EVALUATION OF THE CORROSION RESISTANCE OF PRECIPITATION HARDENED NICKEL ALLOYS IN AN ENVIRONMENT SIMULATING OPERATIONAL IMPACT

J.S.KARZINA¹, L.GALATA², A.A.LAPECHENKOV³, I.A.GOLUBEV⁴ & E.P.SIDOROVA⁵

^{1,2,3,4}National Technology Initiative Center for Advanced Manufacturing Technologies Based on the Institute of Advanced Manufacturing Technologies of Peter the Great St. Petersburg Polytechnic University Polytechnicheskaya, 29, St.Petersburg, 195251, Russia

⁵National University of Science & Technology (MISIS)

ABSTRACT

To date, corrosion control and prevention measures are an actual problem. The corrosive processes to which oil-production equipment is subject are as one main reason for the failures and damage. An important task for in order to achieve safe operation of oil-producing equipment is material selection and execution. In this paper were studied the resistance to sulfide stress cracking (SSC) of nickel corrosion-resistant precipitation hardened nickel alloys UNS N07718 and UNS N09925 under conditions simulating operation (elevated temperature and pressure, the presence of hydrogen sulfide, chlorides, low pH), their microstructure and hardness. Results showed that both studied alloys have an austenitic structure, corresponding to a given material class, actual values of hardness of the tested alloys are exceeded of technical documentation requirements, samples passed corrosion cracking test and did not break during 720 hours of testing.

KEYWORDS: Sulfide Stress Cracking, Corrosion Environment, Pump Equipment, Precipitation Hardened Nickel Alloys, Oil and Gas Industry

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INTRODUCTION

Due to the depletion of readily available resources, the demand for high-strength, corrosion-resistant materials for bottom column assemblies, completion equipment and other expensive equipment with high reliability, such as nickel alloys. The borehole in real conditions is formed with a complex trajectory, the equipment is subjected to additional high loads under operating conditions [1, 2]. Selection of the correct materials can prevent or reduce the number of contingency situations. Along with nickel alloys, there is interest in the use of light aluminum alloys for the manufacture of drill pipes with improved characteristics [3, 4], but their use is limited due to low strength characteristics. Nickel based alloys are used in harsh aggressive environments containing H₂S, H₂O, O₂, S, acids, where the corrosive effect is aggravated by high pressure, temperature, the presence of a flow of mechanical particles, sea water, etc. [5, 6].

Hydrogen sulfide is one of the most dangerous factors under operating conditions and causes various types of cracking associated with hydrogen embrittlement [7, 8]. Hydrogen is formed as a result of hydrogen sulfide dissociation, adsorbs on the surface and penetrates into a metal. Careful selection of the material under the operating conditions is required in case of presence of hydrogen sulfide [9]. Standard ANSI/NACE MR 0175/ISO 15156 «Materials for use in H₂S-containing environments in oil and gas production» [10], has been developed to

reduce the probability of equipment destruction in hydrogen sulfide-containing environments. This standard establishes general requirements and recommendations for the selection of metal materials. According to the standard ANSI/NACE MR 0175/ISO 15156, precipitation hardened nickel alloys UNS N07718 and UNS N09925 are allowed to operate at a maximum temperature up to 232 °C and at a maximum hydrogen sulphide partial pressure up to 2.7 MPa (400 psi) with any chloride concentrations and pH value are allowed.

Manufacturing technology and carrying out heat treatment can result forming deleterious phases, that could reduce corrosion and corrosion-mechanical resistance [11-13]. Therefore, the specification API 6A718 [14] described the corresponding requirements for chemical composition of steel, the type of heat treatment, structure and strength characteristics.

However, despite these specifications, it was defined that nickel-based alloys suffer from environmentally assisted cracking (EAC) (Fig.1) [15-18], which confirms the need to evaluation the alloys for use under specific operating conditions.

Figure 1: The UNS N07718 Failed Casing Hanger [18].

The purpose of this work is to determine the resistance to sulphide stress corrosion cracking of metals used in the design of pumping equipment under conditions close to operation and to evaluate their microstructure and properties.

EXPERIMENTAL

The object of research are bars of industrial production (Table 1). The chemical composition of the studied materials is shown in Table 2.

Table 1: Test Materials

Fabrication	Material	Blank	Operational conditions	Yield Strength, MPa
Pump shaft	UNS N07718	Bars Ø 22 mm	Pressure up to 430 atm, temperature up to 95 °C	1393
Thrust chamber shaft	UNS N09925			1370

Table 2: Chemical Composition of Studied Materials, %

Material	C	Ni	Cr	Mo	Nb	Ti	Mn	Al	Cu	Si	P	S	Fe
UNS N07718	0.015	54.00	19.50	3.20	5.00	1.00	0.03 5	0.50	0.02	0.09	0.00 1	0.00 15	16.6 4
API 6A718 UNS N07718	≤0.04 5	50-55	17-21	2.8- 3.3	5.02	0.8- 1.15	≤0. 35	0.4- 0.6	≤0. 23	≤0. 35	≤0. 01	≤0. 01	Bal.
UNS N09925	0.010	43.08	20.02	2.79	0.03	2.08	0.48	0.23	1.70	0.07	0.01 2	0.00 2	29.2 0
API 6A718 UNS N09925	≤0.02 5	42-46	19.5- 22.5	2.5- 3.5	≤0. 05	1.9- 2.4	≤1. 0	0.1- 0.5	1.5- 2.5	≤0. 35	≤0. 02	≤0. 003	≥22

Metallography

Metallographic studies were carried using an optical microscope Reichert-Jung MeAF - 3A (×500) at a magnification of ×200 and ×500. Determination of grain size was carried by the circular intercept procedure in according with ASTM E 112, ASTM E 1382. Image analyzer "Thixomet Pro" was used for evaluating the results of determination of grain size.

The surface was prepared using abrasive paper 120-1200 grid and polishing fabrics (from 9 to 1 μm) on sanding-polishing machine BUEHLER ECOMET 4. Samples were subjected to etching of «aqua regia» solution (1 part HCl (38%) and 3 parts HNO₃ (56%)) for a microstructure research.

The inclusions were studied by scanning electron microscopy (SEM) using a microscope Zeiss SUPRA 55 WDS-VP.

Determination of test material hardness

Measurement of hardness was carried by Rockwell's method according to ASTM A370 in the direction perpendicular to the deformation at hardness tester TP 5006 using C scale. Hardness was further measured using microhardness tester MICRO-DUROMAT 4000E. Evaluation of microhardness was carried using microscope Reichert-Jung MeAF - 3A (×500) at a magnification of ×200. Image analyzer "Thixomet Pro" was used for to measure the area of the microhardness indentation. The load was 180 grams, applied for 10 seconds. The number of microhardness was determined on the Vickers scale (HV_{0,18}).

Sulfide stress corrosion cracking

C-Ring samples were made according to NACE TM-0177, method C. Testing was carried out at 90% of actual yield strength. Information of samples loading are shown in Table 3.

Table 3: Loading of Samples

Material	Sample №	d, mm	h, mm	E, N/mm ²	% from yield strength	Yield strength, MPa	Deflection, mm
UNS N07718	1	21,76	1,35	205000	90	1392	1,58
	2	21,76	1,35				1,58
	3	21,76	1,35				1,57
UNS N09925	4	21,76	1,35			1370	1,55
	5	21,76	1,35				1,55
	6	21,76	1,35				1,55

Test conditions: temperature – 175 °C, partial pressure: H₂S – 200 psi, CO₂ – 200 psi, stress levels - 90% of actual yield strength.

The test samples were immersed in an autoclave with a testing solution containing 20% NaCl solution at pH = 3.5. After sealing the autoclave, the solution was deaerated. The test setup is shown in Figure 2.

Figure 2: Schematic Arrangement of Test Equipment for Method C, C-Ring Test.

Then the autoclave was slowly blew down H₂S и CO₂ and heated up to 175⁰C while maintaining the temperature with an accuracy of $\pm 3^0$ C. Testing time is 720 hours.

RESULTS

The structure UNS N07718 is austenite. The structure has a distinguishable carbide heterogeneity in the form of dark clusters - inclusions up to 10 μ m in size mainly along grain boundary (Fig. 3, a), which is not acceptable according to API 6A718. These inclusions are probably titanium, niobium and chromium carbides/nitrides of MC and M₂₃C₆ types (Fig. 3-b, 4) [11]. There are no other deleterious phases in the structure, such as δ - phase, σ - phase, Laves phases. Average grain size is 5,12 μ m, G = 11,93.

Figure 3: The Structure UNS N07718 at Different Magnification: a – $\times 200$, b – $\times 500$.

Figure 4: SEM Images Carbides/Nitrides.

The structure UNS N09925 after quenching is austenite with characteristic twin structure inside austenitic grains (Fig. 5, a). There are no deleterious phases in the structure, such as δ - phase, σ - phase, Laves phases (Fig. 5, b). The structure is acceptable according to API 6A718. Average grain size is 17,3 μm , $G = 8,43$. Duplex grain size is observed. There are grains of 10 μm and 30-40 μm .

Figure 5: The Structure UNS N09925 at Different Magnification: a – $\times 200$, b – $\times 500$.

Thus, both studied alloys have an austenitic structure, corresponding to a given material class, in which twin structure are present. Grain size complies with API 6A718 (According to API 6A718 average grain size shall be No. 2 or finer according to ASTM E 112). However, there is a considerable amount of dispersed particles in samples from UNS N07718, which is not acceptable.

Results of the hardness testing are shown in Table 4.

Table 4: Sample Hardness Values

	UNS N07718		UNS N09925	
	HRC	HV0,18	HRC	HV0,18
Average Hardness Value	47,7	530,5	44,4	483,3
NACE MR0175, API6 A718	$\geq (32-34) \text{ HRC} \leq 40 \text{ HRC}$		$\geq 26 \text{ HRC} \leq 38 \text{ HRC}$	

Hardness of the tested alloys is not consistent with requirements of NACE MR0175, API 6A718, actual values are exceeded of requirements by 16-19%, which may indicate an incorrect heat treatment conditions and may lead to environmentally assisted cracking under operating conditions.

The photographs of the samples after sulfide stress corrosion cracking according to NACE TM0177 tests are shown in Figure 6.

Figure 6: Samples After Sulfide Stress Corrosion Cracking Tests.

All samples passed corrosion cracking test at elevated temperature and pressure and did not break during 720 hours of testing. No cracks were observed on the surface of all samples after testing. However, some samples have evidence of corrosion damage, possibly due to the discontinuity of the chemical composition due to carbides precipitations on the grains. The papers [11, 18], show, that such carbides tend to reduced corrosion resistance of the alloy under conditions with high chloride concentration and temperature.

It is worth noting that the testing conditions for SSC resistance are less severe than level VI of NACE MR0175 that is recommended level for nickel-based alloys testing. Though in papers [19, **Error! Reference source not found.**] alloys UNS N07718 and UNS N09925 passed tests at even more aggressive conditions than level VI: at temperature – 260°C, 300°C and pressure – 500 psi, load at 100% of actual yield strength. The test conditions were selected taking into account aggressiveness of the operating environment of the studied materials. Conceivably, more severe conditions of the experiment will lead to a more destructive effect and possible cracking of studied samples.

Sulfide stress corrosion cracking or EAC resistance of alloys given in the standard does not indicate the resistance of a particular material, as properties are influenced by a particular manufacturing technique. The studies show nonconformance with the requirements of standard that can be connected with technologies and or the lack of control. Accordingly, it is necessary to carefully control the technology, chemical composition and structure of the alloys in order to avoid susceptibility to EAC in the operation expensive equipment requiring high reliability.

CONCLUSIONS

Study of precipitation hardened nickel alloys UNS N07718 and UNS N09925 for resistance to sulfide stress corrosion cracking under conditions simulating operation were carried out. According to the test results, the samples of the alloys UNS N07718 and UNS N09925 showed no susceptibility to SCC at the temperature of 175 C, partial pressure of H₂S - 200 psi, CO₂ - 200 psi, stress level of 90% from the actual yield stress for 720 hours. Some samples have evidence of corrosion damage, possibly due to the presence inclusions (M₂₃C₆) along the boundary, which reduce corrosion resistance of the alloy.

Structure of both tested alloys is austenite. Considerable amount of dispersed particles is present along grain boundaries in the structure of the UNS N07718, that is unacceptable industry according to API 6A718.

The hardness values of the UNS N07718 and UNS N09925 samples is not consistent with requirements of NACE MR0175 and exceeded the values by 16-19%. In order to reduce the probability of cracking, the manufacturer needs to select a manufacturing technique and heat treatment conditions that will provide the required hardness of 32-40 HRC, 26-38 HRC, respectively.

When selecting materials for using in aggressive hydrogen sulfide containing environments, it is necessary to control the production technology, chemical composition and structure of alloys in order to reduce the probability of destruction.

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