

Influence of the Surface Condition on the Pitting and SCC Resistance of Alloy UNS N07718 Produced via Selective Laser Melting

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ABSTRACT

Additive manufacturing of alloy 718 (UNS N07718) has been largely accepted into many industries and is steadily gaining a foothold in oil and gas applications, primarily in applications involving limited loading conditions in non-corrosive environments. Wrought UNS N07718, a nickel alloy known for its high strength and purpose-built corrosion resistance, has an impressive record of field performance in very demanding oil and gas applications including directional drilling. A broader applicability for additively manufactured UNS N07718 in corrosion applications is currently conditioned by characterization efforts limited to baseline corrosion testing. Additionally, testing has focused on machined surfaces, which are not representative of the surface resulting from additive manufacturing processes. The lack of information regarding corrosion behavior in the as-printed surface condition for this alloy produced using additive manufacturing continues to limit its applicability to the machined surface condition, thus negating benefits gained during the manufacturing process. In this study, the pitting and stress corrosion cracking behavior of additively manufactured UNS N07718 under a variety of surface conditions is investigated. These conditions included machined, as-printed, shot peened, and treated by a micro-machining process. Results from electrochemical and slow strain rate testing in a 2.25 M chloride-containing environment at elevated temperatures are presented and discussed with regards to the influence of surface quality and microstructural peculiarities determined with surface characterization and advanced microscopy on the corrosion resistance of additively manufactured UNS N07718. Experimental results have shown that as-printed surfaces are clearly more susceptible to corrosion and require adequate post-processing surface treatments to restore the expected corrosion resistance of UNS N07718.

Key words: UNS N07718, Additive manufacturing, Selective laser melting, Pitting corrosion, Stress corrosion cracking, Cyclic potentiodynamic polarization, Slow strain rate test

INTRODUCTION

Additive manufacturing (AM) - a layerwise manufacturing technique in which a base material is consolidated using heat or force to produce a solid, three-dimensional object - has been successfully implemented in many industries, not only as a prototyping technology, but as a serious production technology.¹ This has been primarily demonstrated in the aerospace industry, but recent years have shown the slow adoption of AM processes into the oil & gas (O&G) industry. One of the most widely used AM processes, selective laser melting (SLM), utilizes a focused high-energy laser beam to fully melt metal powder particles together in a layerwise manner to result in a near-fully dense metal component based on an existing 3D computer-aided design (CAD) model.² Utilizing SLM, it is possible to process a wide variety of metal alloys, depending on the weldability of the alloy. One of the most studied alloys processed via SLM is precipitation-hardenable nickel (Ni) alloy 718 (UNS N07718), an alloy which has a long-standing tradition of utilization in demanding oilfield applications such as drilling equipment due to its high strength, good ductility, non-magnetic austenitic microstructure, and excellent resistance to environmentally assisted cracking (EAC).³

Research surrounding additively manufactured UNS N07718 (hereafter referred to as AM718) has primarily focused on microstructure and material properties, with a few studies investigating corrosion behavior. The corrosion resistance of AM718 is becoming an increasingly studied topic, and a few studies to date have been published.⁴⁻⁷ Electrochemical results in acidified chloride-containing environments at elevated temperatures have shown that, with comparable surface quality, AM718 behaves similarly to UNS N07718 wrought material in regard to localized corrosion resistance.⁴ Results from slow strain rate (SSR) testing in a 2.25M chloride (Cl)-containing solution at 125 °C also confirmed that, with comparable surface quality, the AM process does not negatively influence the stress corrosion cracking (SCC) resistance of AM718, which was shown to be equivalent to the SCC resistance of UNS N07718 wrought material.⁷ Existing corrosion studies of AM718 have, to the knowledge of the authors, predominantly used a machined surface condition for testing in order to maintain a comparison to UNS N07718 wrought material. AM718 in the as-printed condition, without any surface treatment applied, has a significantly higher surface roughness than a machined surface. In addition, as corrosion results-to-date for AM718 have been conducted on machined surfaces, the results obtained are therefore only valid for AM718 components having the same surface condition. A machined surface, however, requires an additional post-processing step that limits the lead time benefits typically gained from AM-produced components. Thus, in order to avoid this additional post-processing step, the influence of the surface condition on SCC of AM718 must be understood. The research work presented here examines the influence of the as-printed surface condition and various alternative surface treatments on the SCC resistance of AM718 tested in simulated drilling environments.

EXPERIMENTAL PROCEDURE

Materials and Testing Solutions

The investigated AM718 material was selective laser melted in the vertical orientation to the printing plate using a powder bed machine equipped with twin 400 W lasers and subsequently heat treated. The specimens for SCC testing were printed as either round cylindrical bars, approximately 90 mm long with a 15 mm outer diameter, or as near-net-shape sub-size specimens according to ASTM E8.⁸ The chemical composition of the AM718 material was in accord with the requirements for UNS N07718 included in the API¹ 6ACRA⁹ standard. An appropriate solution annealing step was selected to promote full recrystallization and dissolution of artifacts from the printing process. The subsequent age-hardening step was conducted according to the API¹ 6ACRA⁹ standard for the 140K version of UNS N07718.

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The corrosion tests were undertaken in a simulated drilling environment consisting in a 2.25 M chloride (Cl⁻)-containing alkaline solution. While the chloride content in the test solution was set by adding NaCl and KCl, the pH was adjusted to pH 12 at room temperature (23 ± 2 °C) by NaOH. The pH of the test solution is expected to be between 9 and 10 in the range of temperatures between 75 and 125 °C.

Pitting Corrosion Testing

The influence of the surface conditions on the pitting corrosion susceptibility of AM718 was determined by electrochemical methods including open-circuit potential (OCP) measurements and cyclic potentiodynamic polarization tests. These tests were conducted using L-shaped specimens having a (geometrical) exposed surface of approximately 926 mm². Two surface conditions were assessed electrochemically, namely as-printed and machined condition. The surface of the specimens in as-printed condition was not further processed post-printing. Additional L-shaped samples were taken by wire-cut electrical discharge machining (EDM) from AM718 material, which was printed as previously described. To simulate a machined surface, these specimens were treated by grinding consecutively with silicon carbide (SiC) emery papers up to 600 grit.

Prior to the cyclic potentiodynamic polarization test, the OCP of the material in the test solution was recorded during 60 min. The electrochemical tests were conducted using a typical 3-electrode arrangement including a KCl-saturated Ag/AgCl reference electrode, a TiO₂ covered Ti counter electrode (CE), and the L-shaped sample as the working electrode. The polarization scan was started at -100 mV from the OCP and was then increased in the anodic direction at 0.2 mV/s. After reaching a current density of 0.1 mA/cm², the polarization direction was switched into the cathodic direction to evaluate the repassivation behavior of the material. The pitting potential (E_{pit}) was defined as the potential at which the current density of the investigated material reaches 0.1 mA/cm² during the polarization scan in anodic direction. Correspondingly, the repassivation potential (E_{rp}) was defined as the applied potential necessary to reduce the current density below 0.1 mA/cm² during the reverse scan. All the electrochemical tests were conducted in the testing solution previously described at 75 ± 2 °C. The testing temperature was set and controlled by using a thermostat connected with a thermometer inside the electrochemical cell. At least three electrochemical tests were completed for each experimental condition.

Stress Corrosion Cracking Testing

Evaluation of the SCC susceptibility of the AM718 material was done using slow strain rate (SSR) tests similar to NACE TM0198¹⁰, in which a tensile specimen is subjected to a continuously increasing uniaxial tensile stress imposed by a slow and constant extension rate in the simulated drilling environment at elevated temperature. A tensile testing machine with a maximum load of 49 kN with an integrated autoclave unit was used to carry out the SSR tests. The applied load was controlled by a calibrated load cell, and a displacement transducer was used to assess the elongation of the specimen. Five test specimens per surface condition were tested, with two tested in an inert environment to serve as a reference. The inert environment consisted of deionized water, which was de-aerated with high-quality nitrogen prior to testing. The additional three specimens were tested in the 2.25 M Cl⁻-containing solution previously described. All the tests were conducted at 125 ± 5 °C.

Cylindrical specimens for SSR-testing were machined as per the dimensional requirements of NACE TM0198.¹⁰ The near-net-shape specimens were subjected to various surface treatments in the gage length section in order to improve the surface condition typically obtained after the printing process, with one set of specimens reserved for comparison in the as-printed surface condition. The surface treatments included shot peening and a micro-machining treatment (MMP). The MMP treatment is performed with specially-developed “microtools” that are designed to smooth surfaces through a combination of chemical and mechanical processing.¹¹

In preparation for testing, the specimen was loaded into the test frame, the solution was transferred into the autoclave, the test frame was lowered into the autoclave, and the autoclave heated to temperature. Once temperature was reached, a strain rate of $4 \times 10^{-6} \text{ s}^{-1}$ was applied until specimen failure was achieved. Data collection was stopped after the specimen failed, and the autoclave was left to cool. Once an acceptable temperature had been reached, the specimen was removed, cleaned, and its cross-section was measured with calipers. A total of three measurements were taken, and the average was used for further calculations. Following documentation and analysis, specimens were selected for further fractographic and metallurgical examination. SEM was used to analyze fracture surfaces to determine the failure mode, and selected specimens with the appearance of secondary cracking or pitting, were then cut, mounted, and carefully grinded and polished to fully reveal the crack, pit, or other investigated microstructural features. Mounted specimens were then etched so that secondary cracking and/or pitting could be evaluated. In addition, the stress-strain curves were generated from the data to perform the quantitative calculations according to NACE TM0198.¹⁰

RESULTS AND DISCUSSION

Electrochemical Results

Initially the OCP in the 2.25 M Cl⁻-bearing solution at 75 °C of the mechanically prepared samples (machined) was between -400 and -460 mV_{SHE}. The OCP becomes more noble over time, indicating the stabilization of the passive layer. As shown in Figure 1, the specimens in as-printed condition and treated by MMP have shown a more stable OCP over time. Even though the initial values of these two surface conditions were almost 250 mV more positive compared to the freshly prepared specimens, the OCP-values of all three surface conditions after 1 h in the test solution at 75 °C was very similar (-100 ± 40mV_{SHE}).

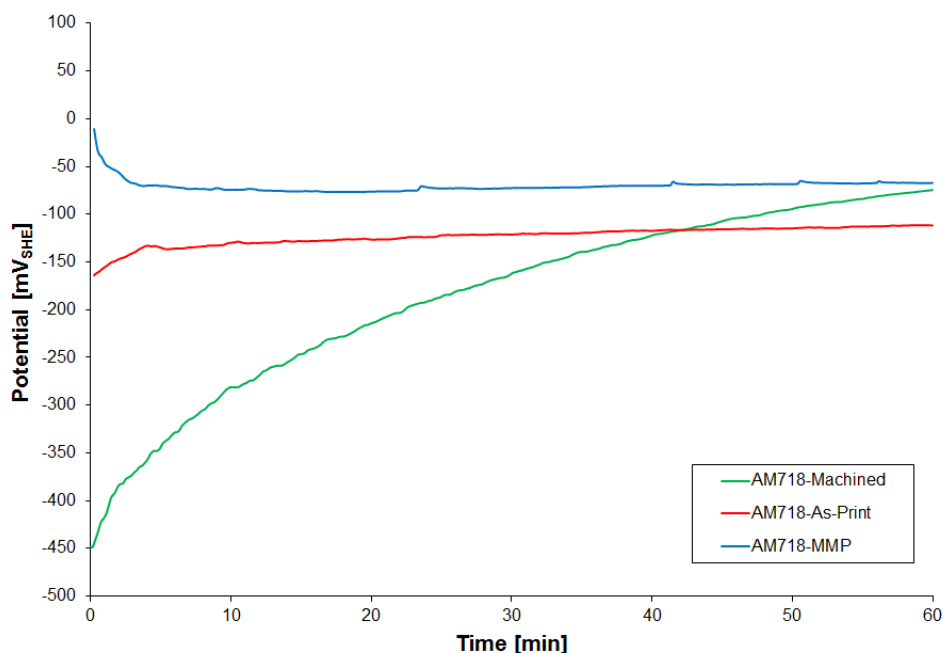


Figure 1: Typical OCP-time records of AM718 with different surface conditions in 2.25 M Cl⁻ solution at 75 °C.

Typical cyclic potentiodynamic polarization curves obtained on the AM718 surfaces in as-printed condition and treated by MMP are included in Figures 2 to 3, respectively. A significant increase in the current density up to 0.04 mA/cm² was reproducibly observed between -50 to +200 mV_{SHE} on the as-

printed specimens (Figure 2). The current density was calculated using the exposed surface (926 mm^2), but the actual surface might be much larger considering the significant roughness of the surface. It is assumed that this loop in the current density with a maxima around $50 \text{ mV}_{\text{SHE}}$ in regards to the stabilization of the passive layer formed on AM718 consisting predominantly in Cr_2O_3 when exposed to the highly alkaline testing solution. After this initial current density increase, the current density drops to values close to 0.003 mA/cm^2 and remains stable. During passivity, however, isolated current transients, which are most likely related to metastable pitting corrosion, could be discerned. These events extended until polarization potentials close to $500 \text{ mV}_{\text{SHE}}$, where the beginning of stable pitting corrosion was determined by the rapid increase of the current density (Figure 2). Once the polarization direction was reversed, the current densities drop after reaching polarization potentials between 365 and $280 \text{ mV}_{\text{SHE}}$ indicating a repassivation of the surface.

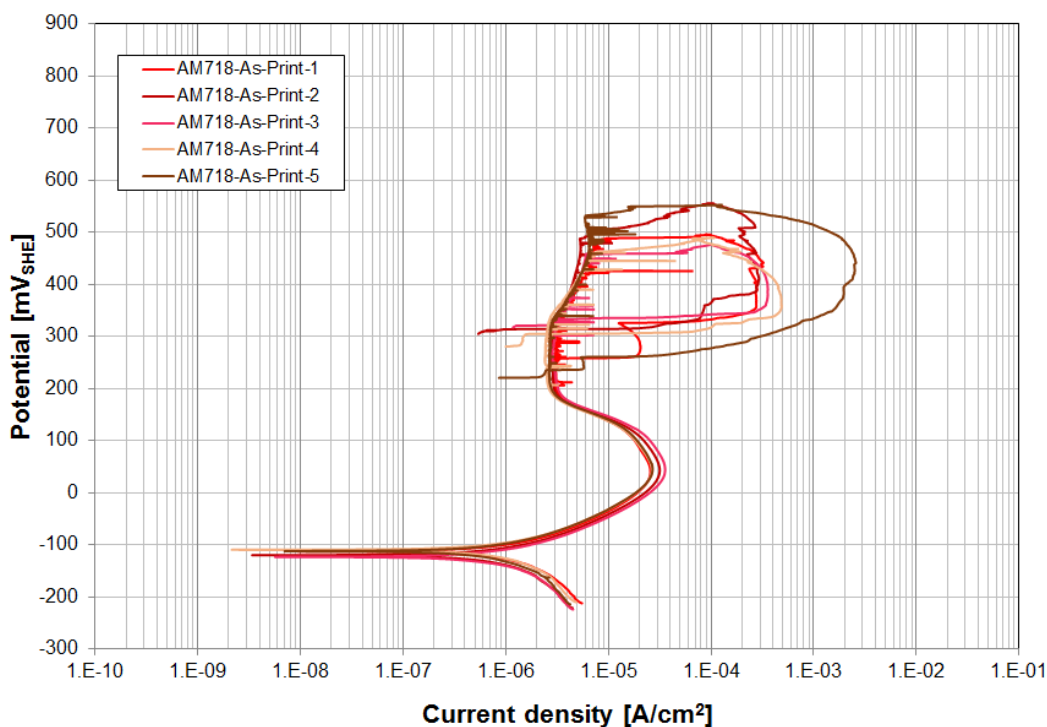


Figure 2: Cyclic polarization curves of AM718 in as-printed surface condition in alkaline 2.25 M Cl-solution at 75°C .