

Performance of Additively Manufactured Alloy 718 In Sour Service

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ABSTRACT

Precipitation hardened nickel alloys are commonly used in oil and gas production equipment where materials must provide both outstanding mechanical strength and corrosion resistance. Additive manufacturing (AM) provides a new approach to the design and manufacture of components from metal powder and provides some unique advantages over traditional manufacturing. However, components produced by AM are not currently acceptable per NACE⁽¹⁾ MR0175/ISO⁽²⁾ 15156 since no AM alloys have been balloted and approved for inclusion. There are uncertainties and knowledge gaps around the performance of AM material in sour service. AM Alloy 718 (UNS N07718) test material was manufactured by laser powder bed fusion (LPBF) with final heat treatment per API⁽³⁾ 6ACRA 150K designation by three suppliers. After benchmarking the basic mechanical properties in air, the AM Alloy 718 test material was evaluated by slow strain rate (SSR) testing and proof ring testing in sour environments similar to those used for the wrought Alloy 718 150K ballot for NACE MR0175/ISO 15156. The resistance to stress corrosion cracking (SCC) and galvanically induced hydrogen stress cracking (GHSC) were investigated and compared to the wrought Alloy 718 150K results.

Key words: Additive Manufacturing, Alloy 718, 150K, UNS N07718, Sour Service.

INTRODUCTION

Alloy UNS N07718 (hereafter abbreviated as 718) is one of the most versatile precipitation-hardened nickel-based corrosion-resistant alloys (CRAs) used for both surface and sub-sea components in oil and gas production service. API 6ACRA¹ provides heat treatment windows and acceptance criteria for 718 in these oil and gas production environments, in which the heat treatment is intended to obtain high strength

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and to minimize the formation of δ -phase at grain boundaries. As pointed out in NACE MR0175 Part 3² (Table 1), field failures of 718 components in sour service are primarily related to stress corrosion cracking (SCC) at elevated temperatures and hydrogen embrittlement in the lower temperature range. The latter is specifically called galvanically induced hydrogen stress cracking (GHSC or GIHSC), which is typically caused by atomic hydrogen uptake from galvanic corrosion or cathodic protection (CP) when 718 is used with steel components in a seawater environment. CP is normally used to protect steel component from corrosion in subsea environments. Therefore, the qualification of precipitation-hardened nickel-based alloys in sour service is aimed to address these two primary cracking mechanisms, as documented in the recent literature^{3,4} for balloting the 150K designation of wrought 718 in NACE MR0175/ISO 15156 Part 3,² Technical Circular 2 (Table A.32, reproduced in Table 2), with link to the heat treatment in API 6ACRA¹ for the 150K grade through footnote c.

Table 1
Cracking mechanisms for precipitation-hardened nickel-based alloys. (Reprint from NACE MR0175/ISO 15156 Part 3, Table B.1)

Materials groups of Annex A	Potential cracking mechanisms in H ₂ S service ^{ab}			Remarks
	SSC	SCC	GHSC	
Precipitation-hardened nickel base alloys (see Clause A.9)	S	P	P	Some Ni-based alloys in the cold-worked condition and/or aged conditions contain secondary phases and can be susceptible to HSC when galvanically coupled to steel.
^a P indicates primary cracking mechanism.				
^b S indicates secondary, possible, cracking mechanism.				

Table 2
Environmental limits for alloy 718 (UNS N07718) for any oil and gas equipment or component. (Reprint from NACE MR0175/ISO 15156 Part 3, Technical Circular 2, Table A.32)

Individual alloy UNS number	Temperature max °C (°F)	Partial pressure H ₂ S PH ₂ S max kPa (psi)	Chloride conc. max mg/l	pH	Sulfur-resistant?	Remarks
N07718 ^{c)}	205 (401)	3 500 (500)	180 000	See "Remarks" column	NDS*	Any <i>in situ</i> production environment pH is acceptable for $PCO_2 + PH_2S \leq 7\,000$ kPa (1 000 psi).
^{c)} wrought UNS N07718 meeting the composition of API 6A CRA is acceptable to a maximum hardness of 45 HRC, when solution annealed and aged as follows: 1) solution-annealed 1 021 °C to 1 052 °C (1 870 °F to 1 925 °F) for 1 to 2,5 hours at temperature, air cool or faster. 2) age hardened 700 °C to 750 °C (1 292 °F to 1 382 °F), hold for 8 hours minimum, furnace cool to 600 °C to 650 °C (1 112 °F to 1 202 °F) and hold for an additional 8 hours minimum, air cool.						

For fabricating high temperature materials via AM 718 has become a primary focus due to its widespread applications in the past 60 years and excellent weldability in either age hardened or annealed condition.⁵ The development of AM technologies and commercialization over the last decade not only make the powder materials and machinery more affordable but also enable building quality metal parts in short turn-over time. Since AM using powder bed fusion (PBF) involves depositing powder, rapid melting and

solidification in a repeated process throughout the body of the manufactured material, the as-built microstructure will vary significantly from wrought material.⁶ Since alloy 718 is a precipitation hardened nickel-based alloy, solution treatment and aging (STA) is typically used to homogenize the microstructure and obtain the correct microstructure for targeting the desired mechanical properties.⁷ A few studies have been conducted in microstructure and heat treatment of AM 718 recently with efforts to understand the parameterization and performance,⁸⁻¹² which could be different from those in the existing standards for wrought counterparts. NACE MR0175/ISO 15156 standard has not been extended yet to AM materials, and so it was of interest to compare materials produced using the AM process at equivalent specifications to the environment limits prescribed for the conventionally produced 718. Likewise, the API 6ACRA¹ does not have prescriptions for AM, but it does provide requirements for 718 heat treatments in order to obtain certain performance requirements, regarding strength, ductility, and toughness at various conditions. It has been suggested that new prescriptions for post-processing, particularly heat treatment, should be pursued for AM 718 for oil and gas applications.¹³ At present, it is unknown whether the heat treatment for 150K grade alloy 718 in the existing standard is sufficient or relevant to provide material properties for AM material that are consistent with the 150 ksi wrought counterpart. Providing further data to address this question is part of the purpose of this investigation.

To support the decision-making around oil and gas industry consideration of AM 718 for sour service, a joint industry project (JIP) was executed with members from across the oil and gas value chain to evaluate the performance of AM 718 samples with compliant heat treatments for 150K designation in API 6ACRA¹. Both mechanical properties and their resistance to SCC and GHSC in sour service were investigated on AM 718 materials supplied from three different material providers.

EXPERIMENTAL PROCEDURE

Material Manufacturing and Heat Treatment

AM 718 materials were contributed by three JIP members who volunteered to have their samples anonymously characterized and tested for sour service. AM 718 test specimens were machined from near-net shape blanks produced by laser powder bed fusion (LPBF) process. Based on the decided test-matrix, an identical build plate design was constructed and used by providing the same STL and .magics files to all three providers. The build plate is shown in Figure 1, which was based on a four-fold axis of symmetry, allowing production of triplicate specimens plus an additional set of spares in the fourth quadrant. The samples were labelled with their orientations and location on the build plate.

Two orientations of samples with respect to the build direction (z-axis) were included to capture the anisotropic variations of AM materials:

- Horizontal (H): load is perpendicular to z-axis (close to x- or y- direction) and crack/fracture occurs on the plane parallel to z-axis (z-x or z-y plane).
- Vertical (V): load is parallel to z-axis and crack/fracture occurs on the plane normal to z-axis (x-y plane).

After AM process, those sample blanks were removed from build plate by electrical discharge machining (EDM). Stress relief (SR) and/or hot isostatic pressing (HIP) were left to the material providers' individual discretion regarding their separately developed approaches to obtaining optimal material properties. The subsequent STA treatments were also selected by the material providers, so long as they remained within the windows specified for alloy 718 150K designation in the API 6ACRA 1st Edition,¹ Addendum 3, as below:

- Solution Annealing: 1870°F~1925°F (1021°C~1052°C), 1 to 2.5 hours, air cool or faster.
- Age Hardening: 1292°F~1382°F (700°C~750°C) for 8 hours minimum, furnace cool to 1112°F~1202°F (600°C~650°C), and hold for 8 hours minimum, air cool.

According to ASTM F3055¹⁴, the sharable build information from three material providers is listed in Table 3. Due to fact that three distinct post-fabrication processes were used, they are not distinguished among three sample providers to satisfy the condition of anonymous distribution of results to all the JIP members. Thereafter the AM 718 materials are identified as materials A, B, and C, and the post-fabrication process are identified as number 1, 2 and 3 (not related to material A, B and C, specifically).

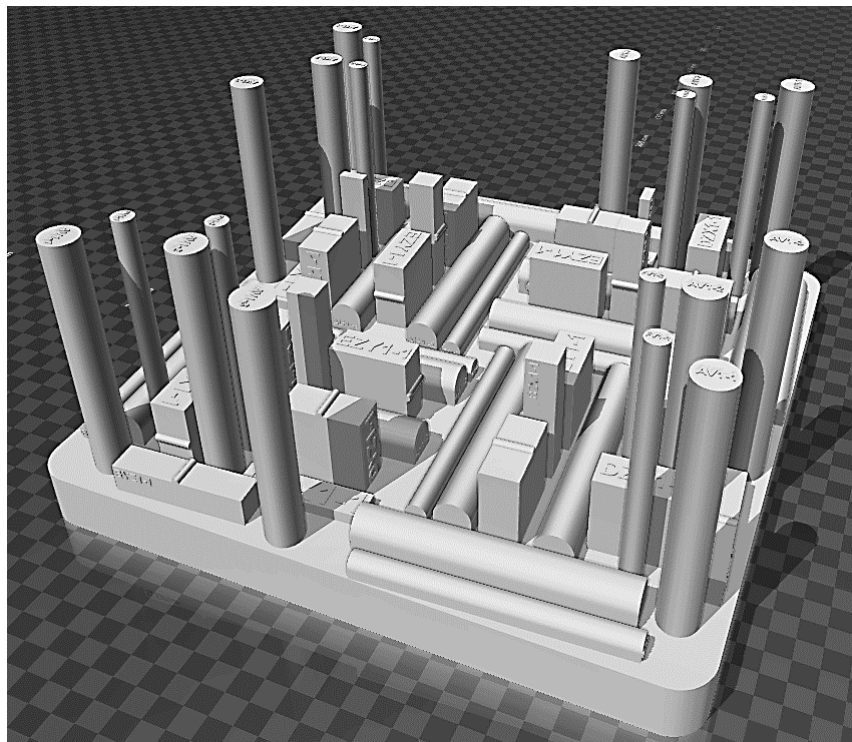


Figure 1: 3D illustration of build platform for test blanks.

Approaches

The characterization of basic mechanical properties provided the baseline of material properties whether it met API 6ACRA¹ 150K designation. The environment test in this work followed the methods to evaluate the performance in sour environments used by Kremel et al.⁴ and Rosenberg et al.³ to ballot 150 ksi (1034 MPa) grade alloy 718 into NACE MR0175/ISO 15156 in 2018. It is key to address each of the primary failure mechanisms listed for precipitation hardened nickel-based alloys in MR0175/ISO 15156 Part 3² Table B.1 (Table 1): SCC and GHSC. Traditional C-ring tests need large specimens, and they can not be accommodated in the current build plate.

1. Galvanically induced hydrogen stress cracking by Uniaxial Tensile “Proof Ring” testing following NACE TM0177¹⁵ Method A in Solution A. This testing will give an indication of the hydrogen embrittlement (HE) resistance of AM 718 vs. wrought counterpart while staying within the standard framework of ballots submitted to NACE MR0175/ISO 15156.
2. Slow strain rate (SSR) tests per NACE TM0198¹⁶ as a screening test using the environmental limits for wrought/cast 718 at 302°F (150°C). SSR testing is an effective screening test, but its value as a qualification test is limited. The literature has shown SSR testing to be conservative for precipitation hardened nickel-based alloys.¹⁷ Static test results will be necessary for balloting AM material into NACE MR0175/ISO 15156.

Basic Mechanical Tests

The basic mechanical properties were evaluated on both horizontal and vertical orientations for each material as summarized in Table 4. ASTM E8¹⁸ was used to characterize the tensile strength in air at room temperature and 400°F (204°C), ASTM E23¹⁹ was used for Charpy impact testing at room temperature, and the fracture toughness at room temperature was measured using ASTM E1820²⁰.

Table 3
AM 718 material build information exchange

AM 718 Material	A	B	C
AM Manufacturing			
Machine Information	SLM Solutions, SLM 280	EOS, M290 SI1924	3D Systems, Prox DMP 320B
Machine Condition	2019	2015	2017
Calibration Information (if qualified)	Valid	valid	valid
Machine Cleaning and History	UNS N07718 only	Cleaned for UNS N07718	Cleaned for UNS N07718
Machine Setting	Customized process	Customized process	Default UNS N07718
Machine/Control Software Version (if automated)	Latest	Latest	Latest
Layer Thickness	50 µm	40 µm	60 µm
Build Chamber Environment	Argon	Argon	Argon
Part Removal Method	EDM	EDM	EDM
Powder Feedstock			
Atomization Process Gas	Argon	Argon	Argon
Size Distribution	15-45 µm	20-45 µm	15-53 µm
Density	4.2 g/cm ³ (Apparent)	4.2 g/cm ³ (Apparent)	3.84 g/cm ³ (Apparent)
Powder Chemistry	API 6ACRA N07718 compliant		
Powder Blends (different suppliers)	Single supplier	Single supplier	Single supplier
Powder Blends (virgin or used)	Used powder	Used powder	Used powder
Post-fabrication process (ASTM F3055 Class*)	1. Class D (SR + HIP + STA) 2. HIP + STA 3. Proprietary process + STA (Note: STA per API 6ACRA N07718 150K, not ASTM F3055)		

* Note: ASTM F3055 Classification for UNS N07718 by full-melt PBF
SR - Stress Relieved, HIP - Hot Isostatically Pressed, STA - Solution Treated and Aged.

Table 4
Test Matrix of mechanical properties for each AM 718 material

	Testing	Standard	Environment	# of orientations	# of replicates per orientation	# of tests
Mechanical Properties	Tensile (R.T.)	ASTM E8	Air	2	3	6
	Tensile (400°F/204°C)	ASTM E8		2	3	6
	Charpy (R.T.)	ASTM E23		2	3	6
	J-R Curve (R.T.)	ASTM E1820		2	2	4

Tensile tests were conducted at room temperature and 400°F (204°C) at strain rate of 0.005/min. Tensile samples had 1.4 inch (35.6 mm) long gauge section with diameter of 0.35 inch (8.9 mm). Both Charpy impact tests and fracture toughness tests were performed at room temperature. Charpy samples had dimensions of 10 mm x 10 mm x 55 mm, and the compact tension (CT) sample for fracture toughness test had length (W) of 1.0 inch (25.4 mm) and thickness (B) of 0.5 inch (12.7 mm).

Proof Ring Test

According to GHSC qualification test NACE TM0177 Method A¹⁵, proof-ring tests were set up to apply 90% of actual yield stress on the tensile sample at room temperature with 100% H₂S purging. Tensile specimens had 1.0 in (25.4 mm) long gauge section with diameter of 0.20 inch (5.1 mm).

Sour test environment for uniaxial tensile (proof ring) tests.

- Room temperature (75±5°F or 24±3°C)
- NACE Solution A: 5% NaCl, 0.5% CH₃COOH, pH 2.7
- 1 atm H₂S flowing through
- 90% AYS
- Test specimen will be galvanically coupled to mild carbon steel coupon
- 3 vertical specimens + 3 horizontal specimen for each material

Slow Strain Rate Test

SSR tests according to NACE TM-0198¹⁶ provide a fast way to assess the sour compatibility of corrosion resistant materials. Since the vertical orientation typically had lower yield and ultimate tensile strength, it was selected for the SSR testing program. The gauge diameter and length were 0.15 inch (3.8 mm) and 1.0 inch (25.4 mm), respectively. Vertical orientation of SSR specimens were both tested in air and in the sour environment at 302°F (150°C).

After the cell was assembled and installed on a load frame, pre-deaerated solution was transferred to fully immerse the gauge section. H₂S and CO₂ were charged to the cell in sequence and reached the target partial pressures after dissolution equilibrium was established. SSR tests started from 75 lbs (334 N) preload (to tighten the load train) and the SSR specimens were pulled to failure at a constant extension rate of 1×10⁻⁶ in/s (equivalent to strain rate of 1×10⁻⁶ /s), which was found to be critical to reveal the susceptibility of 718 in the sour environment.

Sour test environment for SSR tests.

- 302°F (150°C)
- 25 wt% NaCl, unbuffered
- 400 psia (2.76 MPa) H₂S
- 800 psia (5.52 MPa) CO₂
- Sequential gas charging, H₂S and then CO₂
- Nominal strain rate: 1E-6 /s
- Overnight soaking

The broken specimen was removed from the cell within 24 hours of the end of the test. The specimen pieces were rinsed with deionized water and dried with compressed nitrogen. The diameter of the narrowest portion of the necked gauge section was measured with calibrated calipers for calculating the reduction of area. Examination of the specimens after failure consisted of stereographic optical examination at 10-30X magnification and scanning electron microscopy (SEM) for ductile and brittle features.