

Hydrogen embrittlement of Oil Patch Alloy 718 and its correlation to the microstructure

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ABSTRACT

Alloy UNS N07718 (known as Alloy 718) is a precipitation hardening nickel alloy containing additions of chromium, niobium, titanium, aluminum and molybdenum. This combination of elements provides an alloy with a combination of high yield strength and corrosion resistance required in sour service applications. Through the precipitation hardening heat treatment, the alloy precipitates the intermetallic Gamma Prime (ordered fcc Ni_3Al) and Gamma Double Prime (bcc tetragonal Ni_3Nb) phases, which are responsible for elevating the yield strength of the material. Additionally to the importance of these both phases in the hardening process, previous studies showed that the microstructure of Alloy 718 may also have a direct influence on its susceptibility to Hydrogen Embrittlement.

Laboratory melts with modified compositions based on Alloy UNS N07718 were produced and tested to correlate both mechanical and hydrogen embrittlement properties. The testing plan included mechanical testing, Slow Strain Rate Tensile (SSRT) tests under cathodic protection and numerical thermodynamic simulations.

Key words: Nickel alloys, Corrosion Mechanisms, UNS N07718, Alloy 718, Hydrogen Embrittlement, Neutron Diffraction, Hardening Phases, Slow Strain Rate Tensile Test, Oil and Gas industry

INTRODUCTION

Its outstanding mechanical performance and its very good corrosion resistance have turned Alloy UNS N07718 into the most preferred applied nickel alloys in the Oil & Gas industry.¹ However, recent field component failures reported the occurrence of hydrogen embrittlement,² which can be a serious limitation to the material application. The continuing development of oil and gas production industry pushes the needs to develop new materials technology for applications involving high temperatures, high pressures and increasingly aggressive service environments.³

The outstanding mechanical features of Alloy UNS N07718 are resultant of the precipitation of the intermetallic phases Gamma Prime (ordered fcc Ni₃Al) and Gamma Double Prime (bcc tetragonal Ni₃Nb), which is allowed by the presence of the alloying elements niobium, aluminum and titanium.

Previous studies show that the strengthening phases Gamma Prime and Gamma Double Prime play an important role on the corrosion resistance of the alloy UNS N07718.⁴⁻¹⁰ Gosheva et al. have made important contributions clarifying the impact of microstructure on the hydrogen embrittlement susceptibility of UNS N07718.⁸ Their studies concluded that the amount of hydrogen stored in the material during cathodic hydrogen charging was predominantly dependent on the strengthening precipitates and their interface with the Gamma matrix. The precipitates act as trapping sites, slowing down the diffusion process. Klapper et al. showed that the amount and size of precipitates such as Gamma Prime and Gamma Double Prime, as well as the Delta phase, rather than the strength or hardness level only, predominantly affected the hydrogen embrittlement susceptibility and defined the type of embrittlement mechanism.⁹ According to his studies, HEDE- (Hydrogen Enhanced Decohesion) and HELP (Hydrogen Enhanced Localized Plasticity)-assisted shear localization may occur on oil-patch N07718 depending on the amount and localization of Delta phase.

Higher volume fractions of Gamma Double Prime precipitates and / or lower volume fractions of Gamma Prime precipitates were reported to have deleterious effects on the hydrogen embrittlement resistance.¹⁰

Even with the variation of the hardening temperatures, as investigated on previous studies,⁴⁻¹⁰ material with significantly differences in the volume fractions of Gamma Prime and Gamma Double Prime precipitates cannot be produced. To reach significant differences in the volume fractions of both hardening phases, adaptations must be made on the chemical composition, since TTT Diagrams available in the literature show that the curves representing the formation of Gamma Prime and

Gamma Double Prime in alloy UNS N07718 overlap.¹¹ In this context, model alloys with chemical-compositions based on Alloy UNS N07718 were developed, with the aim of producing alloys containing as much Gamma Prime as possible and alloys containing as much Gamma Double Prime as possible. The model alloys were then tested on SSRT tests under cathodic polarization in order to assess the hydrogen embrittlement susceptibility, so that it was possible to correlate the effect of each phase separately to the embrittlement phenomena.

EXPERIMENTAL PROCEDURE

Material

Four laboratory heats were melted in a Vacuum-Induction-Melting (VIM) laboratory furnace with nominal chemical composition as shown in **Table 1**, where the intentionally modified elements are underlined and in bold. The API⁽¹⁾ 6ACRA¹² composition limits are shown in this table for reference. The ingots were homogenized and hot rolled to sheets with 15 mm (0.6 inch) thickness, which were then solution annealed and age hardened according to the parameters given in **Table 2**.

The solution annealing and hardening temperatures were defined firstly by thermodynamic simulations and were then experimentally confirmed. Due to the absence of Aluminum, the Heat C required a higher annealing temperature for solution. After solution annealing the microstructures were checked for recrystallization using optical microscopy and all heats were 100% recrystallized. The hardening temperatures were chosen to be those that experimentally resulted in the greatest hardening, given a single-step heat treatment cycle.

Thermodynamic Simulations

The occurring phases expected after submitting the alloys to different hardening temperatures and their fractions were calculated for the different alloy variations using the Materials Property Simulation Package JMatPro⁽²⁾ Version 10.0 from Thermotech,¹³ with set cooling rate of 3 °C/s.

Microstructure

Samples of each laboratory heat were polished and etched with Kallings 2. Microstructural micrographs of the transversal section were obtained by means of Scanning Electron Microscopy (SEM) in order to check precipitation. SEM was operated at 10 kV accelerating voltage and the free working distance was around 5 mm.

⁽¹⁾ American Petroleum Institute (API), Washington, D.C., USA

⁽²⁾ Trade name. Java-based Materials Properties.

Table 1
Nominal chemical composition of laboratory heats in wt.-%

Element	Heat A	Heat B	Heat C	Heat D	API 6ACRA composition limits
C	0.015	0.014	0.014	0.014	0.045 max
S	0.0032	0.0032	0.003	0.0025	0.010 max
Cr	18.49	18.54	18.53	18.55	17.0 to 21.0
Ni	53.6	54.1	53.69	53.77	50.0 to 55.0
Mn	0.01	0.01	0.01	0.01	0.35 max
Si	0.03	0.02	0.03	0.02	0.35 max
Mo	3.05	2.95	3.05	3.02	2.80 to 3.30
Ti	0.95	0.9	0.94	0.93	0.80 to 1.15
Nb + Ta	4.89	0.02	5.2	3.55	4.87 to 5.20
Cu	0.01	0.01	0.01	0.01	0.23 max
Fe (R)	18.37	22.86	18.53	19.5	Balance
P	0.003	0.003	0.003	0.003	0.010 max
Al	0.51	0.51	0.01	0.54	0.40 to 0.60
Mg	0.001	0.001	0.001	0.001	0.0060 max
Ca	0.001	0.001	0.001	0.001	0.0030 max
Co	0.01	0.01	0.01	0.01	1.00 max
B	0.005	0.004	0.005	0.004	0.0060 max

Table 2
Heat treatment parameters of laboratory heats

Heat	Solution annealing			Age hardening		
	Temp [°C (°F)]	Time [h]	Cooling media	Temp [°C (°F)]	Time [h]	Cooling media
A	1032 (1890)	2	Water	760 (1400)	7	Air
B	1032 (1890)	2	Water	770 (1418)	7	Air
C	1045 (1913)	2	Water	770 (1418)	7	Air
D	1032 (1890)	2	Water	750 (1382)	7	Air

Mechanical Properties

Tensile, Charpy impact and hardness tests were carried out in all model alloys to check for mechanical properties.

Tensile test was conducted at room temperature according to ASTM³ E8¹⁴ on round tensile M10 samples on the transverse direction to the rolling direction.

Charpy specimens with notch in the longitudinal direction were tested at a temperature of -60 °C (-75 °F) according to ASTM E23¹⁵.

Rockwell hardness (HRC) readings were performed according to DIN EN ISO 46508¹⁶ in three different positions in the half-thickness of the material.

Hydrogen Embrittlement Susceptibility Test

In order to evaluate the hydrogen embrittlement susceptibility of the laboratory heats, SSRT tests were carried out with test specimens complying with NACE⁽⁵⁾ TM0198-2011,¹⁷ having a gauge section diameter of 3.81 mm (0.15-in) and length of 25.4 mm (1-in). For each laboratory heat, one specimen was tested in a control environment, which consisted of distilled water purged with nitrogen, and three specimens were tested in an aggressive environment, which consisted of 0.5 M sulfuric acid solution with applied cathodic current density of 5 mA·cm⁻². Water was selected as the control environment, due to its inertness and the better temperature control in comparison to other media such as glycerol and air. No reaction was expected between the distilled water and the material surface. Both solutions were maintained at the temperature of 40°C (104°F) during the tests and the specimens were submitted to a strain rate of 1x10⁻⁶ s⁻¹ (crosshead speed 2.5x10⁻⁵ mm·s⁻¹). Elongation-to-failure, plastic elongation, time-to-failure and tensile-strain were reported, as well as the ratios of these values between aggressive and control environments.

A set threshold of 45% for elongation ratio between inert/aggressive environments is used to qualify Alloy UNS N07718 material as acceptable or not acceptable resistance against hydrogen embrittlement.¹⁸ Elongation ratios greater than 45% are considered acceptable and the higher this value, the less the susceptibility of a material to hydrogen embrittlement.

⁽³⁾ American Society for Testing and Materials (ASTM), West Conshohocken, Pennsylvania, USA

⁽⁴⁾ International Organization for Standardization (ISO), Geneva, Switzerland

⁽⁵⁾ National Association of Corrosion Engineers (NACE) International, 15835 Park Ten Place, Houston, TX 77084

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