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Effect of Sensitization on the Corrosion Resistance of an Advanced Version of Alloy UNS N08825

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ABSTRACT

Alloy UNS(1) N08825 is a titanium-stabilized fully austenitic nickel-iron-chromium alloy with addition of copper and molybdenum. The alloy was designed for applications in the chemical process industry and was later applied in the oil and gas industry.

Because of its high nickel content, UNS N08825 shows an outstanding resistance to stress corrosion cracking in aqueous and acidic chloride-containing solutions. However, the molybdenum content of 2.5 to 3.5 wt.% limits its resistance to pitting and crevice corrosion in highly concentrated chloride-containing environments.

To address this weak point, a new advanced alloy with increased molybdenum content, which will be called Alloy 825 CTP, was developed. Previous corrosion tests showed an increased critical pitting temperature measured on the new alloy[1] and based on these data further corrosion tests were performed.

Alloy 825 CTP has been tested according to ASTM G48 in solution-annealed and in PWHT condition to study the pitting and crevice corrosion resistance. In addition, corrosion tests were performed to study its corrosion resistance considering different mechanisms of environmental cracking. Sulfide stress cracking (SSC) and galvanically induced hydrogen stress cracking (GHSC) tests were performed

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(1) Unified Numbering System for Metals and Alloys (UNS), SAE International, Warrendale, PA

according to NACE(2) TM0177-2016 Method A. Stress corrosion cracking (SCC) resistance was investigated using C-ring tests according to NACE TM0177 Method C at Level VI for 3 months and Level VII for 1 month. Slow strain rate (SSR) tests under cathodic polarization were performed to study the resistance to hydrogen embrittlement (HE).

In terms of resistance to (localized) corrosion and hydrogen embrittlement, the newly developed Alloy 825 CTP was found to perform equally well or even better than the conventional material.

Key words: Alloy UNS N08825, Alloy 825 CTP, PWHT, Ni39Fe28Cr22Mo6, corrosion behavior, ASTM G48-C, ASTM G48-D, SCC, SSC, GHSC, Hydrogen Embrittlement, NACE TM0177

INTRODUCTION

Alloy UNS N08825 is a titanium-stabilized fully austenitic nickel-iron-chromium alloy with addition of copper and molybdenum[2], which is resistant to corrosion in acids and alkalis under both oxidizing and reducing conditions. Thus, it is used for a great variety of applications including (petro)chemical industries, oil and gas production and processing and maritime applications. The high nickel content grants excellent protection against stress corrosion cracking in aqueous and acidic chloride-containing environments. However, alloy UNS N08825 has only a limited resistance to chloride-induced localized corrosion, which is a common corrosion type in the oil and gas production.[3]

Better pitting and crevice corrosion resistance than those of Alloy UNS N08825[1,4] in chloride containing media has been achieved by optimizing the chemical composition of the material. By increasing the molybdenum content, the PRE number(3) was increased from 33 to 42[1]. For the newly created Alloy 825 CTP an improved resistance to localized corrosion was predicted. This improvement confirmed by the increase of the critical pitting temperature (CPT) from 30 °C (86 °F) for alloy UNS N08825 to around 55 °C (131 °F) for Alloy 825 CTP.[1]

It is well known that Alloy UNS N08825 tends to sensitize when it is exposed to temperatures between approximately 600 °C (1112 °F) and 800 °C (1472 °F). These temperatures can occur during welding processes in the heat affected zones (HAZ), during Post Welding Heat Treatment (PWHT) or after cladding process. During the thermal exposure, Cr-rich carbides precipitate and the associated depletion of chromium in the vicinity leads to localized corrosion[5-8]. Based on the literature[5], the best manner to stabilize the material is to precipitate $M_{23}C_6$ at a temperature where the diffusion of Chromium is sufficiently rapid. Still according to the authors, the optimal temperature for UNS N08825 to prevent chromium depletion is in the range of 927-982 °C (1700-1800 °F). Because of the changes in the chemical composition, the precipitation behavior of the improved alloy has been altered, too. When decreasing the titanium content, an important carbide former is missing in the matrix of the alloy. Consequently, a depletion of chromium in areas adjacent may eventually occur. The parameters of proper heat treatment to control the precipitation behavior are therefore of great importance.

The target of this investigational program was to examine the pitting corrosion behavior of Alloy 825 CTP as compared to alloy UNS N08825. For this, tests according to the ASTM(4) G48[9] were performed to determine the CPT and CCT. The influence of annealing temperature and sensitization heat treatment has been also considered.

Moreover, sulfide stress cracking (SSC), galvanically induced hydrogen stress cracking (GHSC), stress corrosion cracking (SCC) – according to the NACE TM0177[10] – and slow strain rate (SSR) tests were performed to examine the corrosion resistance under sour gas conditions and the potential for hydrogen embrittlement.

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

(3) Pitting Resistance Equivalent Number: PRE = %Cr + 3.3 x %Mo

(4) American Society for Testing and Materials (ASTM) International, 100 Barr Harbor Drive, West Coshohocken, PA, 19428

EXPERIMENTAL PROCEDURE

Material

The tested materials with the nominal chemical composition of the main elements are listed in **Table 1**.

Table 1
Chemical composition of tested materials in percentage of mass fraction (wt.%)

Heat ID	Material	Cr	Ni	Fe (balance)	Cu	Mo	Ti
A	825 CTP	22.59	39.28	28.59	2.1	5.66	0.07
B	825 CTP	22.28	39.19	29.02	2.05	5.88	0.06
C	UNS 08825	22.80	39.38	30.27	1.98	3.28	0.81
D	UNS 08825	22.73	39.20	30.66	1.94	3.12	0.69
E	UNS 08825	22.50	39.32	30.73	1.95	3.15	0.76

Heats A and B are plate material from Alloy 825 CTP with 5 mm (0.08-in) and 16 mm (0.25-in) thickness. These heats were produced in an open melting process and continuous casting. After hot rolling, the plates were annealed at 1010°C (1850°F). Heats C, D and E are plates from the standard production and composition of UNS N08825. They are used in this study with the purpose of making comparison between the new Alloy 825 CTP and the standard material from Alloy UNS N08825. The annealing was performed in industrial furnaces unless otherwise stated. Heat D was cold rolled while the other heats were hot formed.

As the material is expected to sensitize when exposed to temperatures between approximately 600 °C (1112 °F) and 800 °C (1472 °F), the sensitization treatment for corrosion testing on Alloy 825 CTP was performed in laboratory furnaces. The sensitization temperature of 675 °C (1247 °F) was used and the soaking duration was varied from 4 to 16 hours. The material was placed inside the furnace after the furnace reached the heat treatment temperature and the time started to be counted immediately. After the desired time, the material was taken out of the furnace and the cooling down was done in air. Details of sensitization heat treatments are summarized in **Table 2**.

Table 2
Heat treatments for sensitization of the tested materials

PWHT	Sensitization Temperature, °C (°F)	Holding time, h
Ⅰ	675 (1247)	4
Ⅱ	675 (1247)	8
Ⅲ	675 (1274)	16

Mechanical Testing / Metallography

Tensile testing and microstructural inspection were carried out to verify the mechanical properties of the material and compare to the properties of the available heats of Alloy UNS N08825. The tensile properties of the new Alloy 825 CTP are expected to be in accordance to the required properties of Alloy UNS N08825.

Microstructure

Microstructural investigations were performed on mechanically polished and chemically etched specimens. For etching, a pickling solution containing 100 mL H_2O, 100 mL HCl, and 10 mL HNO_3 was used. Evaluation of the microstructure was performed using light optical microscopy techniques. The grain sizes were measured according to DIN/ISO 643-2013[12].

Tensile Testing

Tensile testing was conducted according to DIN(5)/ISO 6892-1[13] at room temperature and ISO 6892-2[14] for testing at elevated temperatures. Smooth specimens in transversal direction were machined and tested at room temperature, 175 °C (347 °F), and 205 °C (401 °F).

Corrosion testing

The corrosion testing program was established to meet the requirements of NACE MR0175 / ISO(6)15156-3[11] for qualification of Corrosion Resistant Alloys (CRAs) for H_2S-service, taking SSC, SCC and GHSC into account. Additionally, SSRT testing was performed to determine the susceptibility to hydrogen embrittlement. A third heat is being prepared and will be submitted to the same testing plan. Results of the third heat will be presented in NACE CORROSION 2019.

Pitting and crevice corrosion

Corrosion test according to ASTM G48 Method C was carried out to determine the critical pitting temperature (CTP) in acidified ferric chloride solution. ASTM G48 Method D was used to assess the critical crevice temperature (CCT) in the same solution.
Samples from Heat A (Alloy 825 CTP) were annealed at different temperatures and/or subjected to a PWHT with different soaking times. Four samples each were solution-annealed at 980 °C (1796 °F), 1010 °C (1850 °F) and 1040 °C (1904 °F). One sample of each annealing temperature was maintained on the annealed condition and the other three were submitted to a PWHT at 675 °C during 4, 8 or 18 hours. Specimens with dimensions of 50 x 50 x 5 mm were machined and the surface was grinded using 120-grit abrasive paper. Each sample condition was then tested according to the ASMT G48 Method C.

Sulfide Stress Cracking (SSC) Resistance

SSC testing according to NACE TM0177 Method A was performed at 24 °C ± 3 °C (75 °F ± 5 °F) on triplicate smooth round specimens with a gauge diameter of 6.35 mm (0.25 inch) and a gauge length of 25.4 mm (1 inch). The testing was carried out in Solution A saturated with 100 kPa (14.5 psi) H_2S, resulting in an initial pH of 2.7; the final pH was measured and was less than 4.0. Stress level of 90 % AYS was applied by deflection of the proof ring. Test duration was 720 h (30 d).

Galvanically Induced Hydrogen Stress Cracking (GHSC) Resistance

GHSC testing was performed in accordance with the previously stated conditions for SSC testing. In addition, the tested specimens were electrically coupled by platinum wire to carbon steel, which was fully immersed in the test solution.

Stress Corrosion Cracking (SCC) Resistance

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(5) German Institut for Standardization (DIN) e. V., Am DIN-Platz, Burggrafenstraße 6 10787 Berlin, Germany

(6) International Organization for Standardization (ISO), 7 ch. De la Voie-Creuse, Case Postale 56, Geneva, Switzerland

SCC testing was performed according to NACE TM0177 Method C (C-ring test). The material was tested under Level VI and Level VII test conditions as specified by NACE MR0175 / ISO 15156-3, Table E.1. SCC testing was conducted on triplicate C-ring specimens at 100 % of AYS at the test temperature. Four C-ring specimens were machined from each heat with an outer diameter (OD) of 40 mm (1.57 inch). For each set of four specimens, one C-ring was strain gauged to determine the necessary deflection corresponding to 100 % AYS at test temperature. The determined data was then utilized to deflect the tested triplicate set of specimens. SCC testing was carried out in autoclaves made of corrosion resistant material. After placing the specimens in the vessel, the test solution was added, so that all specimens were completely immersed in the liquid phase. Temperature was daily monitored. Separate specimens were used for 3 months testing at Level VI and 1 month testing at Level VII. After exposure to the corrosive environment, C-ring specimens were rinsed with distilled water and photographed. Examination for evidence of cracking was performed visually at 10x magnification.

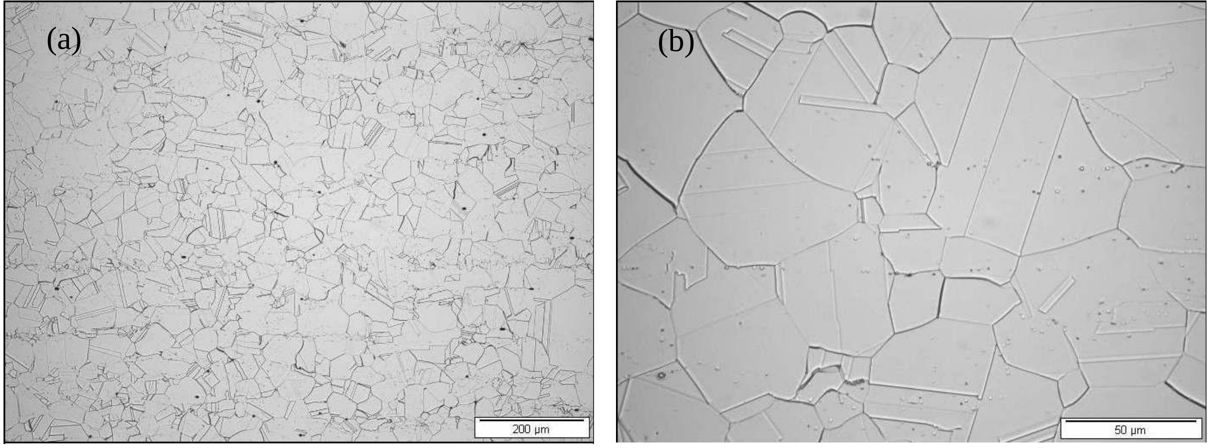
Hydrogen Embrittlement (HE) Resistance

Slow Strain Rate Tensile (SSRT) tests were used to determine the susceptibility to HE of the tested materials. Standard SSRT test specimens according to NACE TM0198-2016[15] with gauge section diameter of 3.81 mm (0.15 inch) and gauge section length of 25.4 mm (1 inch) were used. The tests were performed at 40 °C (104 °F). For each material, one specimen was tested in control environment (distilled water purged with nitrogen) and three specimens in aggressive environment (0.5 M sulfuric acid solution and applied cathodic current density of 5 mA cm^{-2}). The strain rate was 1x10^{-6} s^{-1} (crosshead speed: 2.5x10^{-5} mm s^{-1}).[22] Time-to-failure, reduction-of-area and elongation-to-failure are reported. After test end, all specimens were inspected on microscope at 20x magnification to determine the occurrence of secondary cracking.

RESULTS

Microstructure

In **Figure 1** the homogeneous microstructure of the two heats of Alloy 825 CTP can be seen. The heat A (**Figure 1** (a) and (b)) showed an average grain size of 37.8 µm (ASTM 6.5) according to DIN EN ISO 643-2013. Heat B (**Figure 1** (c) and (d)) showed middle grains with an average grain size of 53.4µm (ASTM 5.5) and small grains with a size of 22.1µm (ASTM 8).
The average grain size of Alloy 825 CTP was about 45.6 µm, the equivalent to an ASTM grain size number of 6, with a homogeneous microstructure, which can be seen in **Figure 1**.



Heat A: GS = ASTM 6.5 = 37.8µm

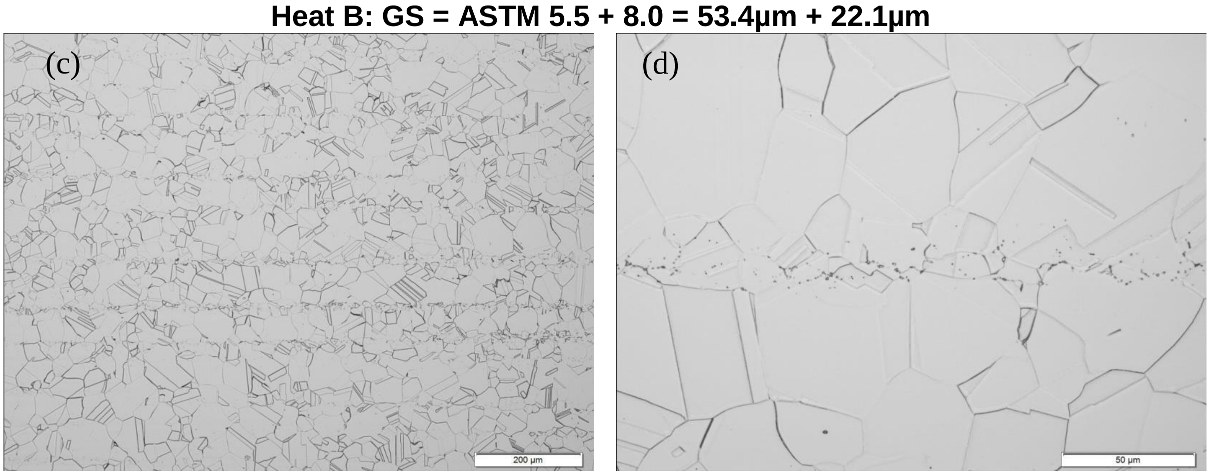

Heat B: GS = ASTM 5.5 + 8.0 = 53.4µm + 22.1µm

Figure 1: Microstructure of Alloy 825 CTP heat A (a) and (b), and heat B (c) and (d)

Mechanical testing

<u>Tensile Testing</u>

Figure 2 shows the average tensile properties at room temperature of the two heats of Alloy 825 CTP compared to the tensile properties of two heats of alloy UNS N08825. Each heat of Alloy 825 CTP was tested five times at the same conditions and the results show the average value of these five measurements.

The influence of the temperature on the tensile properties of the material was studied and the average tensile properties of the two heats are shown in **Figure 3** and **Figure 4**. As expected, the yield and tensile strengths decrease by increasing test temperatures. At the same time, elongation slightly increases while reduction of area slightly decreases.

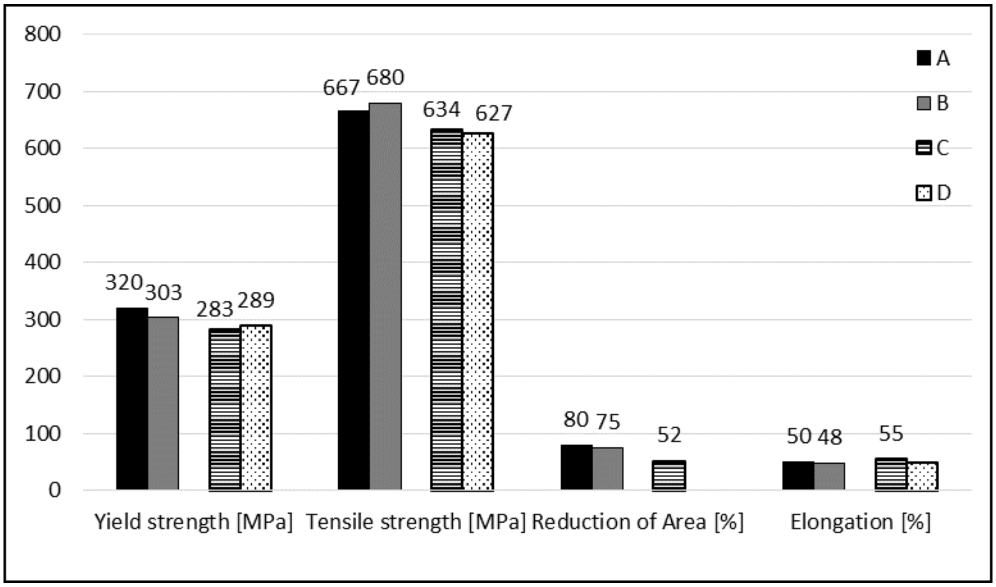


Figure 2: Yield strength, tensile strength, elongation and reduction of area of the heats of Alloy 825 CTP and UNS N08825 at room temperature

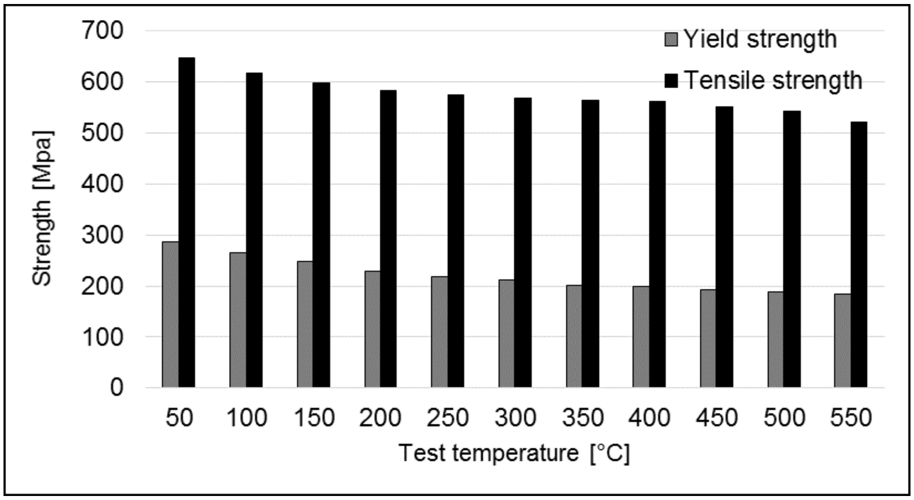


Figure 3: Average of yield and tensile strength of Alloy 825 CTP at different temperatures

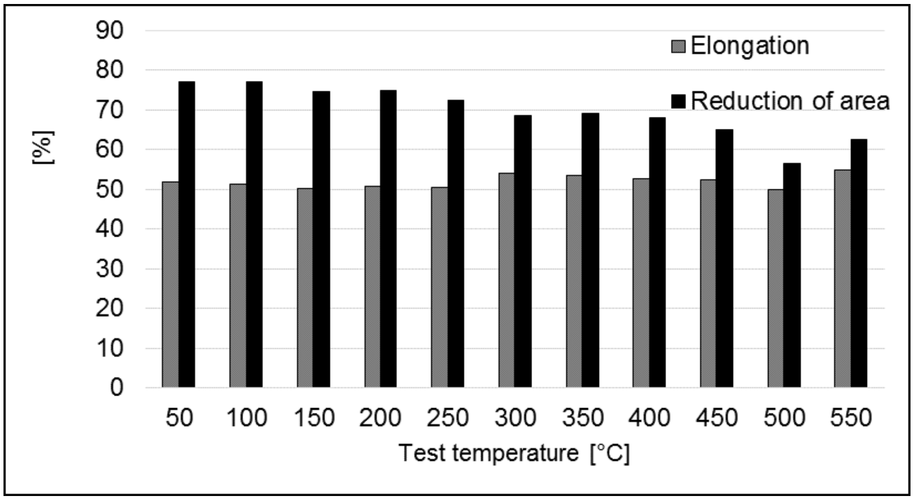


Figure 4: Average of elongation and reduction of area of Alloy 825 CTP at different temperatures

The mechanical properties of heats A, B, C and D are all listed on **Table 3**. The testing was performed at room temperature with specimens taken in the transversal direction. Compared to alloy UNS N08825, the strength of the advanced Alloy 825 CTP showed a slightly higher level. An increase in Reduction of Area could also be seen on Alloy 825 CTP.

Table 3
Mechanical properties of alloy UNS N08825 obtained from the heats D and E of the testing program

Heats	Material	Yield strength	Tensile strength	Reduction of Area	Elongation
		MPa	MPa	%	%
A	825 CTP	320	667	80	50
B	825 CTP	303	680	75	48
C	N08825	283	634	52	55
D	N08825	289	627	No data	49

Corrosion testing

ASTM G48 standard test methods were selected for the determination of the resistance of material Alloy 825 CTP to pitting and crevice corrosion when exposed to oxidizing chloride environments. Furthermore, corrosion testing was performed considering qualification requirements for SSC, GHSC and SCC as given by NACE MR0175 / ISO 15156-3. In case of solid-solution nickel alloys, SCC is stated as primary and both SSC and GHSC as secondary (possible) mechanisms. An overview of the corrosion test environmental conditions is given in **Table 5**.

Pitting and crevice corrosion

CTP and CCT were determined by means of ASTM G48 test methods C and D on samples of "as delivered" Alloy 825 CTP (in solution annealed condition, annealed at 1010 °C, 1850 °F) coming from standard production route (**Table 4)**. As evident from the results, increasing the molybdenum content results in improved CPT of around 50 °C (122 °F) compared with a CPT of 30 °C (95 °F) for the conventional alloy UNS N08825 in the standard production condition.

Table 4
Determined CPT and CCT on samples from heats A and B of Alloy 825 CTP in solution annealed condition

Material	CPT [°C]	CCT [°C]
Heat A	50	25
Heat B	55	15

The sensitization heat treatment was found to have a moderate influence on determined CPT. As the time of exposure to an intermediate temperature increases, the resistance to the formation of pitting decreases, as can be seen on the diagram of **Figure 5**, where the CTP gets slightly lower with the increase of exposure time to an intermediate temperature. Nevertheless, even after 16 hours of exposure to the intermediate temperature of 675 °C (1247 °F), the material still presents pitting resistance comparable or higher to the standard Alloy UNS N08825 on the "as delivered" condition.
Figure 5 summarizes the determined CPT of samples that were heat-treated in laboratory furnaces and later submitted to PWHTs.

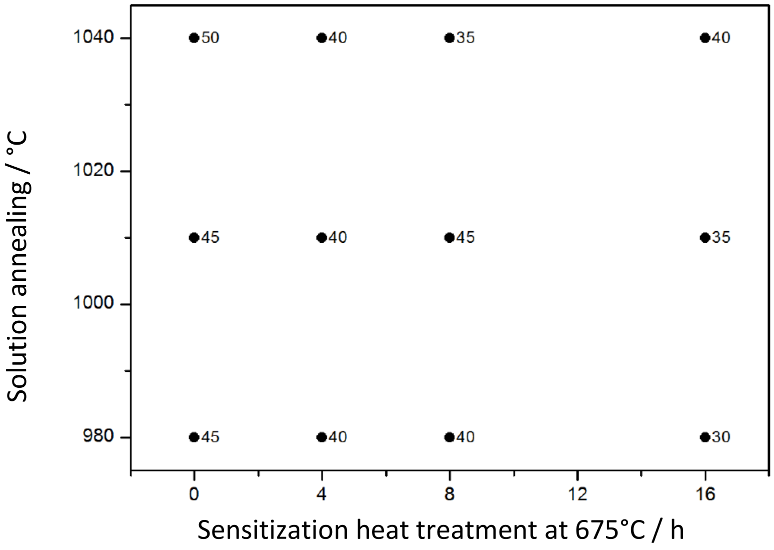


Figure 5: CPT values (°C) determined for heat treated samples of Alloy 825 CTP

Sulfide Stress Cracking (SSC) Resistance

SSC resistance of Alloy 825 CTP was evaluated using uniaxial tensile tests as given by NACE TM0177 Method A. No cracking or other defects were found in any of the tested heats after a duration of 30 days, so that all samples passed the tests (**Figure 6**).

Galvanically Induced Hydrogen Stress Cracking (GHSC) Resistance

GHSC testing was conducted using the same conditions and specimen geometry as for SSC testing, but with galvanic coupling to C-Steel. Because of the different potentials of the metals, a galvanic effect is established and may lead to accelerated cracking process of the tested CRA. However, in case of Alloy 825 CTP no cracking or other defects were observed on the specimens for the test duration of 30 days (**Figure 6**).

Stress Corrosion Cracking (SCC) Resistance

SCC resistance was evaluated by using NACE TM0177 Method C. The tests were performed on C-ring specimens at environmental conditions corresponding to Level VI for 90 days and Level VII for 30 days. For each of the heats from Alloy 825 CTP, three C-Ring specimens were exposed after applying a stress corresponding to 100 % of AYS at the test temperature. After test end, all tested specimens were cleaned. No evidence of cracks or other defects were observed (**Figure 6**).

Table 5
Corrosion test environmental conditions for Alloy 825 CTP

Cracking mechanism	Specimen type	Specimens per heat	Duration / d	T / °C	H_2S /kPa	CO_2 / kPa	pH End	Cl^- / mg L^{-1}	S^0 / mg L^{-1}	Σ / % AYS	Galvanic coupling	Pass / Fail
SSC	*	3	30	24	100	-	2.8 / 2.9	Test solution A***	-	90	No	P
GHSC	*	3	30	24	100	-	3.6 / 4		-	90	Yes	P
SCC	**	3	90	175	3,500	3,500	-	139,000	-	90	No	P
	**	3	30	205	3,500	3,500	-	180,000	-	90	no	P

* acc. NACE TM0177 Method A (round bar tensile specimen)
** acc. NACE TM0177 Method C (C-ring specimen)
*** acc. NACE TM0177

Figure 6: SSC and GHSC specimens after test according to NACE TM0177 Method A, for 30 days; SCC specimens according to NACE TM0177 Method C at Level VII condition for 30 days

Hydrogen Embrittlement (HE) Resistance

The susceptibility to HE of the new Alloy 825 CTP was evaluated by means of SSR testing under cathodic polarization. From the comparison of the ductility parameters determined in the aggressive environment with those determined in inert environment, elongation-to-failure and reduction of area ratios were calculated and are shown in **Table 6**. Values of elongation-to-failure ratio near 100% generally indicate that the material suffers no influence from the aggressive testing medium. Moreover, 45% of elongation-to-failure ratio is regarded as a set threshold for classifying alloys with respect to their hydrogen embrittlement susceptibility[23]. The both tested heats presented elongation-to-failure ratios far above the 45% and close to the 100%, assuming an excellent resistance to HE.

Table 6
Test results of SSR test under cathodic polarization for Alloy 825 CTP

Heat designation	Heat Treatment	Environment	RA		E		TTF		UTS	
			%	RAR [%]	%	EFR [%]	h	TTFR [%]	kN	UTSR [%]
Heat A	hot-rolled and soft-annealed at 1010 °C	Inert	81.8	-	40.8	-	143	-	6.5	-
		Aggressive	78.1	95.5	38.5	94.4	131	91.5	6.3	97.2
		Aggressive	79.4	97.1	36.8	90.2	119	94.5	6.4	98.3
		Aggressive	78.9	96.5	38.2	93.6	130	90.9	6.1	94.7
Heat B	hot-rolled and soft-annealed at 1010 °C	Inert	80.1	-	38.4	-	127	-	6.7	-
		Aggressive	80.9	101	40.2	104.7	141	110.7	6.9	103.0
		Aggressive	84.6	105.6	43.2	112.5	145	114.1	6.6	98.5
		Aggressive	79.7	99.5	39.4	102.6	138	108.1	6.6	98.5

SSR tests to determine the susceptibility to hydrogen embrittlement were carried out also on samples from conventional alloy UNS N08825 (**Table 7**). Based on ductility parameters, no significant differences in the hydrogen embrittlement behavior between conventional alloy UNS N08825 and improved Alloy 825 CTP was detected. It is worth noting that during SSR tests higher strength levels were observed for Alloy 825 CTP compared to alloy UNS N08825, which is in line with tensile testing results mentioned above. No ranking of HE resistance between these materials was possible. For this reason, a future work including the development of (more severe) test environments allowing ranking is planned.

Table 7
Test results of SSR test under cathodic polarization for alloy UNS N08825

Heat designation	Heat Treatment	Environment	RA		E		TTF		UTS	
			%	RAR [%]	%	EFR [%]	h	TTFR [%]	kN	UTSR [%]
Heat E	hot-rolled and soft-annealed at 1010 °C	Inert	78.1	-	42.3	-	141	-	6.0	-
		Aggressive	73.5	94.1	41.8	98.8	140	99.4	6.1	101.7
		Aggressive	75.3	96.4	43.9	103.8	145	102.7	6.1	101.7
		Aggressive	81.1	103.8	42.7	100.9	144	102.5	6.1	101.7

CONCLUSIONS

- Tensile and yield strength of Alloy 825 CTP are higher than of alloy UNS N08825. Other mechanical properties, e.g. Charpy impact and hardness were found to be comparable.

- Improved chemical composition of Alloy 825 CTP enhanced its localized corrosion resistance as compared to alloy UNS N0825. Consequently, CPT and CCT obtained from corrosion tests were significantly increased.

- Heat treatment of Alloy 825 CTP showed moderate influence on the pitting corrosion resistance of the material as shown by corrosion test according to ASTM G48-C. Under the most unfavorable heat treatment conditions the advanced Alloy 825 CTP showed pitting resistance comparable to that of non-heat treated conventional alloy UNS N08825.

- Alloy 825 CTP showed to be sensitized when heat treated at 675 °C (1247 °F). Nevertheless, even after 16 hours of exposure to this intermediate temperature, the material still presents pitting resistance comparable or higher to the standard “as delivered” Alloy UNS N08825.

- Advanced Alloy 825 CTP with higher Molybdenum content showed high resistance against environmental-assisted cracking (SSC, GHSC, SCC).

- Alloy 825 CTP did not show any indications for increased susceptibility to hydrogen embrittlement.

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