

Characterization of novel high performance material UNS N08034

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ABSTRACT

The changing material demands in the chemical processing and related industries calls for new and more performing materials. UNS N08034 is a new super-austenitic stainless steel alloyed with about 27 % chromium and 6.5 %-molybdenum. Further it has an intended addition of nitrogen for austenite stability and strength.

The aim of this work is to present a comprehensive characterization of this material, which is the best in class in the corresponding alloy group, and therefore of high interest for applications in severe corrosive environments of the process industry. Along with the main material properties such as chemical composition, microstructure, mechanical properties, thermal stability, corrosion resistance and fabricability, several examples of application will be described. To approach the influence of fabrication on the material properties base materials studies are exemplarily complemented with results from welded and from clad samples.

Key words: UNS N08034, 2.4692, UNS N08031, NiCrMoFe alloy, 6-Mo steel, mechanical properties, corrosion resistance, fabricability, sensitization, weldments, cladding

MOTIVATION FOR THE DEVELOPMENT OF UNS N08034

The new alloy UNS N08034 is a further improvement of alloy UNS N08031. Its precursor UNS N08031 is a high-chromium alloyed 6 % molybdenum steel and has been successfully used in a wide range of applications for over twenty years now, which has also been reported in several places.^{1,2,3} Main applications are equipment in Chemical Process Industry (CPI) where corrosion resistance is required to mineral acids like sulfuric- and phosphoric acids.

Common for the alloys of the group of austenitic 6-Mo-steels is, that they are prone to precipitate intermetallic phases as sigma phase in particular in a temperature range of 950 °C to 1050 °C for example during hot forming or heat treatments. The sigma phase is a brittle intermetallic phase containing chromium, molybdenum, nickel and iron and needs to be avoided to ensure full corrosion behavior as well as mechanical properties of this kind of wet corrosion materials. To dissolve precipitated sigma phase for example after hot forming, solution annealing followed by quenching is required.

UNS N08031 requires a solution annealing treatment at about 1180 °C followed by fast cooling, which is not easy to perform in every place of manufacturing.⁴ Further, hot roll cladding was not technologically feasible for this alloy. Its outstanding performance lead to the demand of a material with improved handling for cladding while maintaining the excellent corrosion behavior at the same time. In order to achieve this, the chemical composition has been optimized, supported by the use of phase calculation programs and resulted in the new member of the 6-Mo-steels: alloy UNS N08034.^{3,4,5,6}

CHARACTERIZATION OF UNS N08034

Chemical Composition

The nominal compositions of UNS N08034 (last line), its precursor UNS N08031 as well as these of further reference alloys UNS N08926 and UNS N06625 with regard to comparative corrosion investigations are shown in table 1. UNS N08034 is alloyed with 27 % chromium and about 6.5 % molybdenum which is similar to the contents of UNS N08031 regarding these elements. Adaptions are made by the following: the Ni-content has been increased from 31 % to 34 % and a balanced addition of manganese and nitrogen has been conducted to stabilize the austenite microstructure.^{3,4,5,6}

The reference alloy UNS N08926 also belongs to the family of 6-Mo stainless steels and is alloyed with nitrogen as well. But its nickel and chromium contents are lower compared to both alloys mentioned before. Finally, the comparison with the nickel-based material UNS N06625 shows a higher molybdenum content, whereas the chromium content of 23 % is only moderately high. Moreover, the high nickel content supports austenite stability, but on the other hand it also determines the material costs.^{5,7}

Table 1: Nominal composition of UNS N08034 and reference alloys

Designation		Main alloying elements, nominal composition, % by mass				
UNS No.	EN No.	Ni	Cr	Mo	Fe	Other
N06625	2.4856	62	23	9	bal.	Nb
N08926	1.4529	25	21	6.5	bal.	Cu, N
N08031	1.4562	31	27	6.5	bal.	Cu, N
N08034	2.4692	34	27	6.5	bal.	Cu, N

Mechanical Properties

Table 2 shows the minimum mechanical properties for UNS N08034, which are valid for plates (≤ 30 mm in thickness) in the solution annealed condition. These requirements are approved by TÜV and published in the VdTÜV-data sheet 583/1⁸ (german pressure vessel code). The minimum values defined in there are the same as for UNS N08031 (VdTÜV-data sheet 509/1⁹). The last column represents measured values in the solution annealed condition on transverse samples of plates of 8 mm and 25.4 mm thickness for the specified temperatures. The measured values surpass the aforementioned minimum.

Table 2: Minimum mechanical properties for solution annealed alloy UNS N08034 according to VdTÜV-material data sheet 583/1⁸.

Mechanical Property	Temperature (°C)	Minimum Values	Measured Values	
			Plate, 8 mm	Plate, 25.4 mm
Yield Strength $R_{p0.2}$ (MPa)	RT	≥ 280	333 - 342	327 - 336
	500	≥ 135	198	193
Tensile Strength R_m (MPa)	RT	650 to 850	708 - 713	717 - 725
Elongation A (%)	RT	≥ 40	61.0 – 67.1	59.7 – 64.4
V Notch bar impact energy (J)	RT	≥ 150	*142 - 144	262 - 276
	-196	≥ 110	*121	204

*The specified minimum values of the V notch bar impact energy only apply to standard specimens according to DIN EN ISO 148-1¹⁰. Otherwise the minimum value shall be reduced linearly to the cross-section of the specimen in the notch¹¹ which is the case for the 8 mm thick material.

The influence of cold forming on mechanical properties has also been investigated within the TÜV approval. Therefore the plate with a thickness of 25.4 mm was examined. The plate was cold rolled with two different degrees of deformation, 10 % and 15 % which was calculated by the change in plate thickness. As expected, the values of the yield strength and of the tensile strength increase with increasing degree of deformation while the elongation decreases. But even after 15 % cold forming the requirements at RT of the VdTÜV-data sheet are met. UNS N08034 is covered in the following ASTM specifications: ASTM B564-17a (Forging)¹², B581-17 (Rod)¹³, B619/B619M-17a (Pipe)¹⁴, B622-17a (Seamless Pipe and Tube)¹⁵, B625-17 (Plate, Sheet, Strip)¹⁶, B626-17a (Welded Tube)¹⁷ and B649-17 (Bar and Wire)¹⁸.

Thermal Stability

To ensure corrosion resistance as well as mechanical properties of high-alloyed special stainless steels the temperature control during any annealing as well as fabrication procedure has to be designed to suit the material.⁷ Otherwise precipitates are built, for example sigma phase or carbides in the case of UNS N08034 and UNS N08031. Investigations of the time-temperature precipitation (TTP) and time-temperature sensitization (TTS) behavior of UNS N08034 and UNS N08031 have been conducted in order to compare both materials in that matter.

Time-Temperature-Precipitation Diagrams

To establish TTP diagrams of UNS N0834 and UNS N08031 samples of each material have been annealed for periods ranging from a few minutes to 100 hours at temperatures between 550 °C and 1150 °C. Afterwards the precipitation behavior was determined through optical microscope. The symbols in figure 1 describe the progress of the respective precipitation process in the time-temperature precipitation diagrams shown for UNS N08034 in figure 2 and for UNS N08031 in figure 3.

The precipitates at grain boundaries are presented with black symbols and precipitates within the grains with blue symbols.



Figure 1: Symbols used to show the precipitation process in the time-temperature precipitation diagrams for UNS N08034 and UNS N08031

Distinction has to be made between the sigma phase precipitation in the upper temperature range and the precipitation of carbides in the lower temperature range. At temperature below 700 °C the precipitates can be identified as carbides. As to be seen in the diagram for UNS N08034 in figure 2 precipitation starts at grain boundaries. In the temperature range of 950 °C – 1050 °C the building of sigma phase precipitates occurs after about ten minutes annealing time. The blue signs in the diagram highlight that after longer annealing times, additional precipitates are present within the grain. The first visible precipitations within the grain are identified after nearly 5 hours in the temperature range of 850 °C and 950 °C. Compared to that the precursor alloy UNS N08031 shows the first increased precipitations within the grains in the temperature range of 950 °C and 1000 °C at one hour annealing, which is significantly earlier (figure 3).

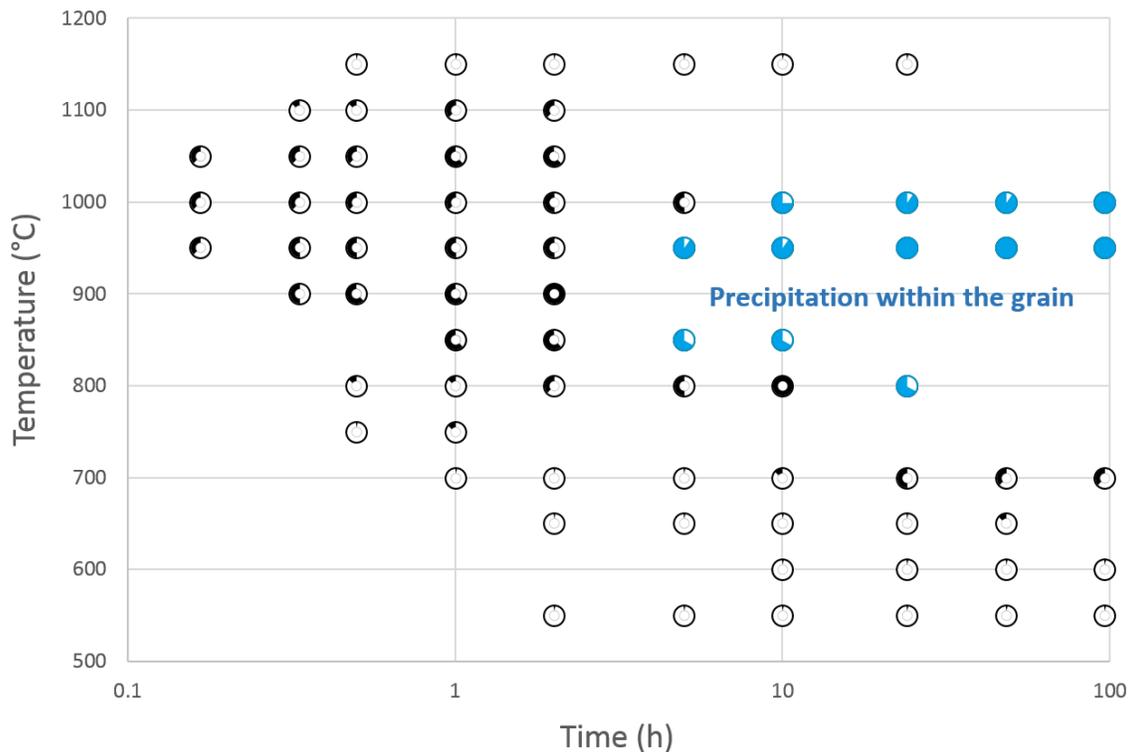


Figure 2: Time-Temperature-Precipitation diagram of UNS N08034 (metallographic investigation)

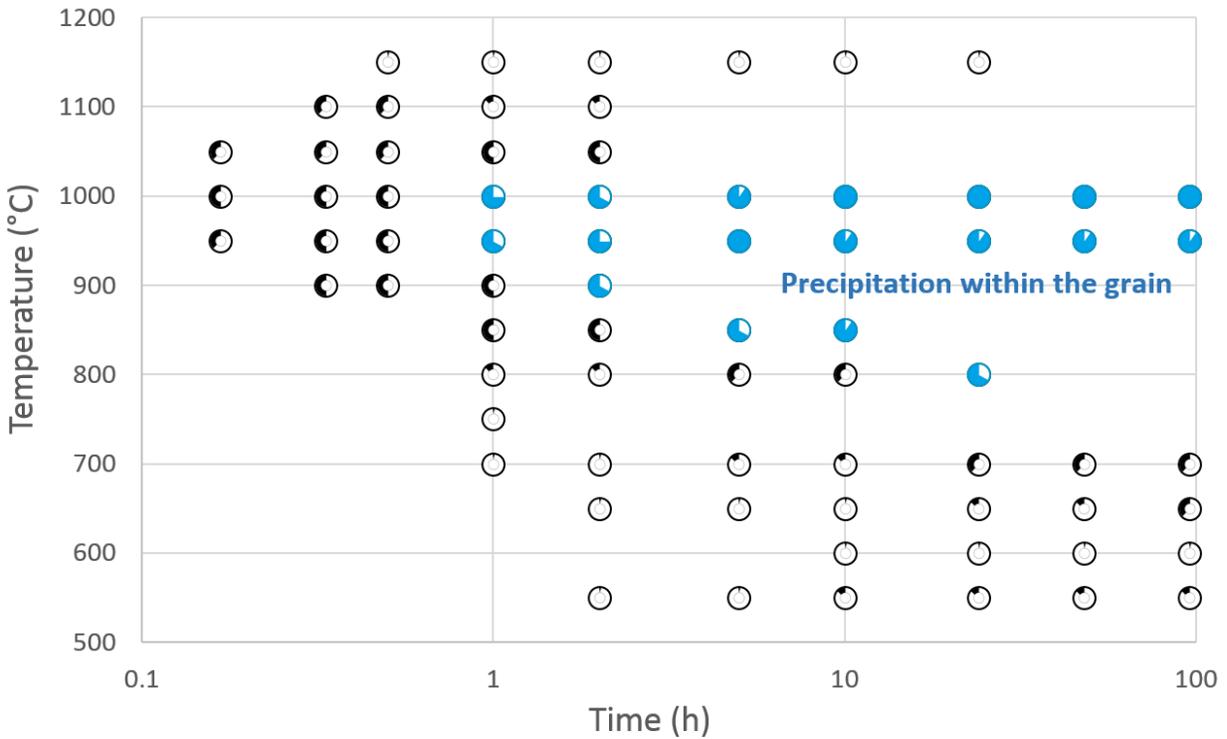


Figure 3: Time-Temperature-Precipitation diagram of UNS N08031 (metallographic investigation)

Also exemplary pictures of the microstructure shown in figure 4 confirm the slower sigma-phase precipitation kinetics of UNS N08034. The progress of sigma phase precipitations at 1000 °C for both alloys are presented at different annealing times, above for UNS N08034 and below for UNS N08031. It is obvious that the grain boundaries for UNS N08034 are less covered than for UNS N08031 at all times. At an annealing time of one hour increased precipitates within the grains are visible for UNS N08031 whereas not for UNS N08034.

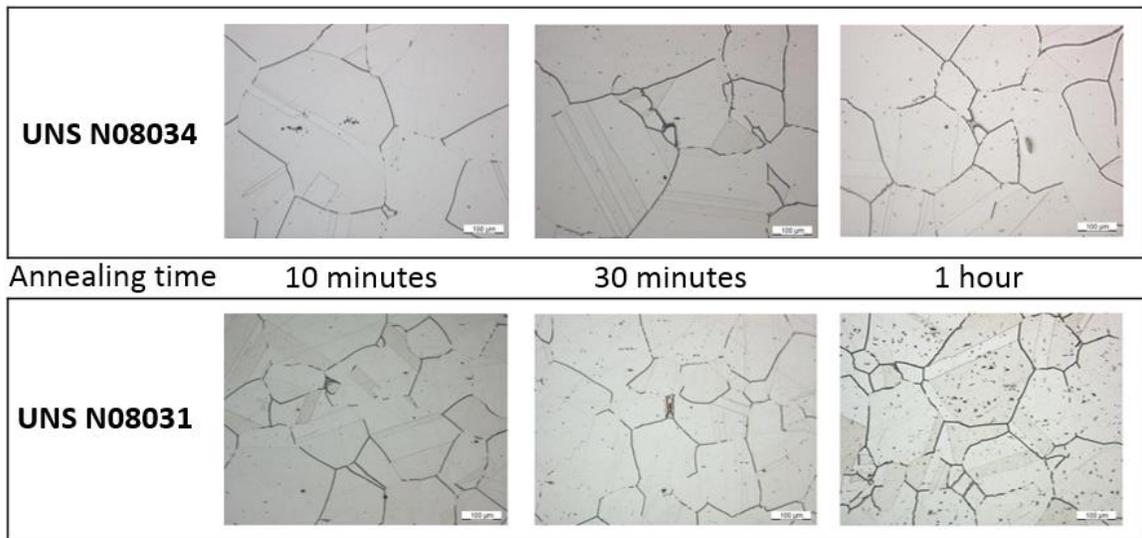


Figure 4: Metallographic examination on UNS N08034 and UNS N08031 after annealing at 1000 °C at different times

Sensitization Behavior

Precipitations, such as carbides, nitrides and sigma phase, may lead to chromium depletion in the grain boundary area which results in preferred corrosion attack of the grain boundaries in oxidizing media. This is called sensitization.⁷ A common test for the evaluation of sensitization degree with regard to intergranular corrosion (IC) for alloys high in chromium is ASTM G28, method A.¹⁹ It is a standardized test in boiling 50 % sulfuric acid (p.a. grade) with an addition of 42 g/l $\text{Fe}_2(\text{SO}_4)_3 \times 9 \text{H}_2\text{O}$ in the solution. The testing time for alloys high in chromium shall be 120 hours²⁰. The common criterion for susceptibility to intergranular corrosion is the penetration depth of 50 μm . Investigations on UNS N08034 show that sensitization in the sense of the before mentioned criteria occurs first in the temperature range of about 700 °C at an annealing time of about two hours. Heubner et al.⁷ plotted a time-temperature-sensitization diagram for UNS N08031 determined under same conditions (ASTM G28, method A²⁰, IC penetration depth > 50 μm) which show that sensitization starts at an annealing time of about two hours and in the temperature range of about 650 °C to 700 °C. This means that the sensitization behavior in respect to intercrystalline corrosion is nearly the same for both materials. The low temperatures suggest that sensitization in the present case is the result of carbide precipitation and not of intermetallic sigma phase.⁷

Charpy Impact Test:

As mentioned elsewhere^{5,21} to evaluate the effect caused by sigma phase, which can form at higher temperatures, it is advisable to conduct tests to determine for example pitting resistance or notch impact toughness instead of a test on intercrystalline corrosion. Specimens of UNS N08034 and UNS N08031 have been tested according to DIN EN ISO 148-1¹⁰ with ISO-V-specimens after sensitization annealing. The tests have been done at -60 °C on transverse samples. The value measured in the solution annealed condition is about 255 J for UNS N08034. As seen in figure 5 the sigma phase precipitation is detrimental to the ductility of the materials. At a constant temperature of 1000 °C the notch impact energy decreases for both materials with increasing annealing time, which correlates with increasing sigma phase precipitation as showed before. For UNS N08031 the loss in notch impact energy is clearly more remarkable.⁵

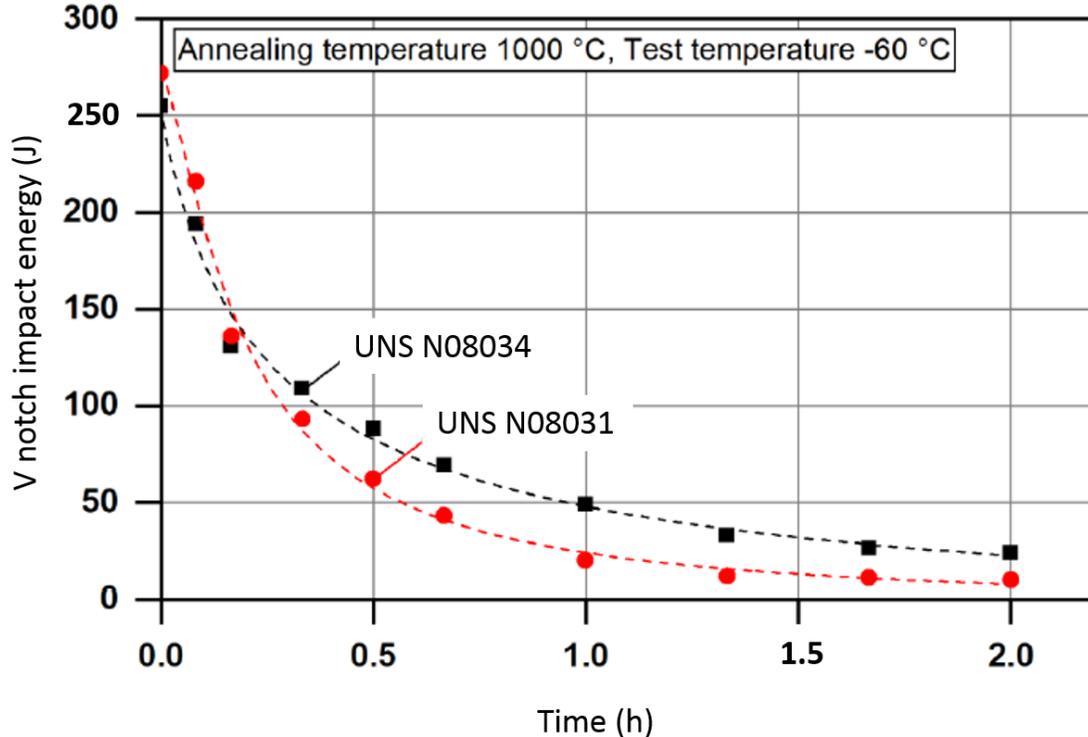


Figure 5: V Notch impact energy of UNS N08034 and UNS N08031 after annealing at 1000 °C at different times

In summary, sensitization behavior due to sigma-phase formation is significantly reduced for UNS N08034. Precipitation of sigma phase needs to be always avoided to achieve optimal performance of this wet corrosion material. However, if formed it can be dissolved by solution annealing followed by quenching. The recommended temperature range for solution annealing of the new material UNS N08034 is 1140 °C – 1170 °C, which is lower than for UNS N08031.^{1,3,4,5,6}

Corrosion Resistance

Localized Corrosion

A first hint regarding resistance to localized corrosion is given by the empirical calculation of the so called Pitting Resistance Equivalent (PRE) Number which is determined from the alloys composition by the following equation: $PRE = \%Cr + 3.3 \% Mo + 30 \%N$. Using this equation different alloys can be ranked. The higher the PRE value the higher the resistance to the testing corrosion media.² Figure 6 represents the Critical Pitting Temperature (CPT) and Critical Crevice Temperature (CCT) of common stainless steels and nickel-alloys as a function of their PRE number.

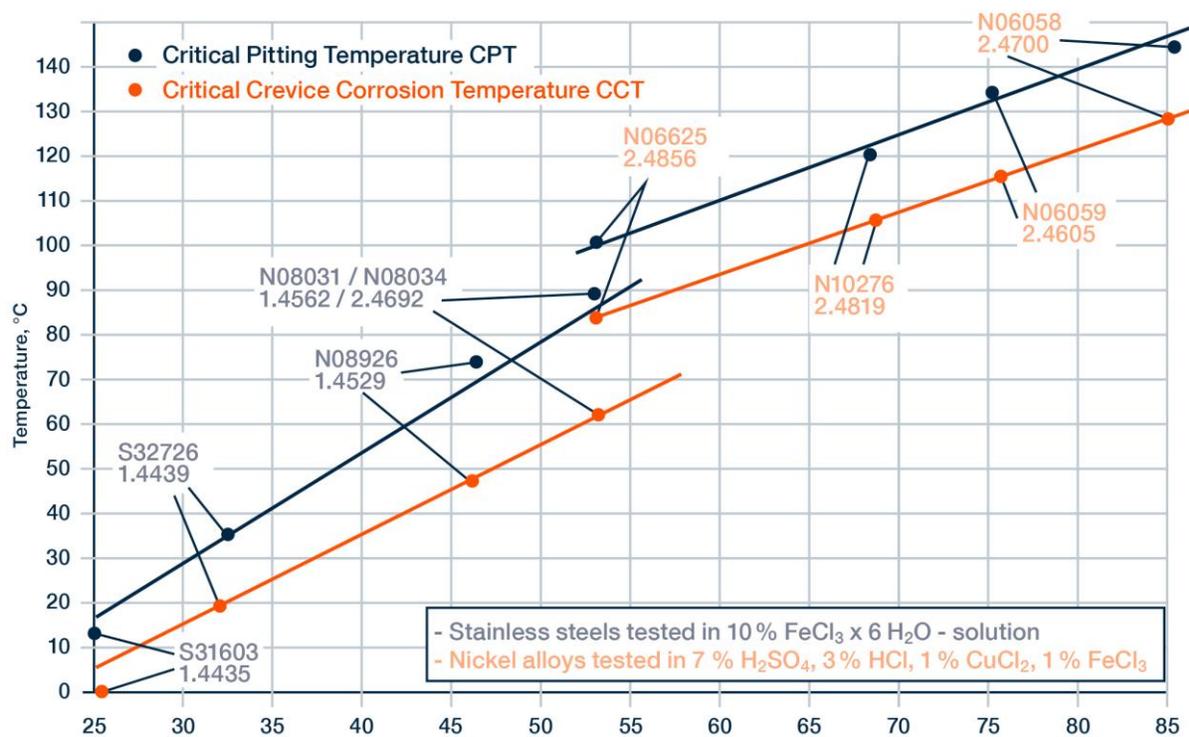


Figure 6: Critical Pitting Temperature (CPT) and Critical Crevice Temperature (CCT) of stainless steels and nickel-alloys as a function of the Pitting Resistance Equivalent (PRE)²²

For stainless steels these values have been determined in 10 % FeCl₃ x 6 H₂O solution whereas the alloys with a high nickel content have been tested in a more aggressive test solution called “green death”. The green death solution contains 7 % H₂SO₄, 3 % HCl, 1 % CuCl₂, 1 % FeCl₃ which simulates acidic condensates of the kind that occur in the flue gas ducts of coal fired power stations.² UNS N08034 has a PRE value of around 54. A very good local corrosion resistance has been shown under the tested conditions.

Hydrochloric acid

Immersion tests in hydrochloric acid have been conducted at three different acid concentrations (table 3). At the lowest concentration of 5 % at 50 °C UNS N08034 and UNS N08031 show a significant lower corrosion attack compared to UNS N08926. At higher concentrations of 10 % and 20 % the corrosion losses of all three materials are similar.

Table 3: Corrosion behavior in 24 hours immersion tests in hydrochloric acid (analytical grade)⁵

Test medium	Temperature (°C)	UNS N08034 Corrosion loss (mm/y)	UNS N08031 Corrosion loss (mm/y)	UNS N08926 Corrosion loss (mm/y)
Hydrochloric acid 5%	50	< 0.01	< 0.01	1.3
Hydrochloric acid 10%	25	0.58	0.63	0.62
Hydrochloric acid 20%	25	0.39	0.42	0.42

Sulfuric acid

Immersion tests have been conducted in boiling sulfuric acid of different concentrations. UNS N08034 and UNS N08031 show similar corrosion losses whereas UNS N08926 shows in comparison a corrosion loss twice as much in 5 % and even more than twice as much in 10 % sulfuric acid (table 4).

Table 4: Results of immersion tests in sulfuric acid⁵

Test medium	Temperature (°C)	UNS N08034 Corrosion loss (mm/y)	UNS N08031 Corrosion loss (mm/y)	UNS N08926 Corrosion loss (mm/y)
Sulfuric acid 5%	boiling	0.32	0.36	0.68
Sulfuric acid 10%	boiling	0.58	0.59	1.6

More tests have been conducted in 50 % analytical grade (p.a.) sulfuric acid with or without the addition of an oxidant. The corrosion losses after 24 hours immersion tests are presented in figure 7. The laboratory tests show that the corrosion behavior of alloy UNS N08034 and UNS N08031 in sulphuric acid depends on acid temperature as well as the presence of oxidants. While at 70 °C nearly no corrosion loss could be measured for both materials, at an increased temperature of 90 °C both materials have been corrosively attacked. By adding a small amount of oxidants (5.6 mg/l Fe³⁺) the passivity range can significantly widen under the same conditions which resulted in negligible mass loss for both alloys.

In conclusion, the corrosion behavior of UNS N08034 as well as UNS N08031 is strongly dependent on the oxidizing potential of the sulfuric acid. It has been extensively described that pure, reagent grade sulfuric acid can be much more aggressive than technical grade acid for UNS N08031.^{1,2,23,24} More investigations are ongoing for UNS N08034 in this field.

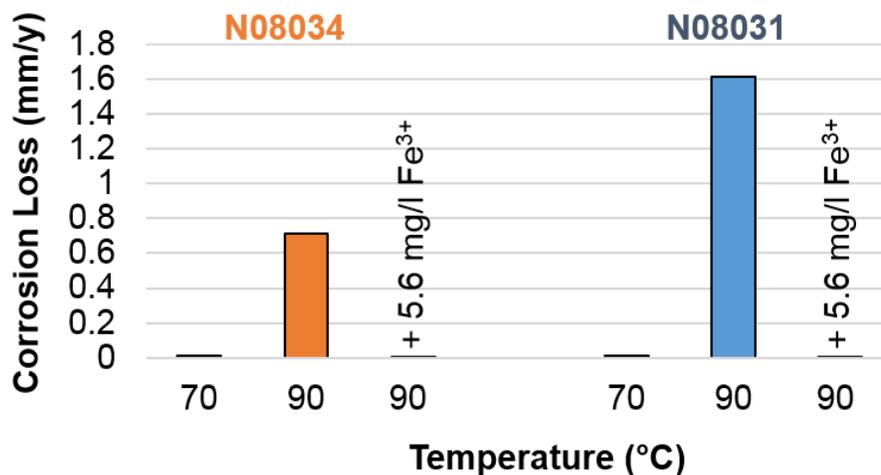


Figure 7: Results of 120 hours immersion tests in 50 % sulfuric acid, analytical grade, with and without additions of ferric ions as an oxidant.

Phosphoric acid

Phosphoric acid plays an important role for the fertilizer production. Alloys UNS N08034, UNS N08031, UNS N08926 and UNS N06625 have been tested in phosphoric acid under different conditions as to be seen in figure 8. The acid concentration, acid temperature as well as additions of chlorides have been varied. The samples were immersed for 120 hours in the test solution and the corrosion losses have been determined by weight loss measurements afterwards.

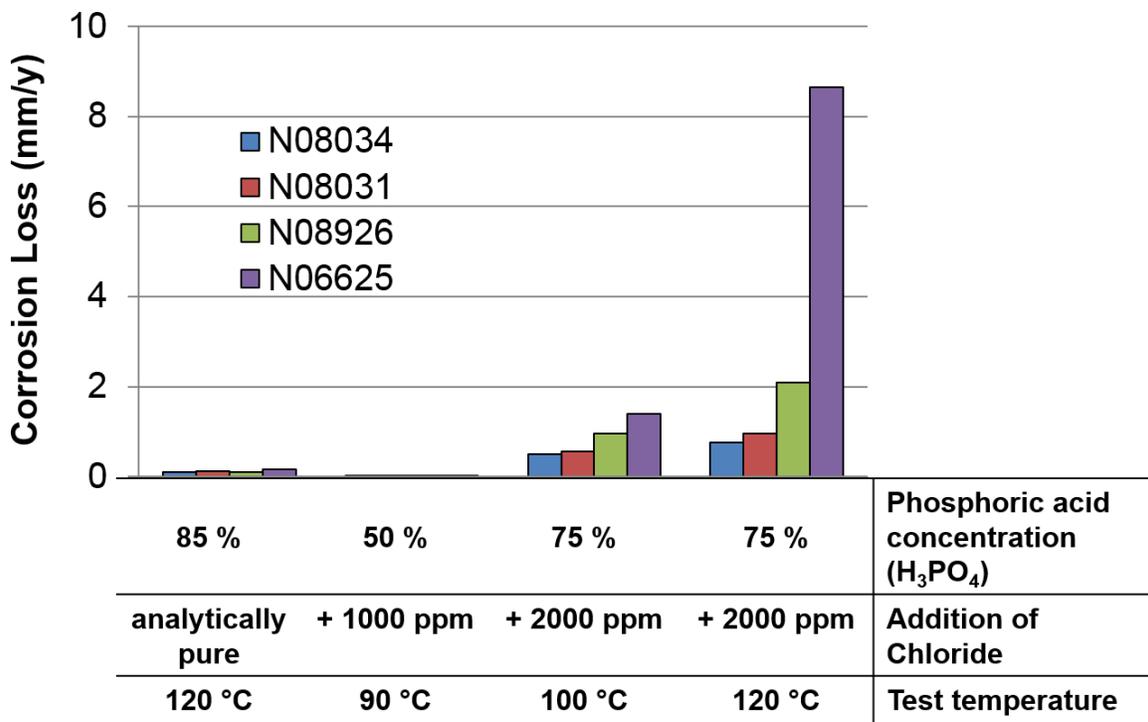


Figure 8: Results of 120 hours immersion tests in phosphoric acid

The new alloy UNS N08034 shows the lowest corrosion loss compared to that of the other alloys tested under the entire test range. Under the most aggressive tested conditions (last column), UNS N06625 with higher nickel and molybdenum but lower chromium contents compared to UNS N08034 (table 1), shows nearly ten times higher corrosion loss. UNS N08926, member of 6-Mo stainless steel, shows also significantly higher corrosion losses under the more aggressive conditions compared to UNS N08034. The results confirm that a higher level of chromium is preferable for resistance to corrosion by phosphoric acid at higher temperatures. Additionally, results have been reported elsewhere⁴ and confirm the exceptional resistance of UNS N08034 in phosphoric acid.

Stress corrosion cracking

The stress corrosion cracking resistance of UNS N08034 and UNS N08031 in calcium chloride solution has been investigated. Round tensile specimens were produced according to NACE TM0177 method A. The stress corrosion cracking tests were carried out in a 50 % calcium chloride solution at a temperature of 130 °C under constant load. Specimens of each material were tested at different loads up to 90 % of the yield strength at the elevated temperature of 130 °C. None of the samples failed due to stress corrosion cracking even under the most severe conditions (90 % load and 1000 h testing time). The additional observation under the stereomicroscope showed no signs of possible local corrosion attack or cracking. Overall, it should be noted that both materials proved to be resistant to stress corrosion cracking under the tested conditions.

Assessment of weldability and corrosion resistance of weld joints

The assessment of UNS N08034 concerning its general weldability and the corrosion resistance of weld joints are essential for its practical use. In this connection the UNS N08034 was subjected to a comprehensive test series within the scope of the required approval procedures. Amongst others, different weld procedures like tungsten inert gas (TIG) and gas metal arc welding (GMAW), which includes metal inert gas (MIG) respectively metal active gas (MAG), had been applied in correlation with different material thicknesses ranging from 0.7 mm (strip) up to 25.4 mm (plate). In all cases besides autogenous welding, the widespread and well-known nickel base filler metal AWS A5.14 - ERNiCrMo-13²⁵ was used. The corrosion testing was focused on the “as-weld” condition without any solution annealing or post weld heat treatment (PWHT).

The corrosion resistance of the weld joints was investigated according to SEP 1877 – II²⁶ (comparable to ISO 3651-2 proc. C²⁷) and ASTM G28, method A²⁰. Both corrosion tests are similar concerning their test solutions (different sulfuric acid concentration), but for SEP 1877 – II²⁶, the welded sample must be bent over a mandrel after the immersion time for a qualitative assessment of the intergranular corrosion resistance. The weldings were carried out with UNS N08034 plates of 5 mm thickness and 70° edge beveling. The plates were manual TIG (141) welded with rods of 1.6 mm and 3.2 mm diameter and a shielding gas of argon + 2 % hydrogen. The welded plates were cut to coupons of the size 40 mm x 20 mm for SEP 1877 – II²⁶ and 100 mm x 100 mm for ASTM G28, method A²⁰.

The evaluation of the corrosion tests for the SEP 1877 – II²⁶ test (10 x magnification of the surface after bending) shows no hints of intergranular attack. The average corrosion rate was determined to 0.19 mm/y. According to ASTM G28, method A²⁰, the average corrosion rate of welded UNS N08034 was determined to 0.39 mm/y. This value is in the range or even slightly below of the unwelded base material. The heat affected zone did not sensitize. It can thus be stated that both tests, the SEP 1877- II²⁶ and the ASTM G28, method A²⁰, prove the resistance to intergranular corrosion of weld joints of UNS N08034 with ERNiCrMo-13²⁵.

Fabrication

Hot roll cladding

Due to the improved thermal stability the hot-roll cladding of UNS N08034 is technically feasible. Recent experiences have been reported elsewhere^{1,6} and highlighted that UNS N08034 roll-bond cladded on carbon steel resulted in a homogenous microstructure of UNS N08034 without sigma phase precipitations. Further corrosion tests according to ASTM G28, method A²⁰, showed no signs of intercrystalline corrosion on UNS N08034 and a low corrosion rate. Its forerunner UNS N08031 is not feasible to be hot roll bonded.

Tube welding

The higher thermal stability and lower solution annealing temperature of UNS N08034 is also advantageous in the production of longitudinally welded tubes. In figure 9 the microscopic micrograph of a TIG weld UNS N08034 is presented on the left side. On the right side a picture showing an overview over a TIG welded UNS N08034 tube is illustrated.²⁸

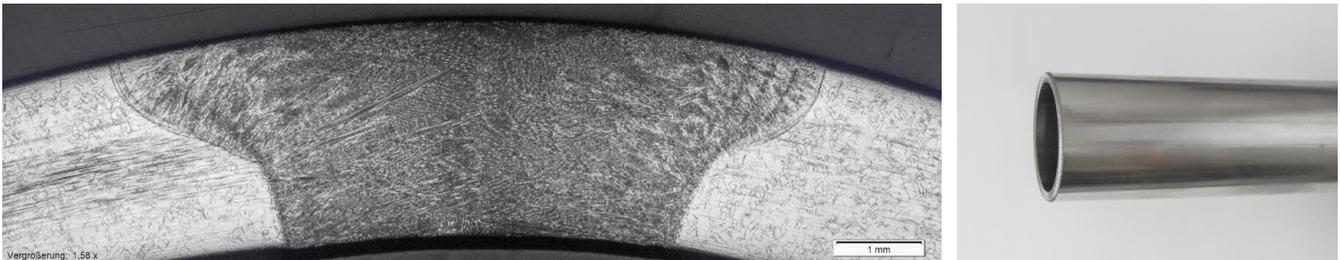


Figure 9: Microscopic micrograph of a TIG weld UNS N08034 (left) and an overview picture of a welded tube UNS N08034 (right)²⁸

Application

The high degree of versatility offered by the high resistance to corrosion in a multitude of different media of alloy UNS N08034 has resulted in its use in a number of demanding applications. Exemplary a crystallizer made of UNS N08034 is shown in figure 10. UNS N08034 is easily fabricated and welded.



Figure 10: Crystallizer made of UNS N08034 manufactured by ZIEMEX†

† Trade name

CONCLUSIONS

The Time Temperature Precipitation and Time Temperature Sensitization behavior confirm the improved resistance to sensitization related to sigma-phase formation of UNS N08034. Further UNS N08034 shows the advantage of a lower solution annealing temperature compared to its precursor UNS N08031.

UNS N08034 has an excellent corrosion resistance to media of the chemical process industry. It is advantageous for use in sulfuric acid service media, in the production of phosphoric acid, in seawater applications, in the pulp and paper industry, in the salt manufacturing industry, in the oil and natural gas extraction under sour gas conditions and in flue gas desulphurization applications in modern coal-fired power stations.

UNS N08034 can be readily welded with common welding processes with higher alloyed filler metal AWS A5.14 - ERNiCrMo-13²⁵.

UNS N08034 can be hot-roll clad using conventional methods and equipment.

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