specimen observations, some reported pitting [Lin 2002, Ebara 2010] while others assumed that pitting would eventually occur and thus performed some tests utilizing pre-pitted specimens [Schönbauer 2015, McMurtrey 2019].

If WFHs are operated with ASTM Type IV water (≤0.05 ppm Cl⁻), or in worst case CRW (<2.5 ppm Cl⁻), it is worth considering whether chloride-induced pitting may occur on BACH over the lifetime of the hub in these low chloride conditions. Unfortunately, there is no way to reasonably estimate this without looking more closely at all the controlling factors (long term dissolved oxygen content, long term evolution of hub water composition) which have a significant amount of uncertainty.

A summary of the effects of MIC was included in the literature review conducted for the USACE [Zhai 2023], but the information was insufficient to assess whether microbial-induced corrosion could be a significant concern for WFH, and it was not experimentally evaluated. As with chloride-induced pitting, the use of ASTM Type IV water in the hub will greatly reduce the chances of MIC occurring.

PNNL suggests that without further investigation, some level of pitting should be expected to occur either due to chlorides, MIC, or galvanic interactions. When considering pitting as a degradation mechanism, the location of where pits form should also be considered, i.e., only pits forming at high stress areas at the surface of components will be of consequence.

6.3.3 Crevice Behavior

The PNNL and literature results were generated with an emphasis on uniform environmental conditions. In contrast, operational experience across a wide range of industries indicates that crevice corrosion often strongly influences stress corrosion and corrosion fatigue life. For WFHs, crevice corrosion can occur in best-case water conditions, and could be accelerated by chlorides, MIC, and galvanic interactions.

Two possible approaches on how to factor this into WFH BACH life estimates are presented here. One approach is to attempt to prevent crevice corrosion conditions from existing, which allows the current data to be fully applicable. Typically, crevice corrosion is prevented or reduced by building sufficient gaps between parts. One challenge to this approach is that press-fit bushings are used for sliding surfaces, and it is very difficult to prevent tight gaps that allow for crevice chemistry conditions to develop. Crevice corrosion may also be reduced because a WFH is rotating at a significant rate which encourages water mixing throughout the hub internals, and this may limit the degree to which crevice corrosion chemistries can form. Deaerated water conditions will also limit the extent to which crevice corrosion

Another approach is to conduct crevice corrosion testing and to incorporate those results in WFH BACH lifetime estimates. This was discussed as a potential part of the work scope, but was given a lower priority over first obtaining HCCF life data in uniform water chemistry conditions. Towards the end of the project, a low-cost attempt was made to conduct a basic evaluation of crevice corrosion behavior, but the experiment was not successful.

6.4 Application of Test Results

Time to specimen failure during an HCF tensile test is dominated by the time to crack initiation. This is illustrated by showing the response of two of the specimens tested for this project. All specimens tested for the USACE were outfitted for in-situ detection of crack initiation using a direct current potential drop (DCPD) technique that PNNL uses for crack initiation studies. This technique consists of running a constant current through a tensile specimen and then monitoring the voltage across the gauge region of the specimen. When a crack forms and grows, the measured voltage increases. Under well-controlled testing conditions using optimized measuring equipment and techniques, it is possible to detect the formation of a crack that has a depth that is 4-5% of the gauge diameter. The details of this approach are described elsewhere [Toloczko 2021].

An example of typical fatigue specimen response using the DCPD measurement method is provided in Figure 41. In this plot, the gauge voltage is monitored as a function of time, and when crack initiation and growth occur, the gauge voltage increased substantially above the baseline voltage range. For this test conducted in 90°C ACRW at a low S_a , FTS002 and FTS004 initiated just before reaching 300 hours of exposure. For both specimens, the time from the moment of obvious crack initiation to specimen failure (FTS002) or stopping before fracture (FTS004) was no more than 2 hours, which is <1% of the total lifetime of the specimens. Time to initiation typically dominates because after a crack forms, a significant stress riser forms along the crack front that accelerates the fatigue process.



Figure 41. Example of crack initiation and propagation for two fatigue tensile specimens.

With regard to time to crack initiation, the most likely mechanism affecting specimen lifetime in this study is hydrogen embrittlement. However, as was discussed, pitting and crevice corrosion driven by various mechanisms (chlorides, MIC, galvanic effects) may occur during WFH operation. Pitting will only be of concern in locations where high stresses are present on the surface of component because stresses at these locations will be most severely affected by loss of material. Locations of high stresses are often also locations where crevice conditions exist, e.g., at load transfer contact patches between components where tight gaps and high local stresses exist.

PNNL suggests that any follow-on work in HCCF of 17-4PH should consider evaluating crevice condition effects. This could consist of finite element analysis to analyze the change in local stresses based on hypothesized amounts of crevice corrosion. Simple, long term crevice corrosion experiments could be conducted to estimate the degree of crevice corrosion that may occur in WFH BACH.

In engineering structures, there are several factors that affect how much of the life of a component is affected by the time for crack propagation. For example, high stresses may exist near a component surface due to contact with another component, while stresses elsewhere may be much lower, thus causing a crack to advance much more slowly once it moves away from the point of initiation.

Thus, it can be understood that tensile specimen S-N data represent worst-case values for component lifetime estimates because the time needed for a crack to advance to produce failure of a component is not measured. A more complete picture of component life could involve measurement of fatigue and corrosion fatigue crack growth rates which could then be factored into a finite element model to estimate time to a critical loading condition after a crack initiates.

7.0 Summary

HCCF life S-N tests were conducted on wrought 17-4PH in various water environments under accelerated corrosion fatigue conditions. Cyclic loading of tensile specimens was conducted at 2 Hz with an R value of 0.07. The method of corrosion and stress corrosion acceleration was to conduct the tests at a mildly elevated temperature. For tests conducted in aerated CRW and in aerated T4W, at ~10⁶ cycles, the HCCF stress amplitude was ~70% of the value in air. For deaerated water, the value was ~80%. These values are consistent with the limited literature data on HCCF life of 17-4PH in water environments.

The measured fatigue life both in air and water greatly exceed the ASME trend curve for high cycle fatigue life of moderate and high strength steels. Measured values also greatly exceed values found in a hydropower-relevant study in the literature on cast 17-4PH [Sonsino 1990].

The HCCF life of 17-4PH exposed to CRW with added Amersite 61W was evaluated as part of this study and found to cause very low failure times compared to all the other water environments that were used. Amersite 61W is a commercial corrosion inhibitor that is typically marketed for use in boiler systems.

Post-test visual examination of the specimens tested in water revealed no signs of pitting or staining anywhere on the specimens, including at the site of primary crack initiation. SEM examinations also revealed that no pits had formed on any specimens. Specimens tested without Amersite 61W typically only had a single crack. Specimens exposed to Amersite 61W had numerous cracks. For both water and air tests, the crack initiation site was often at a particle, likely an inclusion, intersecting the surface of the specimen.

Hydrogen embrittlement is likely to be the environmental degradation mechanism driving the reduced time to crack initiation and specimen failure during the HCCF tests. This is consistent with conclusions made by several other researchers who have investigated HCCF of 17-4PH and similar materials in water environments.

Other degradation mechanisms, in particular, crevice corrosion, may play a role during a 50+ year life of WFH BACH. The potential effects of crevice corrosion on HCCF life were reviewed as part of a literature study performed for the USACE, and crevice-affected HCCF life testing was a desired addition to the test matrix but could not be included due to project constraints. The primary concern for this degradation mode is high surface stress locations. It is recommended that additional consideration be given to crevice corrosion in defining optimal component corrosion fatigue life estimates. This could consist of finite element analysis to analyze the change in local stress based on hypothesized amounts of material loss due to corrosion along with simple, long term crevice testing.

Since tensile S-N testing is essentially a measure of the time to crack initiation, estimated WFH BACH life estimates may also be more fully informed by conducting corrosion fatigue crack growth rate testing of 17-4PH to assess the time needed for a crack to grow to a critical length.

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