

Material Capabilities of Additively Manufactured Alloy UNS N07718 in An H₂S-Containing Environment

Wei Chen, Thomas Dobrowolski, Satya Ganti, Tim Haeberle, Aaron Avagliano, Anjani Achanta
Baker Hughes
2001 Rankin Road
Houston, Texas, 77073
USA

ABSTRACT

Additive manufacturing (AM), specifically Laser Powder Bed Fusion (LPBF), brings benefits to the Oil & Gas industry in the areas of better performance, inventory management, and cost reduction. However, selection of AM alloy UNS⁽¹⁾ N07718 (AM 718) face uncertain material capabilities for critical applications such as sour service, when compared to its wrought counterpart specified in API⁽²⁾ 6ACRA (oilfield 718). This study reveals that AM 718 of modified solution treatment combined with standard API 6ACRA age hardening are capable of meeting the yield strength, tensile strength, elongation, and hardness requirements of API 6ACRA, with reduction of area of only 120K condition below the spec limit. AM 718 of modified solution treatment and API 6ACRA 120K age hardening shows resistance to environmentally assisted cracking (EAC) equivalent to oilfield 718 in slow strain rate (SSR) test per NACE TM0198-2020 at 300°F (149°C) in H₂S-containing environment. The performance of AM 718 does not appear to be influenced by the powder conditions investigated. Results of this study provides data-supported clarity to the selection of AM 718 material conditions and production routes for Oil & Gas application and the H₂S-service qualification.

Key words: UNS N07718, Additive Manufacturing, Laser Powder Bed Fusion, Slow Strain Rate Test, Hydrogen Sulfide, Mechanical Properties, Powder, Solution Treatment

⁽¹⁾ Unified Numbering System

⁽²⁾ American Petroleum Institute, 200 Massachusetts Avenue NW, Suite 1100, Washington, DC 20001-5571. USA

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INTRODUCTION

AM brings significant benefits in better performance, inventory management, and lifecycle cost reduction to the Oil & Gas industry. Both manufacturers and users are working towards AM qualification and standardization in order to realize and sustain these benefits. Starting at the product level, the goal is to ensure the product is sound in its form, fit, and function, and free from macroscopic (surface, sub-surface, internal) anomalies deleterious to its performance. Product qualification is supported by a foundational metallurgical or AM material qualification.¹

At the material qualification level, the goal is to satisfy the pre-established mechanical and metallurgical property targets of the material specification. Due to the lack of commonly accepted AM criteria, the industry opts to align with the requirements for wrought counterpart, for example, alloy UNS N07718 in API 6ACRA² (oilfield 718). Data shows AM 718 is capable of meeting the oilfield 718 requirements albeit with larger variations than bar stocks.³ The uncertainties of qualifying AM 718 to be equivalent to oilfield 718 stem from the perceived variability of AM materials and processes.

At a high level, the critical elements of AM (interchangeable with LPBF herein) are feedstock, printing, and post processing. AM distinguishes itself from conventional manufacturing by uniquely integrating the formation of metallurgical properties with near-net shape production. Advances in machine technology, intensive process development, and growing production experience in each of the three critical areas are all contributing to the continued reduction of variability of AM product performance. Rigorous AM process validation, production and quality control are required to meet the performance and productivity benefits.

Feedstock quality is essential to the AM product quality. Powder feedstock, virgin or reused, is an area of active development. It is generally recognized that powder lifecycle management is important to achieve consistent quality and economics of AM. However, the specifics depend on the production experience and practice of individual AM manufacturers. There is urgent need for validation data on how powder affects critical applications, to support the proper selection and control of powder feedstocks.

AM printing can be a highly repeatable process if machine parameters like volumetric energy density (aggregate parameter of power, layer thickness, melt pool width, and speed), feedstock quality, machine calibration and operational control are optimized and properly maintained. Process repeatability is further supported by the advancement of physics-based process simulation and in-process monitoring, as well as standardization and certification across the industries.

Thermal post processing needs more attention. Oilfield 718 differs from aerospace 718 in microstructure, which results from the composition and heat treatment developed specifically to mitigate the risks of environmentally assisted cracking in aggressive environment involving wet H₂S or H₂.¹² Therefore, AM standards such as ASTM⁽³⁾ F3055,⁴ which originated for general and aerospace uses of AM 718, are not entirely aligned with oilfield experience. Another goal of thermal post processing of AM 718 is to eliminate the anisotropy in microstructure and resulting properties, a defining feature of as-built AM materials. A hybrid approach, which combines the ASTM and oilfield (API 6ACRA) practices, may not be optimum for its unwarrantedly long cycle time. It has been shown that the as-built anisotropy in AM 718 can be recovered by a properly optimized solution treatment.¹⁰

This paper will investigate the mechanical capabilities and EAC resistance of AM 718 as a function of powder feedstock condition and thermal post processing. The objective is to provide data-supported clarity to questions facing the selection of AM 718 material conditions and production routes for Oil & Gas application and for H₂S service qualification.

(3) ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. USA

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EXPERIMENTAL PROCEDURE

Material

Oilfield 718 used in this study is a bar stock per API 6ACRA 120K designation with an outside diameter of 5 inch (12.7 cm). Its chemical composition is shown in Table 1. Tensile and slow strain rate (SSR) test blanks were extracted longitudinally from the mid-radius of the bar stock using wire electric discharge machining (EDM).

Individual AM 718 test blanks were produced on a LPBF machine using identical AM parameters including a layer thickness of 60 microns in two build batches. All test specimens have their longitudinal axis parallel to the Z-axis, i.e., the LPBF build direction. As-built AM 718 exhibits anisotropic mechanical properties with lower tensile and yield strengths along Z-axis than those perpendicular to Z-axis. Various levels of anisotropy may persist as function of heat treatment.³

The two AM batches used reused and virgin 718 powders, respectively. Their chemical compositions, measured using powder samples taken from the build plates, are listed in Table 1. All powders originated from the same powder manufacturer with chemistry conforming to oilfield 718 in API 6ACRA.

Table 1
Chemical Composition (wt. %) of 718 Bar and AM 718 Powders

Batch	C	Cr	Ni	Fe	Mo	Nb+Ta	Ti	Al	O
Bar	0.009	18.4	53.5	18.3	2.88	5.01	1.01	0.53	n/a
Reused Powder	0.037	19.0	53.6	Balance	3.05	4.81	0.96	0.48	0.025
Virgin Powder	0.035	19.3	52.7	Balance	3.15	4.88	0.98	0.47	0.022
API 6ACRA	0.045 max	17.0 to 21.0	50.0 to 55.0	Balance	2.80 to 3.30	4.87 to 5.20	0.80 to 1.15	0.40 to 0.60	n/a

Two types of solution treatments and two types of age hardening cycles were applied to the AM 718. See Table 2 for summary of powder and thermal post processing conditions.

Solution treatment (ST):

- Modified ST (Mod) was developed by Baker Hughes specifically for AM 718 to achieve optimum and isotropic material properties in directions parallel and perpendicular to Z-axis.
- Standard ST (STD) is 1920°F (1049°C) for 2 hours followed by rapid inert gas cooling, which is the same as specified for the 120K and 150K designations of alloy UNS N07718 in API 6ACRA.

Age hardening (Age) per API 6ACRA 120K or 150K designation:

- 120K: 1440°F (782°C) for 8 hours, inert gas cooling.
- 150K: 1325°F (718°C) for 8 hours, furnace cooling, 1150°F (621°C) for 8 hours, inert gas cooling.

Table 2
AM Process Variables and Conditions in This Study

AM Process Variable	Conditions
Powder	Reused, Virgin
Solution Treatment	Modified, Standard
Age Hardening	120K, 150K

Metallography

Metallography samples were taken from longitudinal prisms with transverse cross section of 0.394 inch (10 mm) by 0.394 inch (10 mm). The polishing planes are parallel to the Z-axis. They were etched using Kalling's No. 2 according to API 6ACRA.

Mechanical Test

Tension Test

Tension test is per ASTM E8⁵ at room temperature, with gauge length of 1.00 inch (25.4 mm) and gauge diameter of 0.25 inch (6.35 mm). Three tension specimens were tested for each material condition.

Hardness Test

Hardness test is per ASTM E18⁶ Rockwell C-scale (HRC) at room temperature. Specimens are the same group of prisms used for metallography. Three indentations were taken on the side surface parallel to the Z-axis for each specimen. Three specimens were sampled for each material condition.

H₂S (Sour) Test

Slow Strain Rate (SSR) Test

SSR test is per NACE TM0198-2020⁷ with specimen gauge length of 25.4 mm (1.00 inch) and diameter of 3.8 mm (0.15 inch). The test condition is summarized in Table 3. Each material condition includes one test in air and three tests in sour condition. Three material conditions were selected for comparison:

- Bar, API 6ACRA 120K
- AM 718, reused powder, Mod ST, 120K age hardening
- AM 718, virgin powder, Mod ST, 120K age hardening

Table 3
SSR Specimens and Test Conditions (L: Length, D: Diameter)

Specimens per Batch (Condition)	Specimen Reduced Section, mm (inch)	Strain Rate, per sec	Temperature, °C (°F)	Partial Pressure H ₂ S, bar (psi)	Partial Pressure CO ₂ , bar (psi)	NaCl, wt. %
1x (air)	L: 25.4 (1.00) D: 3.8 (0.15)	4 × 10 ⁻⁶	149°C (300°F)	n/a	n/a	n/a
3x (sour)				27.6 bar (400 psi)	55.1 bar (800 psi)	25%