

Optimization of Hydrogen Stress Cracking Resistance of High Strength Precipitation Hardened Nickel Alloys

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ABSTRACT

The precipitation hardened nickel alloys are designed for Oil & Gas applications requiring high mechanical strength and toughness combined with high corrosion resistance in sour environments. Over recent years there has been increasing industry demand to improve quality control and categorise the various PH Nickel alloy grades resistance to Hydrogen Stress Cracking (HSC) for critical High Pressure-High Temperature environments. This is a complex corrosion mechanism with many factors including composition, strength, microstructure and grain boundary cleanliness^{1,2}. Evaluation efforts have used multiple techniques to measure the effects of HSC resistance with this paper concentrating on the Slow Strain Rate Test (SSRT) according to TM0198 Appendix C and the quality control of API6ACRA.

The purpose of the paper is to present results using the TM0198 slow strain rate test method in a hydrogen charging environment and show the Hydrogen Stress Cracking resistance of the UNS grades N09945, N09946, N07718 and N07725 in relation to mechanical properties and microstructural characterization of mill heats. Process control of the composition and processing has shown the HSC resistance of N09946 can be optimized with the plastic elongation ratio values in the range of 0.7 to 0.4 dependent on the grade and heat treatment. The processing of N07725 manufactured according to API6ACRA has shown the materials elongation ratio as a measure of resistance to HSC can be significantly improved from values of approximately 0.16 to ratio's above 0.45. The SSRT results are compared with mechanical properties determined according to API6ACRA and detailed microstructural analysis.

Key words: Precipitation hardened nickel alloys, Hydrogen stress cracking, Slow strain rate testing

INTRODUCTION

The Precipitation Hardenable (PH) Nickel alloys N09925, N07718, N09945, N09946 and N07725 are widely used for critical downhole oil field applications such as high strength tubing hangers and completion equipment. The materials are particularly useful in High Pressure/High Temperature wells where high strength and corrosion resistance are required in H₂S containing production fluids. Over the last 20 years a limited number of field failure investigations in PH Nickel alloys have been related to the presence of sufficient amounts of intergranular precipitates promoting hydrogen embrittlement, which results in brittle cracking of UNS alloys N07718^{3,4} and alloy N07725⁵ and N07716⁶. The investigations on these failures of Nickel PH alloy 718 have studied the potential for grain boundary precipitates to impair the materials resistance to hydrogen stress cracking by causing brittle fracture by decohesion at the particle/ matrix interface. Cassagne et al⁴ suggested that hydrogen embrittlement is promoted by any inter-granular second phase precipitate irrespective of chemical composition. Liu et al in a separate study on alloy UNS N07718 also showed local transgranular cleavages were initiated from the delta phase/ matrix interfaces in the presence of pre-charged hydrogen⁶. Liu et al⁷ in a separate study on alloy 718 also showed local transgranular cleavages were initiated from the delta phase/ matrix interfaces in the presence of pre-charged hydrogen⁽⁶⁾ Mannan et al⁸ has also shown that the presence of significant amounts of any second phase lowers time –to- failure, % elongation and reduction of area ratios for high temperature sour environment SSRT tests. In this investigation Slow Strain Rate Testing (SSRT) on plain specimens at ambient temperatures in a Hydrogen charging environment have been used to show the effects of hydrogen on a range of high strength PH Nickel alloys mechanical properties.

This work is designed to show the performance of various grades of PH Nickel alloys including N07718, N09945, N09946 and N07725 and their performance in the SSRT hydrogen embrittlement test in comparison with the results reported in a Joint Industry Program (JIP) comparing the performance of Nickel alloy bar grades from a number sources^{9,10}. The materials reported were manufactured to the API6CRA(1) standard with 0.2% proof yield strengths in the range 120 -150ksi (827MPa – 1034Mpa) minimum . The JIP program ranked the various materials for Hydrogen Stress Cracking (HSC) resistance and found the higher molybdenum containing grades of N07716 and N07725 showed low ductility in the CP test environment. There were also lower reported ductility's reported for the high strength grades of N07718-150ksi and N09946. The lower strength UNS grades N09925, N09945 N07718-120ksi had higher plastic elongation in the test environment and plastic elongation ratios Er_{CP}/Er_{inert} . The highest performing Nickel alloy grade in the test program was N09945 with an Er_{CP}/Er_{inert} value of approximately 0.74 at 130ksi 0.2% proof strength

Table 1 shows the nominal compositions of the PH Nickel alloys studied in this investigation. The general alloying principle for the PH Nickel alloys uses high levels of Nickel, Chromium and Copper for sour corrosion resistance, Chromium, Molybdenum and Niobium for Chloride localized corrosion resistance and Nb combined with a controlled combination of Titanium and Aluminium to provide the age hardened strengthening mechanism. The heat treatments used to solution anneal and age harden the various grades are shown in Table 2. The N07718-120ksi grade has a single stage aging treatment while all the other PH grades use a double stage aging treatment to optimize strength and toughness while minimizing the formation of deleterious grain boundary precipitates.

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Table 1. Chemical compositions of PH Nickel alloys in weight percentage.

Alloy	Fe	Ni	Cr	Mo	Cu	Nb	Ti	Al
N09925	22min	42- 46	19.5-22.5	2.5 - 3.5	1.5- 3.0	0.08 - 0.5	1.9 - 2.4	0.1 - 0.5
N07718	Bal	50- 55	17- 21	2.8 - 3.3	0.23max	4.87 - 5.2	0.8- 1.15	0.4 - 0.6
N09945	Bal	46.5- 48	19.5- 23	3.0 - 4.0	1.5- 3.0	2.8 - 3.5	0.5 - 2.5	0.01 - 0.7
N09946	Bal	52- 55	19.5- 23	3.0 - 4.0	1.5- 3.0	2.8 - 3.5	0.5 - 2.5	0.01 - 0.7
N07725	Bal	50- 55	19- 22.5	7.0 - 9.5	-	2.75 - 4.0	1 - 1.7	0.35max

Table 2. Heat treatment parameters for solution annealing and age hardening the PH Nickel alloys.

UNS grade	Material designation	Solution Annealing		Age Hardening Temperature
		Temperature	Time hrs	
N09925	110K	1825°F-1925°F	0.5 - 4	1325°F - 1400°F for 4-9hrs, FC to 1125 - 1275°F hold 12hrs min.
		996°C-1052°C		718°C - 760°C for 4 - 9 hrs, FC to 607 - 660°C hold for 12hrs min.
N07718	120K	1870°F-1925°F	1 - 2.5	1425°F – 1475°F for 6 - 8 hrs
		1021°C-1052°C		774°C - 802°C for 6 - 8 hrs
N09945	125K	1800°F-1950°F	0.4 - 4	1250°F - 1350°F for 4 - 9 hrs, FC to 1110°F - 1190°F hold 12hrs min
		982°C - 1066°C		677°C - 732°C for 4-9hrs, FC to 599°C - 643°C hold 12hrs min
N09946	150K	1800°F-1925°F	0.5 - 4	1250°F - 1350°F for 4 - 9 hrs, FC to 1110°F - 1190°F hold 12hrs min
		982°C - 1052°C		677°C - 732°C for 4-9hrs, FC to 599°C - 643°C hold 12hrs min
N07725	120K	1875°F-1950°F	0.5 - 4	1325°F - 1425°F for 4 - 9 hrs, FC to 1125°F - 1275°F hold 12hrs min
		1024°C-1052°C		718°C – 774°C for 4 - 9 hrs, FC to 607°C - 691°C hold 12 hrs min

EXPERIMENTAL PROCEDURE

The experimental methods for the slow strain rate testing in a hydrogen charging environment according to TM0198 'Slow Strain Rate Test (SSRT) method for screening CRA's for SCC in sour service' are described as follows:

Plain Slow Strain Rate Test Method

The slow strain rate test (SSRT) used sub-size specimens 0.115 in. (3.8mm) gauge diameter with a 1" (25.4mm) gauge length machined from samples taken in the longitudinal direction from the mid-radius bar or mid tube wall position. The samples are polished with a SiC 1200 grit paper rinsed in demineralized water, degreased in acetone, dried and inspected for any defects. The exposed surface is used to calculate the applied current of 5 mA/cm². The tests were carried out at 40°C in an inert environment and

under CP allowing hydrogen charging into the slowly strained specimen. The CP was performed in 0.5M Sulphuric Acid and a current density of 5mA/cm². Prior to testing the solution is de-aerated in a separate vessel using Ar-10% H₂ gas, and introduced in the cell. The CP is applied as soon as the solution is in contact with the specimen and no precharging was applied. When the environmental conditions have been established, the strain rate of 10⁻⁶ s⁻¹ is applied to the specimen. The plastic strain to failure is measured from the stress-strain curve and the ratio calculated by comparing with a reference test from the same heat conducted in an inert environment.

RESULTS

Table 3 shows the mechanical properties of the materials selected for evaluation. All the materials were selected from full scale production heats. The samples were examined using light microscopy for evidence of grain boundary precipitation. This study used materials assessed as having clean grain boundaries according to the microstructural photographs provided by API6ACRA.

The alloys N07718 and N09945 were processed using standard mill practices while the N09946 composition was controlled within an optimized composition tolerance band.

The N07725 -1 & 2 heats were processed using a modified composition within the range for alloy N07725 and the heat treatment practice was modified within the normal API6ACRA parameters. The mechanical properties of strength and impact toughness were typical of the N07725 120 – 150ksi grade.

Table 3. Shows the mechanical properties of the PH Nickel alloys investigated in this study

Alloy	Bar Dia	0.2% P.S.	Tensile Strength	Elong.	R of A.	Average Impacts (trans)	Grain Size	Hardness
		ksi	ksi	%	%	ft.lbs	#	HRC
N07718	8	133.9	178.9	28.2	37.5	50.3	3	39
Grade 120K		120 -145	150min	20min	35min	35min	2 or finer	40max
N09945	14	136.4	163.9	33.6	51	72.2	2	38.4
Grade 125K		125 -155	150min	18min	25min	40min	2 or finer	42max
N09946	9.25	161	186	27	50	55.9	3	41.9
Grade 150K		150 -170	170min	18min	25min	35min	2 or finer	46max
N07725-1	6.5	138.9	177.4	34.6	52.9	65	3	39.3
N07725-2	6.5	141.5	179.9	33.2	51	60.8	4.5	39.8
Grade 120K		120 -150	150min	20min	35min	37min	2 or finer	43max