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Alloy for resistance to polythionic acid stress corrosion cracking for hydroprocessing applications

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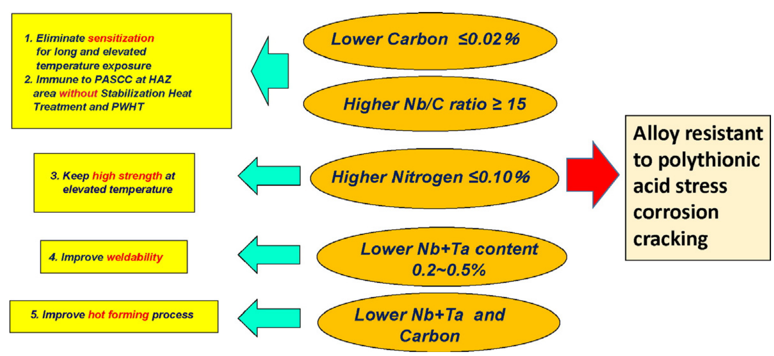
HIGHLIGHTS

- Proprietary version of type 347LN with low C, high N and optimum level of Nb did not sensitize after 10,000 h at 565 °C.
- No evidence of sensitization of a hydrocracker heater tube after 100,000 h of operation at average tube wall temperature of 460 °C
- Alloy does not require thermal stabilization heat treatment.
- No need to neutralize when exposed to sensitization conditions and H2S to prevent polythionic acid stress corrosion cracking.

GRAPHICAL ABSTRACT



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ABSTRACT

In certain process units, such as hydrocracking, soda ash washing (neutralization) of austenitic stainless steel is required during turnarounds to mitigate the potential for polythionic acid stress corrosion cracking (PTA SCC). Soda ash washing can be a costly and time consuming endeavor for the refiner. This paper introduces a grade of austenitic stainless steel, a proprietary version of Type 347LN, that does not sensitize with long-term exposure to elevated temperature, thus rendering it immune to PTA SCC. ASTM A262 Practice A corrosion test results will be presented for samples isothermally aged at 565 °C for a duration of up to 10,000 h. These data will be compared to samples of conventional Type 347, Type 321, and Type 304H similarly aged. Photomicrographs will be shown that demonstrate the lack of grain boundary sensitization, and also the lack of grain boundary ditching in the oxalic acid test. Replica analysis of a heater tube from commercial service at 460 °C average tube wall temperature for 12 years showing no evidence of sensitization or grain boundary precipitation will also be presented.

1. Introduction

Hydrotreating and hydrocracking are two widely used processes in the petroleum refining industry [1,2]. Hydrotreating is used by refiners to remove impurities such as nitrogen, sulfur and metals for the preparation of feeds for various other process units such as catalytic reformers and fluid catalytic crackers as well as to upgrade straight run products and those from other refining processes such as visbreakers and delayed cokers in order to meet current fuel standards. Typical reactor operating conditions are 2.5 to 10 MPa pressure at temperatures of 325 to 425 °C. Hydrocracking is a very versatile refining process, which can process any fraction from naphtha to vacuum gas oils to produce almost any desired refinery product having a molecular weight lower than that of the charge stock. During the hydrocracking reaction sulfur, nitrogen and oxygen are almost completely removed, olefins are saturated and

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some aromatics converted to other materials. The resulting products are a mixture of essentially paraffins and naphthenes with reduced aromatics content. Typical products include LPG, naphtha, jet fuel, diesel, and lubricating oils. Reactor operating temperatures are 350 to 450 °C with operating pressures between 10 and 17 MPa.

At these operating conditions normal austenitic stainless steel will sensitize with Cr-carbides forming at the grain boundaries. The feeds to both of these processes contain S contaminant with the S reacting with H_2 to form H_2S. H_2S then reacts with metal resulting in an iron sulfide scale on the surface of the austenitic stainless steel. The combination of a sensitized microstructure, sulfide scale, moisture, oxygen and residual or applied tensile stress are necessary conditions to produce polythionic acid stress corrosion cracking (PTA SCC) [3].

Polythionic acid is defined as $H_2S_xO_6$; but it is the tetrathionic acid ($H_2S_4O_6$) that typically induces stress corrosion cracking in sensitized austenitic stainless steel. Once the metal is sensitized, sulfide scale on the metal can react with moisture and air, typically during shutdowns, to form polythionic acid. This acid then attacks the metal down the Cr depleted grain boundaries.

Because of the susceptibility of austenitic stainless steels to polythionic acid stress corrosion cracking during a shutdown, a procedure as described in NACE SP0170 [4] such as soda ash washing prior to opening the equipment to the atmosphere, or circulating dry air is required. Such procedures are costly and time consuming. To minimize the potential for PTA SCC, application of chemically stabilized Type 321 or 347 austenitic stainless steels are often used, but these do not eliminate the need for neutralization because ultimately they sensitize and are then susceptible to PTA SCC [5–8].

One of the necessary requirements for PTA SCC to occur is a sensitized microstructure where there is chromium depletion adjacent to the grain boundaries. Sensitization occurs between 370 and 400 °C (depending on the grade of austenitic stainless steel) to 815 °C [3]. It is the precipitation of the Cr-rich carbides, such as $(Cr, Fe)_{23}C_6$ that causes the depletion of chromium in the austenitic phase adjacent to the grain boundaries. When the Cr content in this depleted zone falls below a critical value required for passivity, the material becomes susceptible to PTA SCC. There are a number of approaches for evaluating the susceptibility of an alloy to sensitization and PTA SCC, including the various practices in ASTM A262, ASTM G35 and ASTM G108. Various studies have been conducted to further elucidate the mechanism of PTA SCC using tetrathionate ($K_2S_4O_6$) solution as the aggressive agent [9,10].

Resistance to intergranular attack from PTA can be obtained by grain boundary engineering [11–13]. Using this approach a patented [14], proprietary version of Type 347LN (UNS S34751) has been developed that does not sensitize after long-term exposure at elevated temperatures. This alloy is commercially named smiLLe™. The laboratory tests reported here will demonstrate that this alloy does not sensitize after exposure for 10,000 h at elevated temperature and thus is immune to PTA SCC. Additionally, field testing of a commercial fired heater tube will demonstrate that the alloy did not sensitize after >100,000 h of operation.

2. Method

As a preliminary test for sensitization the alloys were heated to 675 °C for 1 h and 10 h, and evaluated for grain boundary ditching per ASTM A262 Practice A. To fully evaluate sensitization and susceptibility to PTA SCC, coupons of Types 304H, 347L and proprietary 347LN were encapsulated in a nitrogen purged and sealed quartz ampoule to minimize surface oxidation, and then heated to 565 °C for 3000 and 10,000 h. The 565 °C temperature was selected because it is the nose of the TTS diagram for Type 347 stainless steel [15]. Coupons were isothermally aged and tested in both the welded and unwelded condition. Samples that were tested for <3000 h were not sealed in a quartz ampoule.

3. Results and discussion

The composition of the proprietary alloy used in this study along with the composition range for generic Type 347LN, as well as the weld consumable used for the welded coupons is shown in Table 1. The composition range for Type 347 is also shown for comparison. This alloy has a low carbon content of <0.02% and optimized nitrogen content of 0.06–0.10%, which is required to compensate for the lower carbon content in order to meet the strength requirements. With the lower carbon content, less niobium (columbium) is required for stabilization which contributes to improved weldability of the alloy.

3.1. Sensitization testing

Evaluation of susceptibility of the proprietary alloy to sensitization and PTA SCC was initially conducted using the oxalic acid grain boundary etching technique per ASTM A262 Practice A. As shown in Fig. 1, after 3000 h at 565 °C the 304H alloy had a fully ditched grain boundary structure. After 3000 and 10,000 h Type 347L also exhibited ditched grain boundaries. No ditching was observed for the proprietary Type 347LN after 3000 and 10,000 h.

No observable susceptibility of proprietary Type 347LN to intergranular corrosion per ASTM A262 Practice C boiling nitric acid was observed using the criteria [16] of ≤24 mpy at 675 °C for 1 h. ASTM A262 Practice C requires that extra low carbon and stabilized grades be tested after a sensitizing heat treatment that maximizes carbide precipitation. For this analysis both the plate (unwelded) and the weldment were tested. Extending the heat treatment to 10 h did not appreciably change the corrosion rate. The samples isothermally aged at 565 °C were also evaluated for intergranular corrosion, and the corrosion rate for proprietary Type 347LN was substantially less than for Type 347L or Type 304H (Table 2). The increase in corrosion rate with aging of the proprietary 347LN alloy may be the result of sub-micron sigma phase.

PTA SCC testing of welded proprietary Type 347LN without thermal stabilization heat treatment, after aging for 10,000 h at 565 °C using U-bend samples per ASTM G35 with 720 h immersion was conducted. Similarly fabricated and isothermally aged coupons of Type 321 were also tested. The Type 321 coupons exhibited evidence of PTA SCC after aging for as little as 100 h at 550 °C and 600 °C (Fig. 2, top row).

Intergranular corrosion testing of welded proprietary Type 347LN without thermal stabilization heat treatment was tested per copper-sulfate-16% sulfuric acid as described in ASTM A262 Practice E. After 72 h immersion in boiling sulfuric acid, no evidence of intergranular cracking or crazing upon bending was seen for the proprietary Type 347LN. However, Type 347 exhibited slight attack after only 30 h, and heavy attack after 100 h at 550 °C (Fig. 2, bottom row).

Table 1
Chemical composition.

	C	Si	P	S	Mn	Ni	Cr	Nb	N
Generic Type 347LN	0.005–0.020	1.00 Max	0.045 Max	0.030 Max	2.00 Max	9.0–13.0	17.0–19.0	0.20–0.50 15 X C Min	0.06–0.10
Type 347	0.08 Max	0.75 Max	0.045 Max	0.030 Max	2.00 Max	9.0–13.0	17.0–19.0	10 X C Min 1.00Max	–
Proprietary Type 347LN (this study)	0.007	0.36	0.028	0.001	1.48	10.0	17.2	0.31	0.08
Proprietary Type 347LN welding consumable	0.016	0.43	0.003	0.003	1.70	9.4	20.6	0.44	0.16

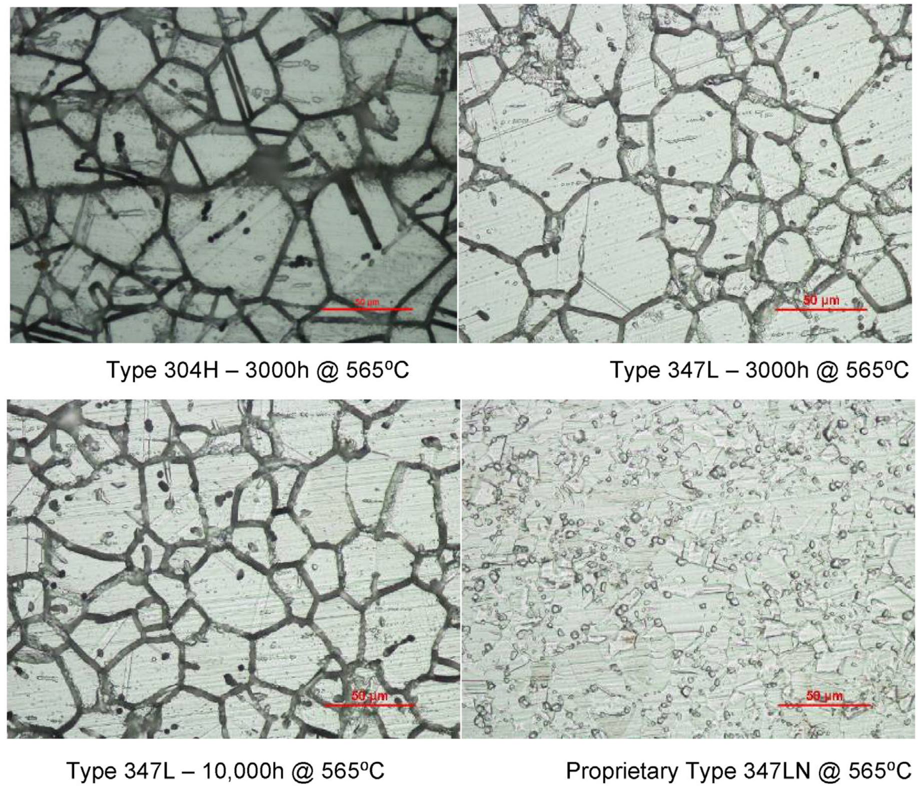

Fig. 1. Results from oxalic acid grain boundary etching per ASTM A262 Practice A. After 10,000 h the proprietary Type 347LN did not exhibit any grain boundary ditching. Grain boundary ditching is observed for Type 347L after only 3000 h at 565 °C.

U-bend samples of the proprietary Type 347LN after isothermally aging for 10,000 h at 565 °C did not crack after exposure to 1% $K_2S_4O_6$ acidified by H_2SO_4 to pH 2.0 at room temperature for 100 h per the method described by Hosoya et al. [17] as shown in Fig. 3. However, Type 347H isothermally aged for 300 h at 600 and 650 °C, and not given a thermal stabilization heat treatment did crack (Table 3). No cracking was detected for the Type 347H after a 900 °C thermal stabilization heat treatment for 1 h in this test. These results confirm that thermal stabilization heat treatment will improve the PTA SCC resistance of Type 347, but such thermal stabilization heat treatment is not required for proprietary Type 347LN.

Electrochemical reactivation (EPR) analysis per ASTM G108 was another technique used to evaluate whether the proprietary Type 347LN was susceptible to sensitization. This method uses a potentiodynamic sweep from the passive to active regions of electrochemical potentials with the measured amount of charge being associated with corrosion of the Cr-depleted zones adjacent to the grain boundaries. This test showed no sensitization for proprietary Type 347LN after aging for 3000 and 10,000 h at 565 °C, while Type 304H aged for 3000 h at 565 °C demonstrated sensitization. Type 347H aged for 1000 h at 565 °C also exhibited sensitization (Fig. 4).

Table 2
Nitric acid test ASTM A262 practice C corrosion rate.

	Corrosion rate (mpy) unwelded/welded
Proprietary 347LN un-aged	17.1/11.7
Proprietary 347LN 1 h @ 675 °C	21.3/23.7
Proprietary 347LN 10 h @ 675 °C	22.4/26.3
Proprietary 347LN 2000 h @ 565 °C	52.2/58.5
Proprietary 347LN 10,000 h @ 565 °C	53.0/67.8
347L 10,000 h @ 565 °C	470.7/not tested
304H 2000 h @ 565 °C	2758.7/not tested

In addition to wrought material, a cast version of this material (proprietary CF8C) has also been developed. The cast material was isothermally aged at 565 °C for 3000 and 10,000 h, both in the welded and unwelded conditions. Testing was conducted using the oxalic acid grain boundary etching technique per ASTM A262 Practice A (Fig. 5). No grain boundary ditching was observed in either the base metal or the weld metal. Additional testing was conducted using ASTM A262 Practice E, and no cracks were observed, whereas both CF8 and standard CF8C showed cracking after aging for 10,000 h (Table 4).

3.2. Microstructure

Transmission electron microscopy (TEM) was used to identify the precipitates in the proprietary Type 347LN. After a 700 °C aging treatment for 24 h, fine precipitates of NbC and NbCrN were identified (Fig. 6). These precipitates were on average about 70 nm in size and were located throughout the grains. After 3000 h at 565 °C the precipitates may have grown slightly but remained scattered throughout the grains, with the number present in grain boundaries remaining virtually unchanged. However, for higher carbon levels than allowable for 347LN, the precipitates were NbC, Cr_7C_3 and $Cr_{23}C_6$ in the grain boundaries. For higher nitrogen, the precipitates were NbC, NbCrN, CrN and $Cr_2(C, N)$ with many in the grain boundaries.

Thus, there is an optimum amount of carbon, nitrogen, and niobium (columbium) that prevents precipitation of chromium carbides, chromium nitrides, or chromium carbonitrides along the grain boundaries. These results are similar to those reported by Ayer et al. [18] for conventional Type 347LN treated at 600 °C for 8000 h. For higher nitrogen contents, Downey et al. [19] found that for Type 316LN heat treatment in the sensitization temperature range produced chromium nitride precipitation in the grain boundaries that depleted the chromium in the vicinity of the grain boundaries, resulting in sensitization.

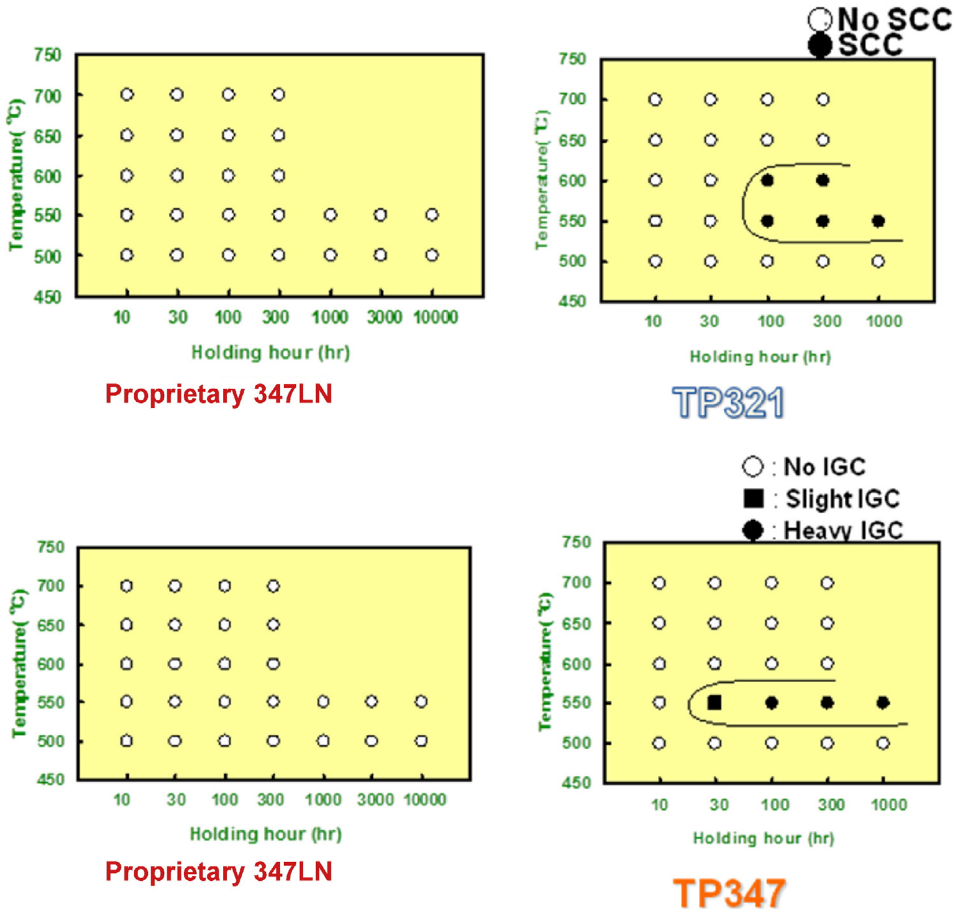

Fig. 2. Effect of aging on PTA SCC resistance for weldments per ASTM G35 of U-bend specimens (top) and on IGC resistance for weldments per ASTM A262 Practice E (bottom).

3.3. Mechanical properties

Because of the addition of nitrogen, the reduced carbon level does not appreciably impact the high temperature strength of the alloy. As shown in Fig. 7 the high temperature tensile and yield of proprietary Type 347LN closely track the values of Type 347H up to about 750 °C.

3.4. Weldability

Because of the lower niobium (columbium) content of proprietary 347LN, the weldability of the alloy is improved compared to conventional Type 347.

The Varestraint weld-cracking test is used to evaluate the susceptibility of austenitic stainless steels to hot cracking, where the resistance to cracking is evaluated by the length of cracks generated by bending along a 300 mm radius shaped tool while GTAW welding without a filler metal on the surface. Cracks will then be generated at the portion which receives the bending load right after solidification. The length of these cracks indicates the susceptibility of the alloy to hot cracking.

As shown in Fig. 8, the observed crack length for proprietary Type 347LN was similar to that of Type 304 and substantially better than Type 347. Proprietary Type 347LN was also superior to Type 316 and Type 321 in this regard. By balancing the N level with the Ni equivalent modifier level in the weld consumable to that of the base metal, hot cracking and other cracking susceptibility issues were eliminated. Thus the targeted δ-ferrite of 5 to 10% appears to eliminate the impact of nitrogen content in the Type 347LN on weld cracking [20].

A matching filler metal has been developed with a composition as noted in Table 1. Weld procedure specifications for the welding methods SMAW, GTAW, FCAW, SAW, and ESW (single layer and double layer) have also been developed. As noted the weld did not sensitize or crack in any of the conducted tests.

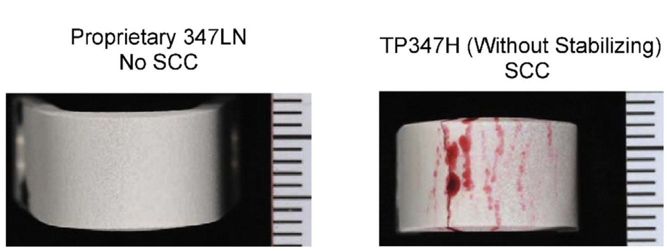

Fig. 3. U-bend samples after exposure to 1%$K_2S_4O_8$ acidified by H_2SO_4 showing no SCC for proprietary Type 347LN and SCC for Type 347H.

3.5. Field experience

This alloy has been used for commercial fired heater tube applications in Japan. To evaluate the resistance of the alloy to sensitization in long-term service, metallographic extraction replicas were made of a hydrocracker heater tube. The tubes had been in service for over 100,000 h at an average tube wall temperature of 460 °C and maximum operating temperature of 525 °C.

The weld metal, heat affected zone, and base metal were examined. Neither the base metal nor the weldment had been given a thermal stabilization heat treatment. No precipitation (sensitization) was found in the grain boundaries as examined optically (Fig. 9) from an extraction replica prepared per ASTM E1351 or in the transmission electron microscope (TEM).

Table 3
U-bend tests in $K_2S_4O_6$.

	565 °C		600 °C			650 °C			700 °C		
	3000 h	10,000 h	300 h	1000 h	10,000 h	300 h	1000 h	10,000 h	100 h	300 h	1000 h
Proprietary Type 347LN	No SCC	No SCC	No SCC	No SCC	No SCC	No SCC	No SCC	No SCC	No SCC	No SCC	No SCC
Type 347H											
W	–	–	No SCC	No SCC	–	No SCC	No SCC	–	No SCC	No SCC	No SCC
W/O	–	–	SCC	SCC	–	SCC	–	–	No SCC	No SCC	No SCC

Proprietary Type 347LN not thermally stabilized. Type 347H with and without 900 °C 1 h thermal stabilization heat treatment.

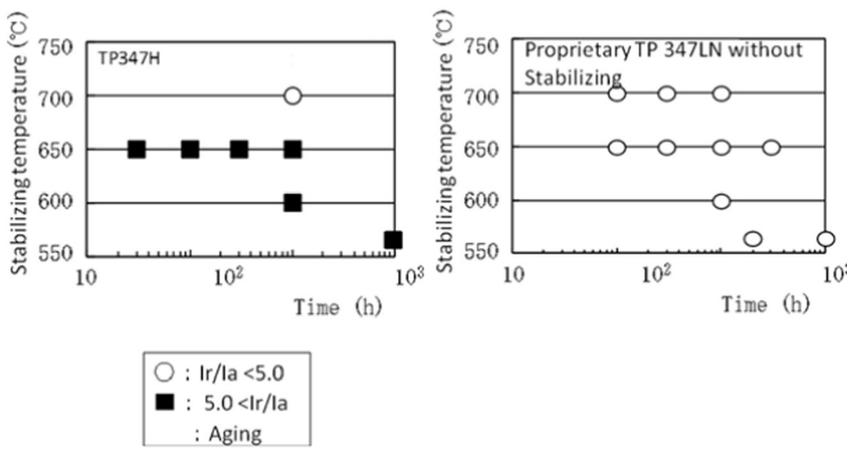

Fig. 4. EPR test showing no sensitization for proprietary 347LN but sensitization for 347H.

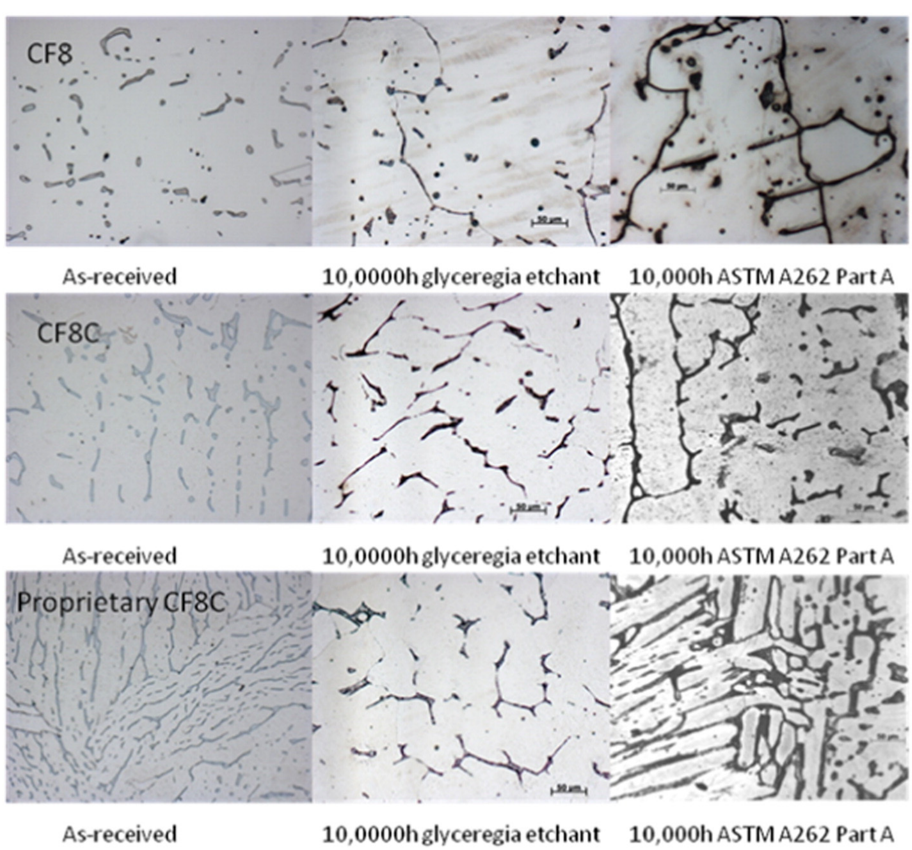

Fig. 5. Cast version of proprietary CF8C showing no sensitization after aging for 10,000 h at 565 °C.

Table 4
Sensitization results of cast alloy.

	Type	Cast		Weld Metal	Wrought	
		Base metal	HAZ		HAZ	Base metal
Proprietary CF8C	Cast	O (O)				
CF8	Cast	X (Δ)				
CF8C	Cast	X (O)				
Proprietary CF8C welded to proprietary wrought TP347LN	Weld	O (O)	O (O)	O (O)	O (O)	O (O)

O: no crack, Δ: crack length ≤ 50 μm, X: crack length > 50 μm test results after 10,000 h at 565 °C.
( ): test results after 3000 h at 565 °C.

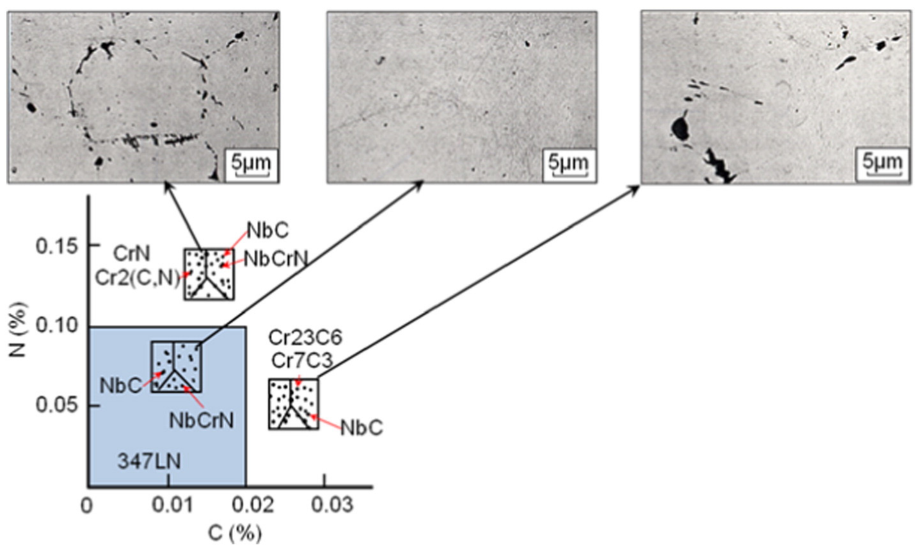

Fig. 6. Effect of C and N on precipitates of Type 347 aged at 700 °C for 24 h. Only fine NbC and NbCrN precipitates are found for proprietary Type 347LN in bright-field transmission electron micrographs.

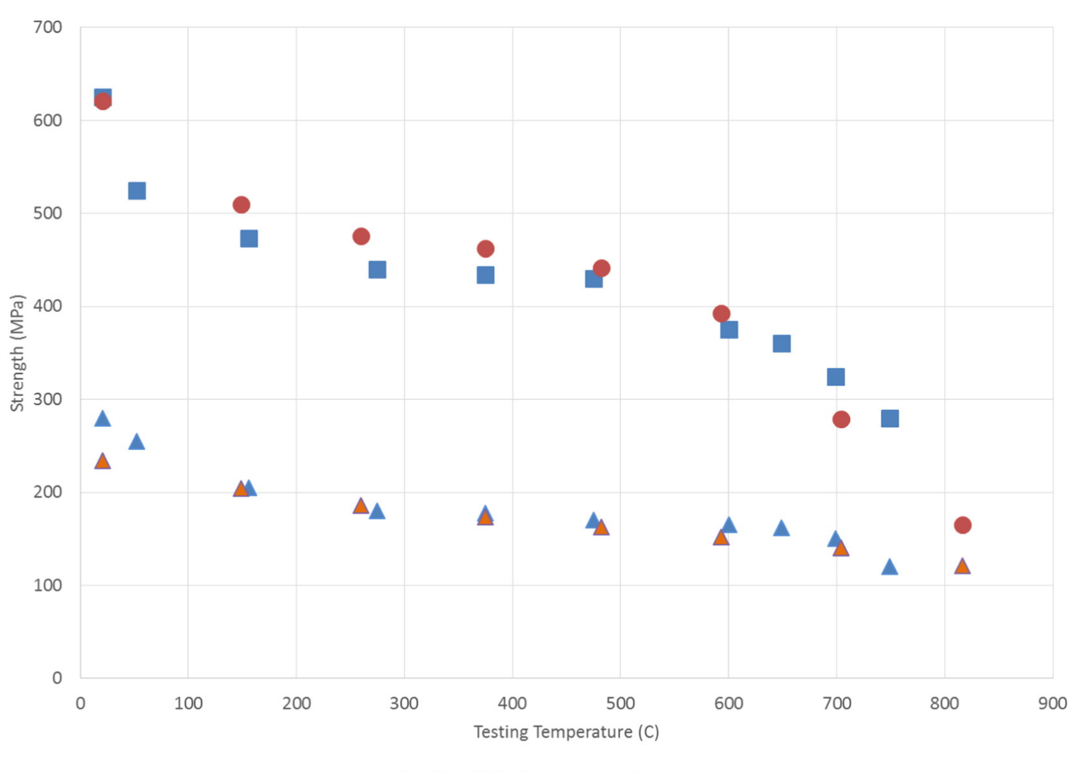

Fig. 7. Comparison of tensile and yield strength for proprietary Type 347LN and Type 347H showing them to be reasonably comparable to 750 °C.

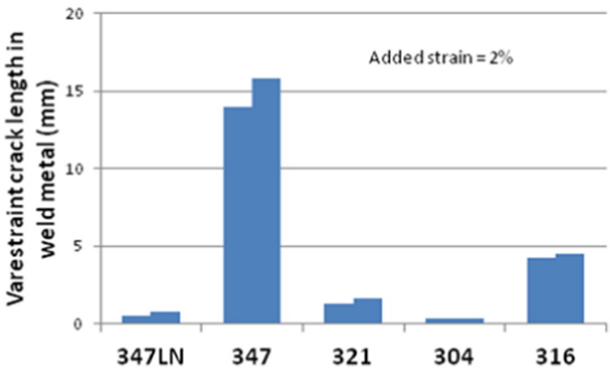

Fig. 8. Results of Varestraint test showing hot cracking tendency comparing proprietary Type 347LN with Type 347, Type 321, Type 304, and Type 316 stainless steels.

Electrochemical potentiometer reactivation (EPR) test per ASTM G108 on the OD of the fired heater tube at all locations produced a Pa value of 0 coulombs/cm^2. These results demonstrate that the metallurgy did not sensitize after the prolonged service.

3.6. Product forms

The product forms necessary to design a unit using proprietary Type 347LN are covered by various ASTM/ASME specifications. These are listed in Table 5.

For fired heaters, creep curves have been submitted to API 530 for inclusion in that document. As of this writing, the curves have not yet been adopted by API 530. However, currently fired heater coils can be designed using the rules in API 560.

3.7. Codes and standards

The proprietary Type 347LN stainless steel described in this paper is governed by ASME Code Case 2196-3, and can be used up to a maximum design temperature of 650 °C. The Code allowable stresses for proprietary Type 347LN from 95 to 540 °C are slightly lower than those for Type 347. However, from 600 °C to 650 °C the Code allowable stresses for proprietary Type 347LN are considerably higher. This same trend is seen in Fig. 7.

The Code Case also gives chemical requirements and mechanical property requirements for the alloy, as well as yield and tensile strength properties as a function of temperature.

Table 5
Applicable ASTM specifications.

Plate	ASTM A240-347LN
SMLS pipe	ASTM A312-347LN
SMLS tube	ASTM A213-347LN
Welded pipe	ASTM A358-347LN
Fittings	ASTM A403-WP347LN
Pipe, flange, forged fitting, valve and parts	ASTM A182-F347LN
Forgings	ASTM A965-347LN
Bolts and nuts	ASTM A193 B8C/ASTM A194 8C
Wire	ASTM A580-347LN
Casting	ASTM A351 CF8C

3.8. Application to hydroprocessing metallurgy

For hydroprocessing applications the high H_2 pressure and temperature require the specification of austenitic stainless steel or stainless steel weld overlay on low alloy steel. As noted for these process conditions, normal austenitic stainless steel alloys are susceptible to PTA SCC. An alloy that is immune to PTA SCC and has acceptable mechanical properties is the proprietary Type 347LN and thus is suitable for such applications. It is not known as to whether compositions of 347LN outside of the tighter compositional limits for the proprietary Type 347LN or that specified in the patent will have immunity to PTA SCC. As suggested by the work of Ayer et al. [18] there appears to be an optimum C, N and Nb composition that retards the formation of $Cr_{23}C_6$. Similar studies as described here would be required to answer that question. Other technologies where this proprietary Type 347LN alloy may have applicability include biomass conversion [21–24] and air pollution control processes [25].

4. Conclusions

1. The proprietary version of Type 347LN has been shown to be immune to PTA SCC as demonstrated by sensitization and cracking tests reported herein. Therefore, downtime protection in accordance with NACE SP0170 is not necessary, saving the refiner considerable time and money at each turnaround.
2. Analysis of a commercial heater tube fabricated from this alloy that has been in operation for >100,000 h in a hydrocracker fired heater exhibits no evidence of sensitization.
3. This alloy does not require thermal stabilization heat treatment as is sometimes specified for Type 347.
4. With the addition of nitrogen, the alloy has comparable high temperature strength to that of Type 347H austenitic stainless steel up to 750 °C.

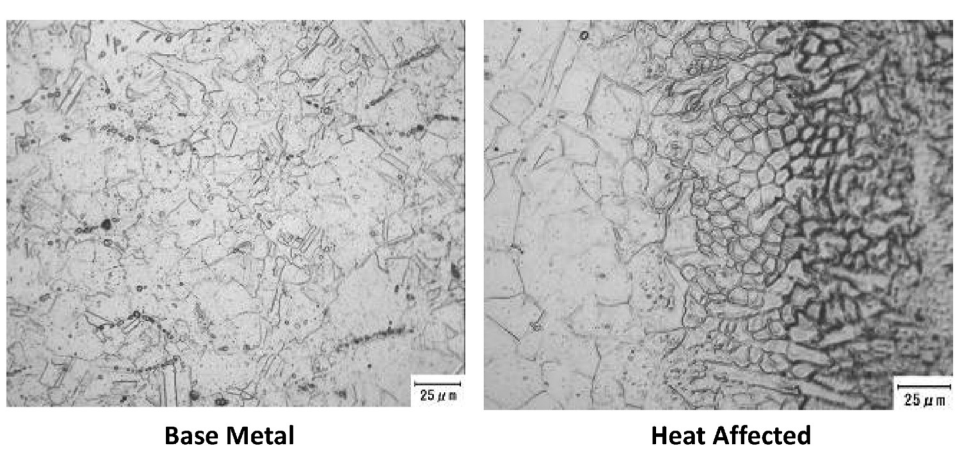

Fig. 9. Optical micrograph of extraction replica taken from a fired heater tube showing no sensitization after >100,000 h of operation, etched with glyceregia.

5. The proprietary version of Type 347LN is quite acceptable to be used in applications where the temperature of operation is between 350 and 750 °C and there is sufficient amount of H_2S to produce a continuous sulfide scale. Applications where PTA SCC is an issue and special precautions to prevent its occurrence are prime examples. One such application is for hydroprocessing heater tubes and the entire reactor circuit.
6. This alloy is being extended into weld overlay so that the entire reactor circuit does not need to be neutralized during a shutdown. Sensitization testing is currently in progress.

Appendix A. Supplementary data

Supplementary data to this article can be found online at http://dx.doi.org/10.1016/j.matdes.2016.07.067.

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