

High-Strength Material for Hydrogen Service, and Experimental Investigation

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ABSTRACT

As an energy source, hydrogen has been a topic of discussion for some time. To support future energy demands, efficient means of hydrogen compression will be required. Centrifugal compression may play a larger role in a market generally dominated by reciprocating compressors. However, centrifugal compression is highly dependent on gas molecular weight. Aerodynamic head is developed by increasing the gas velocity to increase kinetic energy levels, then by converting the kinetic energy to potential energy through diffusion. The resulting impact is an increase in static gas pressure. The amount of kinetic energy imparted into the gas is a function of gas velocity and molar mass. To achieve high gas velocities, an increase in impeller rotational speed is required. This imparts higher stress on centrifugal impellers, which can quickly exceed guidelines provided by American Petroleum Institute (API) Standard 617, eighth edition (2014). To utilize the benefits of higher rotational speed, new strength limits for the application of materials in hydrogen gas compression are required. In this work, the relative Hydrogen Induced Cracking (HIC) resistance of several common impeller alloys was compared utilizing National Association of Corrosion Engineers (NACE) standard test method TM0177 as a guideline. The results presented here aimed to determine a test procedure and validate high-strength materials suitable for use in a hydrogen compression environment. The chemistry, microstructure, mechanical properties, and heat treatments were examined to explain the results observed and to seek predictors for high-strength materials in hydrogen service.

INTRODUCTION

Hydrogen is one of the most abundant elements on earth and a grand potential source for clean energy. When consumed in a fuel cell, hydrogen produces only water, electricity and heat. Governments and private sectors have been researching hydrogen energy for years, for a broad range of applications across all sectors. These include, but are not limited to, distributed- or combined-heat-and-power; portable power; auxiliary power for trucks, planes, rail, and ships; and use in passenger vehicles including cars, trucks, and buses.

Most production methods for hydrogen yield a relatively low-pressure stream (20-30 bar) that must be compressed prior to transport. Most compressors used for gaseous hydrogen compression are either positive displacement or centrifugal by design. Centrifugal compressors are a preferred option for transferring hydrogen via pipeline, thanks to their moderate compression ratio and high throughput. Centrifugal compressors achieve pressure rise by adding potential and kinetic energy to a continuous flow of gas through a rotating impeller. The kinetic energy is converted to additional potential energy, in the form of pressure, by the diffuser. The rise in potential energy in the diffuser is, in most cases, similar to the rise in the impeller. The potential pressure is dependent on the kinetic energy created by the impeller's rotational speed.

More widespread use of centrifugal compression to compress hydrogen will likely require the compressors to be manufactured from higher strength materials. Why is this? Because, potential pressure is dependent on the kinetic energy created by the impeller's rotational speed. Hydrogen is known to cause Hydrogen Induced Cracking (HIC), also commonly known as Hydrogen Embrittlement (HE) or Hydrogen Assisted Cracking (HAC), in high stressed materials. HIC is caused by the absorption and diffusion of hydrogen into and throughout the metal lattice, per ASM Handbook Volume 13A (2003). Hydrogen can not only lead to initiation of cracks at high load, but also reduce the Yield Strength (YS) by reducing the required energy to move dislocations. While research has been conducted to determine a metal's susceptibility to HE, it unfortunately is insufficient to provide guidance on fit-for-service envelopes as represented by Carlisle (1959).

According to API 617 eighth edition, the maximum YS for all materials in hydrogen service has been limited to 120 Ksi (827 MPa). Due to material variations, production variables, and process variations, many OEMs will set design strength limits somewhat below 120 Ksi (827 MPa), such as 110 Ksi (758 MPa). This limited strength affects the speed at which a centrifugal compressor can operate.

There has been a precedence on several centrifugal compressors where impellers with YS greater than 120 Ksi (827 MPa) were started up in hydrogen environments and operated trouble free for many years. This successful experience suggests the 120 Ksi (827 MPa) limit is conservative and that it is possible to use higher strength materials in hydrogen centrifugal compressors.

As a hydrogen atom enters the metallic lattice structure of a component, the lattice structure is placed under strain (Figure 1). The strain caused by the hydrogen atom reduces the bonding forces between the metallic atoms (i.e. embrittlement). Hydrogen first absorbs in either its atomic form (H) or as molecular hydrogen (H₂). Hydrogen molecules cannot enter a metal lattice themselves but must first be split into their representative atoms through a catalytic process (e.g., plastic deformation during plate rolling manufacturing, acid pickling, plasma welding, or cathodic protection, among others). In the case of a compression service, this would require a chemical reaction, where hydrogen is cathodically evolved on the surface of a metal part.

The hydrogen atoms can then enter the lattice and diffuse to the base metal grain boundaries. From there the atoms can accumulate at

the metal grain boundaries, exerting pressure on the metal grains and reducing fracture ductility, strength, and endurance limits, ultimately leading to fatigue cracks. The HIC of steels, therefore, is a consequence of the absorption of these hydrogen atoms.

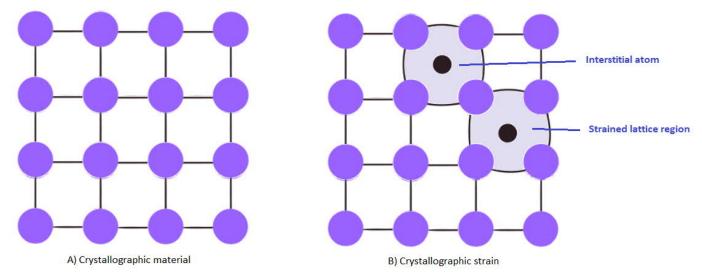


Figure 1. Figure 1A (left) depicts a crystallographic material in which metal atoms position themselves in a repetitive threedimensional pattern in which each atom is bonded to its nearest neighboring atom. Figure 1B (right) depicts a crystallographic strain in which strain is caused when an interstitial atom is crowded into the metallic crystalline lattice, which introduces distortions and strain in the surrounding lattice.

In a centrifugal compressor application with greater than or equal to 90 percent molecular weight, or 100 psi (6.9 bar) partial pressure of hydrogen, it is unlikely that chemical reactions will occur to split the hydrogen molecules. This is because these machines are generally clean with little to no contaminants. This provides a potential path to higher material strength limits for hydrogen environments per API 617 eighth edition.

A commonly accepted standard is a Slow Strain Rate Test (SSRT). As a screening tool, SSRT uses cathodic polarization to evaluate a material's susceptibility to HIC. The SSRT will invariably produce fractures either by HE, mechanical failure, or both. Therein lies the problem: the industry needs a test that simulates the conditions in a compressor that is suitable to qualify materials fit for hydrogen service. The work presented here aimed to determine a test procedure and validate high-strength materials suitable for use in a hydrogen compression environment. This work expands on the SSRT testing of high-strength material in hydrogen environments per Kagey (2018), Li (2019), Botinha (2019), and Morana (2019).

TEST METHOD

The testing method used to simulate the compressor environment for hydrogen applications employed a modified NACE TM0177 and used a proof ring specimen based on research perform by Trautmann (2019). The proof ring specimen is the name given by NACE TM0177 for the offset tensile bar with machined O-ring grooves (Figure 2). The test environment was created using a representative gas composition, which was established from this OEM's extensive compressor experience. Testing was conducted in triplicate. Statistical analysis was conducted on a material represented by a minimum of three separately processed material heats.

Figure 2. Proof ring specimens for hydrogen tests

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Materials

Three different materials were selected for testing and provided by two separate suppliers. The three materials have increasing chemical resistance to SSC due to their chemical composition per Table 1. The first material, ASTM A 182 Gr. F6NM (X3CrNiMo13-4, DIN 1.4313), commercially known as F6NM, is a martensitic stainless steel (Figure 3) with 13 percent chromium and 4 percent nickel that has a Body-Centered Cubic (BCC) lattice structure (Figure 6). Its corrosion resistance is similar to UNS S41000, commonly known as 410 stainless steel, but the addition of nickel and molybdenum improves its resistance to general corrosion and chloride attack.

The second material was ASTM A 705, Type XM-12 (X5CrNiCuNb15-5, DIN 1.4545), commercially known as 15-5. This is a Precipitation Hardening (PH) stainless steel (Figure 4) with 15 percent chromium and 5 percent nickel with a BCC lattice structure. In most environments, the corrosion resistance of 15-5 PH stainless steel is between that of 300 series and 400 series stainless steels, while in many chemical applications it favors the more resistant 300 series corrosion characteristics. 15-5 is very similar to ASTM A 705, Type 630 (X5CrNiCuNb16-4), commercially known as 17-4, with minor differences in chromium and nickel. Both grades are considered equivalent per NACE MR0175 (ISO 15156).

The third material was industry standard AMS 5663 (DIN 2.4668), commonly known as 718. This is a precipitation hardening nickelbase alloy that has an austenitic microstructure (Figure 5) and a Face-Centered Cubic (FCC) lattice structure (Figure 6). The alloy is highly resistant to SCC and pitting in environments that contain chlorides and hydrogen sulfide.

All materials had mechanical properties exceeding the limits per API 617 eighth edition. See Table 2 for yield strengths.

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Alloy	Heat No.	С	Si	Mn	Р	S	Cr	Mo	Ni	Cu	Nb	Ν	Fe	Та
F6NM ⁽¹⁾	720539	0.025	0.38	0.68	0.016	0.0020	12.68	0.45	4.42			0.021	81.12	
15-5	374468	0.018	0.08	0.38	0.015	0.0012	15.20	0.04	4.35	3.17	0.25			< 0.01
	420925	0.03	0.05	0.03	0.007	0.0010	18.51	3.05	52.9	0.03	5.32		18.39	< 0.01
Alloy 718 ⁽²⁾	C80604	0.023	0.07	0.09	0.008	0.0003	17.60	2.95	54.0	0.06	5.08	0.005	18.18	< 0.02
	D81841	0.025	0.06	0.07	0.009	0.0004	17.91	2.94	53.7	0.05	5.23	0.004	18.13	< 0.02

Table 1. Chemical composition (in weight percent) of the tested specimens

(1) Further elements: C+N = 0.046

(2) Further elements: A1 = 0.60, Pb = 0.0002, Co = 0.10, B = 0.004, Se < 0.0003, Bi < 0.00003, Nb+Ta = 0.252

			1		5		
Alloy	Min. YS	Impact Test	Hardness		Tensile Test		
		KV2		YS	UTS	Е	RA
	ksi (MPa)	J @ RT	HRC	ksi (MPa)	ksi (MPa)	%	%
F6NM	131 (900)	140	33.0	137 (946)	145 (1002)	16	59
15-5	145 (1000)	206	34.3	158 (1090)	162 (1120)	15.5	67
718	160 (1100)	27	48.5	184 (1272)	211 (1460)	21.6	43
(\mathbf{a})	11 • .•	1 0 1	.1	1	1 1 0	.1 *	

Table 2. Overview of the mechanical properties for the alloys tested⁽³⁾

(3) Abbreviations can be found in the nomenclature section at the end of this paper.

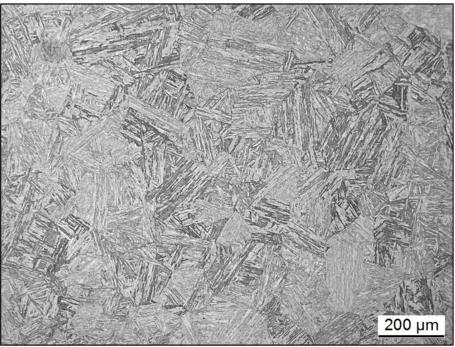


Figure 3. Microstructure of the testing material for F6NM. The microstructure is tempered martensite.

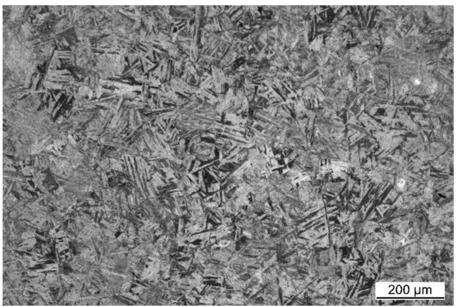


Figure 4. Microstructure of the testing material for 15-5. The microstructure is tempered martensite.

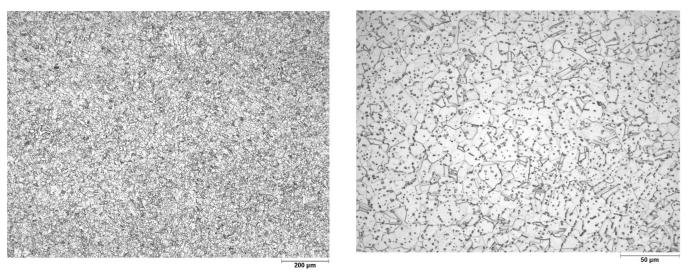


Figure 5. Microstructure of the testing material for Alloy 718 taken at 100X and 500X magnification. The material is a fine grain austenitic microstructure with δ phase distribution and MC carbides.

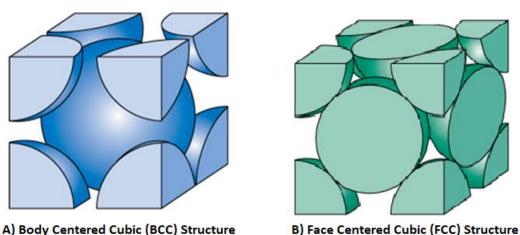


Figure 6. Graphical representation of crystal lattice structures. Images courtesy e-Education Institute by Penn State University

Figure 6A depicts a Body-Centered Cubic (BCC) structure with a total of nine lattice points—eight lattice points at the corners plus one lattice point in the center. Figure 6B depicts a Face-Centered Cubic (FCC) structures with a total of 14 lattice points—eight lattice points at the corners plus six lattice points, one for each face.

Experimental set-up

To investigate the influence of hydrogen on the susceptibility to HIC, tests were carried out under a compressed hydrogen atmosphere in heated test pressure vessels (Figure 7). These vessels are designed for pressures up to 1450 psi (100 bar) and temperatures up to 392°F (200°C). The first series of tests were conducted at a hydrogen pressure of 145 psi (10 bar), which is above the 100 psi (6.9 bar) limit per API 617 eighth edition.

In the general test arrangement, three specimens per material were tested at an Applied Yield Strength (AYS) in the test pressure vessels. The specimens were loaded to 95 percent of the YS for F6NM and 15-5 and 90 percent of the YS for Alloy 718. The AYS was the maximum stress achievable due to limits of the test rigs. Multiple specimens of each material were prepared, and the surfaces were mechanically polished. The tests had a minimum duration in the test pressure vessel of 30 days (720 hours) or 90 days (2,160 hours) without interruptions. These test durations are in line with the industry-accepted test duration that establishes suitable environmental limits for Sulfide Stress Cracking (SSC) and Stress Corrosion Cracking (SCC) per NACE TM0177.

The 720-hour duration is considered acceptable to reveal failure in materials susceptible to HIC by Raymond (1988). This is in part due to the nature of both SCC and HIC to cause quick failures in service. Therefore, 720 hours was considered the acceptable test duration for HIC for these tests. Much like developing a Nelson Curve that is used to prevent high temperature hydrogen attack, 90-day testing was conducted to exhibit a material's ability to endure an infinite number of hours without cracking. Infinite number of hours is only based on assumed conditions and assuming that other factors are not changing.

Following hydrogen gas exposure, all specimens were inspected for evidence of cracking. The proof ring specimen success criteria included no visible cracks at 10X magnification after the 30-day (720 hours) and 90-day (2,160 hours) test durations. Upon completing the visual inspection, specimens were then Non-Destructively Tested (NDT) using liquid dye penetrant. Specimens showing cracks after testing were further investigated as to the nature and cause of the cracks.



Figure 7. Heated test pressure vessels (autoclaves)

Static Tensile Load test procedure

The testing sequence for all tests started with purging the test vessels with inert gas, applying hydrogen gas, holding for more than 12 hours, and then applying static tensile load at room temperature. In the first investigations, tests were carried out in clean molecular hydrogen (known as environment 1). The objective of these tests was to determine if hydrogen-induced cracks can generally occur when the limits from API 617 eighth edition are exceeded, and further to determine if there are differences in the materials used under these conditions.

With the information gained from the first test results designed to represent pressures and simulate clean compression environments, the next phase was to move from molecular hydrogen to atomic hydrogen. To accomplish such, acids were introduced to create corrosion at the metal surface to generate atomic hydrogen in a chemical reaction (known as environment 2). For that reason, the pressure vessels were loaded with carbon dioxide in an aqueous solution that included acetic acid until a pH of 3.0 was achieved. The specimen was immersed in the solution.

FINDINGS

The results indicate a strong dependence of HIC susceptibility that correlated with either microstructure, crystal lattice structure, or both. In other testing performed by the OEM, similar dependencies were found which showed how stress corrosion cracking (SSC) susceptibility correlated with retained austenite. Previous work by the authors Botinha (2019) and Li (2019) indicated higher strength materials with a specific microstructure were resistant to HIC.

The modified NACE TM0177 testing yielded HIC that will allow for identifying fit-for-service environmental envelopes. An overview of the results is given in Table 3. The results and findings of this work are discussed below.

Alloy	Environment 1 (env 1):	Environment 2 (env 2):			
	Initial hydrogen atmosphere (H ₂)	Corrosive Hydrogen Cell Simulating Production Environment			
F6NM	- Three specimens tested and all three passed after 720 hours.	 One specimen from env 1 tested and passed after 720 hours, specimen was tested with an additional 720 hours and passed. One specimen from env 1 tested and cracked after 305 hours. One specimen from env 1 tested and passed after 720 hours. 			
15-5	- Two specimens tested, and both passed after 720 hours.	- Two specimens from env 1 tested and failed after 35 and 144 hours.			
718	- Three specimens tested and all three passed after 720 hours.	 Three specimens from heat 1 (i.e., env1) tested and passed after 2,160 hours. Three specimens from heat 2 tested and passed after 2,160 hours. Three specimens from heat 3 testing and passed after 2,160 hours. 			

Testing in initial hydrogen atmosphere – Environment 1

Initial testing exposure under high stress in pure hydrogen (H_2) atmosphere did not lead to any HIC. Both F6NM and 15-5 test materials behaved the same. Testing of 15-5 started with three specimens, but one specimen was inadvertently overloaded and was rendered unusable. The other two specimens passed the 720-hour testing at 95 percent AYS without developing cracks.

Testing in a corrosive hydrogen cell – Environment 2

As the initial hydrogen atmosphere did not result in any HIC, the investigations were performed in acetic acid with a partial pressure of 145 psi (10 bar) hydrogen and 73 psi (5 bar) carbon dioxide. The aqueous solution was adjusted until a pH 3.0 was achieved. This solution was intentionally chosen from representative hydrogen production environments. The thought is that these environments could create hydrogen atoms, thus resulting in HIC.

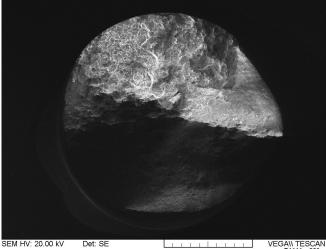
This procedure was created to generate hydrogen atoms by corrosion on the surface. The process was designed to focus the corrosion along the specimen gauge length. This procedure proved to be effective, and HICs were then achieved.

F6NM investigations showed that at a 95 percent AYS in the initial hydrogen atmosphere (env 1), no crack formation was observed after 720 hours for all three specimens. In the corrosion hydrogen cell atmosphere (env 2), two of the three specimens resulted in no crack formation after 720 hours. One specimen was tested for an additional 720 hours and resulted in no crack formation. The single failed specimen ruptured after 305 hours.

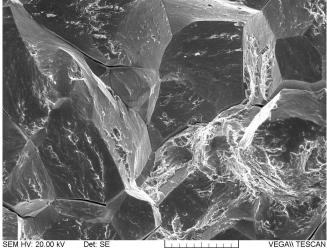
The failed F6NM specimen is shown in Figure 8. There was no visible necking or orange peel effect as would be seen on an overload failure. The surface has a matt finish. The fractography examination revealed features typical of a hydrogen induced failure (Figures 9 - 12) that are smooth grain facets and cracks at the grain boundaries. Based on these results, one can conclude that the specimen failed due to hydrogen induced embrittlement.



Figure 8. Overview of the F6NM test specimen.



View field: 7.33 mm WD: 41.24 mm 2 mm R1111_360 Figure 9. Fracture surface of broken F6NM specimen.



View field: 220.0 µm WD: 14.77 mm 50 µm R1111_338 Figure 11. Intergranular cracking along the grain boundaries.

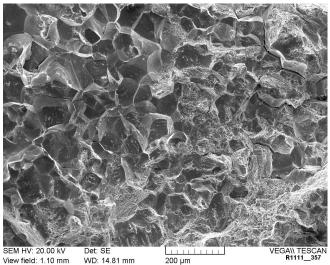


Figure 10. Transition zone: Fracture surface with inter crystalline cracking and forced fracture.

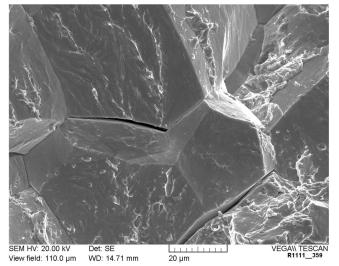


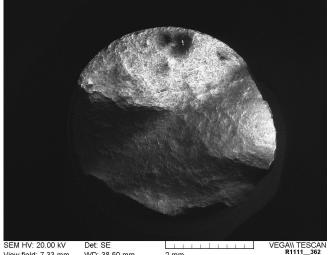
Figure 12. Higher magnification of Figure 11 with grain facets and opened grain boundaries.

The 15-5 investigations showed that at a stress load of 95 percent AYS in the hydrogen atmosphere (env 1), no crack formation was detected on two of two specimens after 720 hours. In the corrosion hydrogen cell atmosphere (env 2), both specimens underwent crack formation with rupture occurring after 35 and 144 hours.

The investigation of one broken 15-5 specimen is shown in Figures 13 - 15. The specimen has a flat undeformed fracture surface which changes over to a shear lip. Figure 13 displays the transition zone of the fracture where the embrittlement fracture type changed to a ductile forced fracture. In the detailed view, a typical intercrystalline fracture surface caused by hydrogen induced embrittlement with opened grain boundaries and visible grain facets can be observed. These results suggest that the specimens failed due to hydrogen induced embrittlement.



Figure 13. Detailed view of the 15-5 specimen.



View field: 7.33 mm WD: 38.50 mm 2 mm Figure 14. General view on the fracture surface.

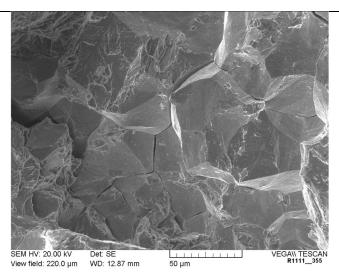


Figure 15. Inter crystalline fracture with grain facets and cracked grain boundaries.

The alloy 718 tests were completed with a stress load of 90 percent AYS. In all three tests, the alloy 718 specimens showed no cracks or fractures with a YS of minimum 160 Ksi (1100 MPa). In each test lot, three specimens from three heats were examined in the corrosion hydrogen cell atmosphere (env 2); no crack formation was detected after 720 hours.

While Raymond (1988) considered 720 hours as acceptable to predict failure in materials susceptible to HIC, a longer test period was desired to gain acceptance of the data, particularly in borderline cases per NACE TM0117. Borderline cases occur when a specimen meets the post 720-hour testing acceptance criteria, but cracks are detected beyond the acceptable criteria of 10X during NDT, or general corrosion is observed on the proof ring specimen.

Testing was therefore extended; this was accomplished by an additional 1,440 hours of testing. In each test lot, three specimens from three heats were examined in the corrosion hydrogen cell atmosphere (env 2). The test results showed no crack formation that could be detected via 10X or NDT after a total of 2,160 hours.

CONCLUSIONS

The trend toward using hydrogen as a fuel source is accelerating. The current machinery standards for testing and service of centrifugal compressors do not directly relate to simulated production environments or define fit-for-service limitations. Some traditional (e.g. mechanical properties and hardness) and non-traditional (e.g. microstructure and lattice structure) material properties were evaluated on metals F6NM, 15-5, and 718. Importantly, all these materials exceeded the compositional maximum hardness and yield strength limits specified in API 617 eighth edition.

The following observations were made:

In Hydrogen Induced Cracking (HIC) tests in a clean hydrogen environment (H_2) at a partial pressure of 145 psi (10 bar) stressed at or above 90 percent AYS, the metals F6NM, 15-5, and 718 all survived the 720 hours. No post-test visual findings were observed on any specimens. In addition, non-destructive testing (NDT) did not detect cracks. These results therefore indicate that F6NM, 15-5, and 718 are acceptable materials in a clean hydrogen environment, with mechanical properties exceeding API 617 eighth edition limits.

HIC tests of F6NM, 15-5, and 718 in the corrosive hydrogen cell at a partial pressure of 145 psi (10 bar)—stressed at or above 90 percent AYS—showed mixed results. While 718 survived 90-day (2,160-hours) testing, both F6NM and 15-5 had HIC failures. There may be multiple factors that resulted in the performance of F6NM and 15-5, including but not limited to, heat treatment, AYS, microstructure, crystal lattice structure, or any combination of variables. As the root cause is not fully understood, additional evaluation may be needed.

This study concludes that a modified NACE TM0177 test can produce HIC. Using this modified test, the OEM can achieve different behavior of different grades and validate acceptable material strengths at different application ranges. The evidence suggests that Alloy 718 at a minimum design yield strength of 160 ksi (1100 MPa) is acceptable for representative hydrogen production environments, with mechanical properties exceeding API 617 eighth edition limits.

This OEM concludes that after testing nine samples from three heats provided by two suppliers, no differences in results were observed between the 30-day and 90-day tests. Therefore, a 30-day (720-hour) duration is an acceptable limit for determining acceptable material strength for hydrogen production per API 617, eighth edition. This is supported by the F6NM and 15-5 failures that occurred before the mid-point on the 30-day test and shows that HIC progress rapidly.

The OEM's results identify a window of operation for alloy 718, with a higher yield strength than is allowed by API 617, eighth edition limits. It is the responsibility of the oil & gas compression community at large to determine acceptance and safety margins. The challenge is better fit-for-service environments. This work shows a path to achieve new hydrogen production limits and rules within API 617, eighth edition.

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NOMENCLATURE

- HIC = Hydrogen Induced Cracking
- HAC = Hydrogen Assisted Cracking
- HE = Hydrogen Embrittlement
- YS = Yield Strength
- SSRT = Slow Strain Rate Test
- AYS = Applied Yield Strength
- SSC = Sulfide Stress Cracking
- SCC = Stress Corrosion Cracking
- NDT = Non-Destructively Tested
- KV2 = V-notched Impact Specimen
- J@RT = Joules at room temperature
- UTS = Ultimate Tensile Strength
- E = Elongation
- RA = Reduction in Area
- HRC = Rockwell Hardness

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