Corrosion Science 78 (2014) 151–161

Contents lists available at ScienceDirect

Corrosion Science

journal homepage: www.elsevier.com/locate/corsci

Influence of surface modifications on pitting corrosion behavior of nickel-base alloy 718. Part 2: Effect of aging treatment

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ARTICLE INFO

Article history:
Received 3 April 2013
Accepted 25 September 2013
Available online 2 October 2013

Keywords:
A. Superalloys
B. TEM
B. XPS
B. EIS
B. Polarization
C. Pitting corrosion

ABSTRACT

A two-step aging treatment is applied to nickel-base alloy 718 that has been previously surface-treated by mill finishing (MF) and machine hammer peening (MHP). As a result, a Cr-enriched oxide layer is formed along with a nano-precipitates layer that consists of high precipitate fractions of γ'/γ'' on the top surface. Surface hardness increases after aging and the compressive residual stress is almost relaxed. The synergistic effects of MHP and aging on pitting corrosion behavior are studied. The results show that the corrosion resistance of the MHP specimens decreases after aging, although the corrosion resistance is still higher than the MF condition.

1. Introduction

In Part 1 of this two-part paper, the influence of surface modifications induced by the MHP surface treatment on pitting corrosion resistance of nickel-base alloy 718 in a 3.5 wt.% NaCl solution has been presented [1]. It has been shown that MHP could improve the pitting corrosion resistance of alloy 718, as indicated by a significant increase in critical pitting potential (+134 mV) accompanied with lower corrosion current density and higher polarization resistance due to surface smoothing, the generation of larger compressive residual stresses in the near surface region, and the formation of nano-grains and nano-twins produced by the MHP surface treatment [1]. It is well known that nickel-base alloy 718 is an age-hardened superalloy that can be heat-treated properly to achieve the optimum microstructure and required mechanical properties [2–13]. Although there have been some reports concerning corrosion behaviors of alloy 718 [2–4,6,14–25], the effect of heat treatment on the corrosion resistance of alloy 718 has not been extensively investigated to date, especially with respect to changes in surface-treated components. Hereby, it is meaningful to study the effect of aging treatment on alloy 718 to determine whether the pitting corrosion resistance of MF and MHP surface-treated alloy can be maintained after heat treatment.

Nickel-base alloy 718 was originally developed for use in aircraft gas turbine engines in aerospace industry, however, its combination of high strength, good corrosion resistance, non-magnetic properties and ability to be heat treated to various strength levels made it a corrosion resistant alloy that has been extensively used by the oil and gas industry for a variety of applications such as wellhead distribution equipments, completion components and drill tools [2–7]. Nevertheless, due to the presence of a significant amount of δ phase and a distribution of γ'/γ'' designed for maximum strength and creep resistance, the aerospace-grade alloy 718 is not desirable for maximum resistance to general and localized corrosion, environmental induced cracking including stress corrosion cracking, sulfide stress cracking, and hydrogen embrittlement as well as corrosion fatigue in extremely corrosive oil wells. Moreover, alloy toughness is extremely important for pressure containment boundary components due to the "leak before burst" failure criteria [4,7]. Thus the redesign of alloy 718 to improve the corrosion resistance and environmental induced cracking resistance while balancing strength and toughness was needed in order to better serve the oil and gas industry.

Oil-grade alloy 718 has been defined by API Specification 6A 718 for nickel-base alloy 718 used in oil and gas drilling and production equipment in terms of composition, microstructure, heat treatment and mechanical properties. The oil-grade alloy 718 is

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http://dx.doi.org/10.1016/j.corsci.2013.09.010

solution-treated at the temperature of 1021–1052 °C for one hour minimum to two and a half hours maximum, followed by single-step aging treatment at 774–802 °C for six to eight hours [2,3,7]. The single-step aging treatment is specified to increase stress corrosion cracking resistance and sulfide stress cracking resistance by sacrificing some strength and hardness in the mechanical properties. As the modern oil and gas industry explored into coastal and marine locations, deeper and deeper wells have been drilled with high pressure high temperature, making the demand for increased strength and toughness super critical for alloys used in high pressure high temperature ultra-deep wells drilling [16–19]. As a result, the two-step aging treatment, i.e., at 760 °C and then 650 °C, which is used to maximize the yield strength of alloy 718 for aerospace components, has been adopted and modified for oil-grade alloy 718 in recent years. However, the aging time for oil-grade heat treatment is decreased to four to five hours for each step as compared to the ten hours for aerospace-grade. It has been reported in our previous research that the corrosion behaviors of alloy 718 in high chloride solution at room temperature did not change in spite of different heat treatment processes, however, the fatigue strength was significantly increased by the two-step aging treatment [6].

In Part 2 of this study, the MF and MHP alloy 718 specimens were aging hardened by the two-step aging treatment. The aim of the present work was to determine how the pitting corrosion behavior of the MF and MHP alloy 718 specimens change due to aging heat treatment in a 3.5 wt.% NaCl solution at room temperature. For the sake of completeness and depth of understanding of the synergic nature of the two processes (surface treatment followed by aging treatment) on pitting corrosion resistance, the results of the second part are compared with those previously presented in Part 1 of the investigations. In this part of the research, surface modification, including near surface microstructure, surface oxide film chemistry, microhardness, and residual stress, have been investigated and are discussed with respect to changes that occur for each due to the two-step aging heat treatment.

2. Experimental

2.1. Materials and specimen preparation

The specimens used in this study were previously surface-treated by MF and MHP, which was introduced in detail in our earlier paper, Part 1 of this study. The MF and MHP specimens were then cut in half. One half of each specimen was double aged at the 760 °C for 4–5 h followed by 650 °C for 4–5 h. Details on the surface modification, heat treatments and specimen designations are found in Table 1.

2.2. Specimen characterization

The near surface microstructure of all the specimens was observed by using optical microscopy (OM, Meiji MT 7000) and scanning electron microscopy (SEM, JEOL JSM-7600F) on prepared cross-sections. The specimens mounted in epoxy were mechanically ground using a series of SiC abrasive papers, progressing from 120 grit through 1200 grit. Final polishing used 3 μm and 1 μm diamond suspension, respectively. After mechanical polishing, the specimens were chemically etched using a mixture of 200 ml methanol, 200 ml hydrochloric acid and 10 g $CuCl_2$ for 4 min. For the samples prepared for transmission electron microscopy (TEM, JEOL JEM-2010) analysis, they were first ground to about 50 μm in thickness before final twin-jet electrolytic polishing. The electrolytic polishing solution consisted of 10% perchloric acid and 90% ethanol. Polishing was done at a current of about 90 mA at −20 °C.

A PHI 5000 VersaProbe system was used for X-ray photoelectron spectroscopy (XPS) analysis, utilizing monochromatic Al Kα radiation to examine the oxide films formed on the surface of specimens after aging heat treatment. The C1s peak from carbon contamination at 284.8 eV was used as a reference to correct for charging shifts. Argon ion bombardment at 2 kV was used for depth profiling the oxide films. The quantification of the species in the oxide films was performed by using XPSpeak 4.1 peak fitting software.

Vickers micro-hardness measurements were made according to ASTM E384-05. A 300 g load (0.3 N force) with 15 s dwell was used to make indentations on cross-sections of the specimens. Ten indentations were placed on each sample with an average value determined.

The surface residual stresses were evaluated by X-ray diffraction (XRD) according to the $\sin^2 \Psi$ method [26] by using X-ray Diffraction (XRD, Bruker D8 Discovery).

2.3. Electrochemical measurements

The electrochemical measurements were made using an aerated 3.5 wt.% NaCl solution, prepared with de-ionised water at room temperature. The electrochemical samples had dimensions 0.5 cm × 0.5 cm. The MF or MHP surfaces were exposed after epoxy mounting. At this point the specimens were rinsed with de-ionised water, dried in air at room temperature and transferred into the test corrosion cell. The specimens were not ground as per ASTM standard practice because it might lead to a possible loss of information on the corrosion behavior difference due to the formation of oxide layers on the mechanically modified surfaces.

Electrochemical tests were performed in a standard three electrode system in a 1.5 L glass cell. A three-electrode cell setup was employed consisting of the specimen as working electrode, a pair of graphite counter electrodes, and a saturated calomel electrode (SCE) serving as the reference electrode. Electrochemical measurements were recorded using a Solartron SI 1287 electrochemical interface and a SI 1260 impedance analyzer. Each test was conducted at least three times and the average of the data was used. At the beginning of each experiment, the working electrode was first cathodically polarized at −1 V_{SCE} for 10 min to remove any air-formed surface oxides and to create a similar passive surface as the starting initial state. After that, the samples were immersed in the test solution to attain a stable value of open-circuit potential (OCP). Potentiodynamic polarization curves were determined using a Solartron SI 1287 electrochemical interface. For this series of tests, the samples were excited from −0.5 V_{SCE} towards the anodic direction at a scanning rate of 0.5 mV/s. Electrochemical impedance spectroscopy measurements were carried out using a Solartron SI 1287 electrochemical interface and a SI 1260 Impedance analyzer. Electrochemical impedance spectroscopy (EIS) measurements were conducted at OCP after 24 h immersion in a 3.5 wt.% NaCl solution. The frequency ranged from 10 kHz to 10 mHz, and the alternating current (AC) amplitudes were 10 mV (rms – root mean square). The impedance spectra were fitted using ZSimpWin software. The morphology of corrosion pits formed after

Table 1
Surface treatment and heat treatment conditions of surface-treated specimens after aging treatment.

Specimen	Surface treatment and heat treatment conditions
MFA	1032 °C/2 h + milling + 760 °C/4–5 h, 650 °C/4–5 h
MHP1A	1032 °C/2 h + milling + machine hammer peening (2 m/min) + 760 °C/4–5 h, 650 °C/4–5 h
MHP2A	1032 °C/2 h + milling + machine hammer peening (4 m/min) + 760 °C/4–5 h, 650 °C/4–5 h