



CORROSION PRODUCTS DEVELOPMENT ON HAYNES® 282® GAMMA - PRIME (γ') STRENGTHENED ALLOY AT 550 °C UNDER SALT MIST CONDITIONS FOR 500 HOURS

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ABSTRACT

Prior to utilizing new advanced materials in coal power plants, a large number of experimental testing is required. Test procedures are needed in specialized high temperature laboratories with state of the art facilities and precise, accurate analytical equipment capable of performing tests at a variety of temperatures and environments. In this study, the results of a unique technique involving salt spray testing at high temperatures are presented. The Haynes® 282® gamma – prime (γ') strengthened alloy fabricated by means of three different manufacturing processes: HAYNES® 282® WROUGHT alloy, Haynes 282-SINT alloy, and finally Haynes 282-CAST alloy have been tested. The materials have been exposed to a salt spray corrosion atmosphere using 1% NaCl - 1% Na₂SO₄. Post exposure investigations have included SEM, EDS and XRD examinations.

The test using salt spray of 1% NaCl - 1% Na₂SO₄ water solution at 550 °C for 500 hours indicted no influence on the corrosion products formation, where Cr₂O₃ has been developed in all three alloys, whereas NiO has been found only in Haynes 282-CAST material. On the other hand, it has been found that the fabrication process of HAYNES® 282® alloy strongly influences the corrosion products formation under the high temperature exposures.

Keywords: salt spray corrosion, Haynes® 282® alloy, SEM, EDS, XRD

1. INTRODUCTION

Eco friendly generation of coal fired power plants are necessary in global economy and global electricity market. Higher and more restricted law around the globe related to CO₂ emission, especially in the European Union (EU), forces the development of more friendly technologies, higher efficiency and higher output of energy produced from coal. Reduction in CO₂ emissions from pulverized coal fired power plants can be achieved by increasing the operating temperature (and pressure) of the steam systems, which results in an increase in overall plant efficiency. The growing interest in more efficient electricity generation is driving research to high temperature resistant alloys. The main driver behind this is a clear dependence between efficiency of the power plant and steam conditions (temperatures and pressures) [1]. Typically 1% increase in absolute efficiency results in as much as 3% reduction in CO₂ emissions [2]. Nevertheless, increased steam temperatures significantly influence the oxidation rate of the materials in contact with it. More severe conditions increase oxidation rates and can lead to the formation of thicker, less protective scales, that can reduce power plant performance and component lifetimes. Among materials currently employed for boiler tubing there is a limited number of alloys which could exhibit acceptable steam oxidation and mechanical properties at high temperatures (650 – 750 °C). Austenitic steels are currently employed for the final stages of the super-heater (SH) and re-heater (RH) tubing; among them TP347HFG, Super 304H are most commonly used [1]. Regrettably, at harsher conditions even austenitic steels may fail due to the formation of nonprotective scales rich in Fe₂O₃, Fe₃O₄ with high tendency to exfoliation and spallation. Therefore, Ni based alloys with better mechanical and corrosion resistance need to be considered for the hottest sections in pulverised power plants [3]. In this regard, the paper aims to investigate the high temperature corrosion performance and phase development induced via exposure in 1% NaCl - 1% Na₂SO₄ (1g NaCl + 1g Na₂SO₄ in 100 g of H₂O) salt spray solution at 550 °C for 500 hours. In the test Haynes® 282® alloy fabricated via three different processes has been used: HAYNES® 282® WROUGHT alloy, Haynes 282-SINT alloy, and finally Haynes 282-CAST alloy. The materials have been exposed in a salt spray corrosion atmosphere of 1% NaCl - 1% Na₂SO₄. The tests have been carried out to study influence of salt spray mixture on phase development at 550 °C. The surface and cross-sections of the exposed alloys have been investigated analytically using Scanning Electron Microscopes coupled with Energy Dispersive Spectrometer (EDS). Phase development under high temperature exposure was carried out by means of an X-Ray Diffractometer using Bragg-Bentano and In-plane, out of plane configurations. Mass change data was examined every 100 hours using high accurate electronic balance.

2. Experimental procedure

2.1 Materials

In this work the same alloy fabricated according to three different methods have been tested:

- 1) Commercially available HAYNES® 282® WROUGHT alloy rods produced by Haynes International (USA);
- 2) As cast alloy (Haynes 282-CAST) produced at the Foundry Research Institute (Krakow, Poland) by melting HAYNES® 282® WROUGHT alloy rods in atmospheric air in a medium-frequency furnace using 40 kg crucible capacity lined with alumina. During heating and melting the alloy surface was covered with argon and deoxidized with nickel-magnesium master alloy. The molten alloy was then poured under gravity condition at 1560 °C into phenol-formaldehyde resin sand coated with zirconia-based binder [4]. The alloy cooled down to room temperature without any forced conditions.
- 3) HIP/PM alloy (Haynes 282-SINT) made by Carpenter Powder Products Inc. (USA) in cooperation with Energy Industry of Ohio (USA) from argon gas atomized powder that was produced from HAYNES® 282® WROUGHT alloy using Micro-Melt® process and then compacted under high neutral gas (argon) isostatic pressure at elevated

temperature [5]. Chemical composition by weight percentage (wt%) of the powder used to produce H-282 alloy is listed in **Table 1**:

Table 1 Powder chemical composition for Haynes 282-SINT alloy fabrication

Ni	Cr	Co	Mo	Ti	Al	Fe	Mn	Si	C	B
57	20	10	8.5	2.1	1.5	1.5	0.3	0.15	0.06	0.005

The raw powder was screened to a -60 mesh yielding a maximum particle size diameter of ~ 250 μm and down including fines. HIP parameters are listed in **Table 2**:

Table 2 HIP parameters used to fabricate Haynes 282-SINT alloy

Temperature [$^{\circ}\text{C}$]	Time [min]	Pressure [bar]	Atmosphere
1162.8 +/- 3	240 +/- 15	101.7	Argon

The alloy has been heat treated under a three stage process. In Stage 1, the alloy was Solution Annealed at 1121 – 1149 $^{\circ}\text{C}$ for 12 hours then gas fan cooled (GFC). In Stage 2, the alloy was precipitation age hardened at 1010 $^{\circ}\text{C}$ for 2 hours, then air cooled. In Stage 3, the alloy was reheated to 788 $^{\circ}\text{C}$ and held for 16 hours, then air cooled. [6]. **Figure 1** provides a diagram depicting the processing methods of commercial Haynes[®] 282[®] alloy used in this study.

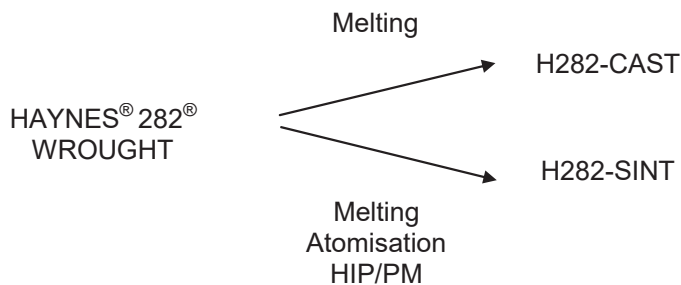


Figure 1 Fabrication processes of commercially available Haynes[®] 282[®] alloy

Prior to the high temperature tests, the samples with a 10 × 10 × 2 mm dimension have been ultrasonically cleaned for 15 minutes at 40 $^{\circ}\text{C}$, the dimensions have been accurately measured using a digital balance with accuracy of 0.001 mg. The average area of the sample was 1.5 cm^2 . The samples chemistry has been characterized using Energy Dispersive X-Ray Spectrometry (EDS). The outcomes of the measurements are shown in **Table 3**. The analytical investigations were compared with the nominal composition provided by Haynes International [7].

Table 3 Chemical composition measured in wt% by EDS on the samples prior to steam oxidation tests

Composition by EDS in FRI	Material	Elements, wt%										
		C	Ni	Cr	Co	Mo	Ti	Al	Fe*	Mn	Si	B**
	Haynes 282-CAST	0.1	Bal.	18.3	10.0	8.2	2.1	1.9	0.3	0.3	0.17	-
	Haynes 282-SINT	0.1	Bal.	19.0	10.7	7.7	2.5	1.5	0.7	0.3	0.19	-
	HAYNES [®] 282 [®] WROUGHT	0.1	Bal.	20.0	10.2	8.5	2.1	1.5	1.5	0.3	0.15	0.005

*maximum, ** undetectable

2.2 Salt spray experiment

Schematic illustration in **Fig. 2** presents salt spray corrosion experimental rig.

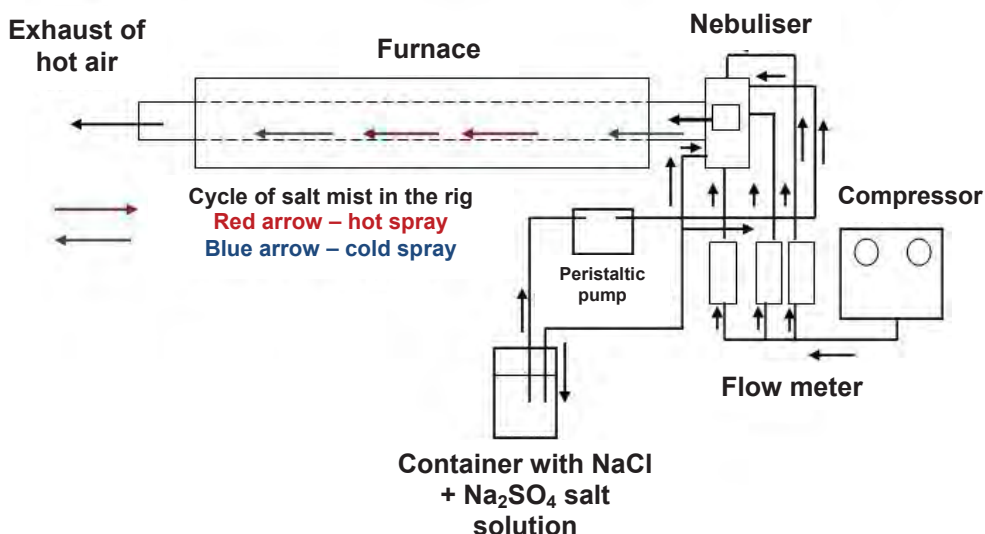


Figure 2 Schematic illustration of salt spray test rig in FRI containing 1% NaCl – 1% Na₂SO₄ salt solution used in this work

The Ni based samples listed in **Table 3** were placed in experimental test rig and have been exposed to salt spray at 550 °C. The salt spray conditions have been created from the salt solution containing 1% NaCl and 1% Na₂SO₄. Low concentration salt spray solution has been used in this test in order to avoid clogging of the nozzle; the nozzle undergoes clogging while more concentrated mixture of salt is used due to the tiny dimension of the nozzle hole. The samples after preparation procedure described previously have been placed in the calibrated hot zone of the furnace. When the furnace reached 200 °C, the compressor has been turned on in order to pump the salt solution from the container placed below the furnace. The salt solution has been pumped from the container by peristaltic pump to the nebuliser where tubing with compressed air was connected in order to create the spray under the pressure slightly exceeded 1 bar. The pressure of the mist entered the furnace has been controlled by flow meter controllers. When the cold salt spray entered the nozzle under the pressure and reached the hot zone of the furnace (where the samples have been placed), transformation of the salt solution to steam and salt crystals containing 1% NaCl – 1% Na₂SO₄ occurred. The salt spray under the pressure washed the samples around placed on the Al₂O₃ ceramic holder in the furnace. When salt spray solution passed the exposed Ni based alloys in the furnace, the solution exhausted from the furnace by the hole in the back of the end flange attached to stainless steel cylinder. In order to avoid any reaction with stainless steels cylinder, the cylinder has been lined with high purity of Al₂O₃.

3. Results and discussion

3.1 Kinetics

Figure 3 shows kinetic data of re fabricated Haynes[®] 282[®] Ni based alloy.

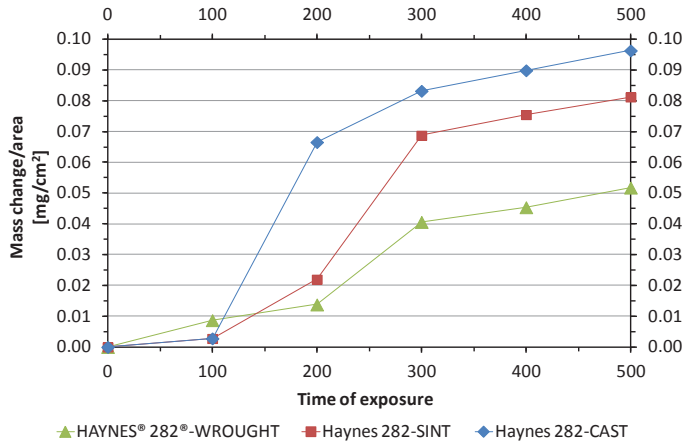


Figure 3 Kinetic data obtained in salt spray test containing 1% NaCl – 1% Na₂SO₄ salt solution for 500 hours at 550 °C

The graph shows that fabrication process influenced the kinetic behaviour. The shape of kinetic curves looks similar from alloy to alloy, nevertheless, the highest value for oxidation rate has been observed for the cast material, the lowest for HAYNES® 282® WROUGHT alloy. The middle values have been observed for sintered material. It has been observed that after 100 and 200 hours the exposed materials showed acceleration in mass gain at 550 °C, further exposure showed steady state corrosion products formation and further weight gain in the exposed alloys.

3.2 Surface microstructures

Figure 4 shows surface microstructures developed under salt spray corrosion tests at 550 °C for 500 hours.

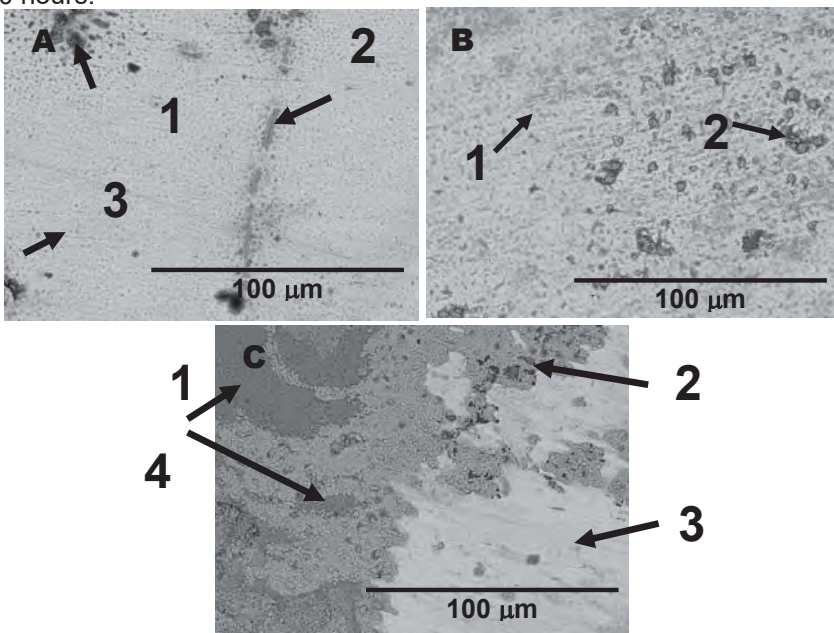


Figure 4 Surface morphologies of: A) HAYNES® 282® WROUGHT, B) Haynes 282-SINT and C) Haynes 282-CAST alloys after salt spray test containing 1% NaCl – 1% Na₂SO₄ salt spray solution for 500 hours at 550 °C

The exposure of fabricated Haynes[®] 282[®] alloys showed some differences in terms of corrosion products developed on the exposed surfaces. The EDS analyses are shown in **Table 3**.

Table 4 Chemical composition measured in wt% by EDS on the exposed samples, numbers in the table corresponds to the numbers in SEM micrographs shown in **Figs. 4**

Alloy		Ni	O	Cr	Co	Mo	Na	Ti	Al	Cl	S	Mn	Fe
HAYNES [®] 282 [®] WROUGHT	1	50.5	15.3	12.3	7.1	8.1	2.1	2.8	1.5	-	0.3	0.2	-
	2	44.2	21.1	16.9	8.4	6.2	1.7	1.3	1.0	0.1	-	0.1	-
	3	39.0	21.9	17.5	5.9	4.8	2.1	1.6	1.2	-	0.2	0.1	0.4
Haynes 282-SINT	1	41.7	16.9	14.5	6.0	7.0	3.4	2.5	1.8	0.2	0.5	-	-
	2	37.6	23.9	15.4	5.2	4.7	6.7	1.0	0.9	-	0.3	0.2	-
Haynes 282-CAST	1	9.8	37.7	39.4	1.4	0.7	0.5	4.1	1.1	-	-	0.8	0.1
	2	35.5	41.2	5.9	10.8	1.1	1.5	18.8	6.9	-	0.7	0.2	0.4
	3	40.7	12.9	7.4	8.2	6.4	1.1	0.4	1.7	-	0.2	-	0.4
	4	41.1	22.7	9.9	8.7	7.6	0.1	1.1	1.0	-	-	0.3	-

The analyses showed high degree of variations, corrosion products, bearing in mind that alloys have been exposed at 550 °C, the formation of extremely thin oxide scale is assumed. Therefore, in order to find corrosion products formed on the surfaces XRD investigations have been carried out.

3.3. XRD analyses

The XRD investigations have been carried out using two techniques for thin films observation, i.e., out of plane and in plane measurements respectively [8, 9]. The out of plane technique is used to obtain crystal structure information perpendicular to the samples surface (or stacking direction). In order to obtain information parallel to the investigated material in plane technique needs to be considered. In plane technique is important to receive the following information:

- 1) Diffraction from the lattice planes normal to the surface of the sample can be observed.
- 2) Diffraction from the extremely thin film is required.
- 3) Diffraction and background intensities from the substrate is highly reduced.
- 4) A non destructive depth profile analysis is possible by using various angles of the incident X-ray beam.

In this study, the exposed materials resulting in the formation of extremely thin oxide scales have been investigated using various angles of the incident X-ray beam from 0.5 to 2°. Measurements conditions for the in plane and out of plane technique are shown in **Table 4**.

Table 5 Measurement conditions for out of plane and in plane techniques

Measurement conditions in plane technique			
Method	Range (deg)	Step (deg)	Speed (deg/min)
2Theta	30-100	0.144	1
Measurement conditions out of plane technique			
Method	Range (deg)	Step (deg)	Speed (deg/min)
2Theta	20-120	0.015	0.5

The XRD analyses in the form of pie graphs are shown in **Fig.5** The pie graphs in [%] present concentration of phases found under the salt spray corrosion test using 1% NaCl – 1% Na₂SO₄ solution at 550 °C for 500 hours.

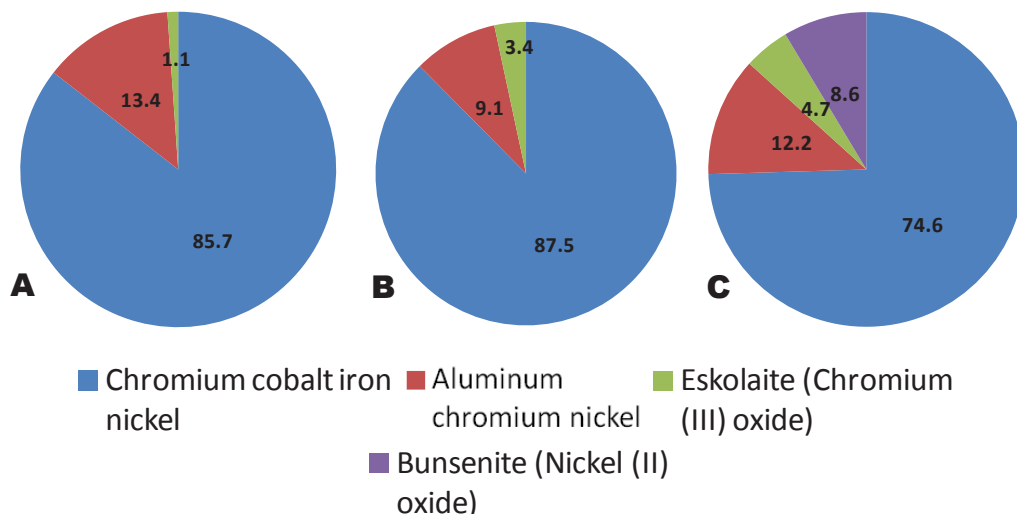


Figure 5 XRD analyses on pie type graphs for A) HAYNES[®] 282[®] WROUGHT, B) Haynes 282-SINT and C) Haynes 282-CAST alloys after salt spray test containing 1% NaCl – 1% Na₂SO₄ salt solution for 500 hours at 550 °C

The performed XRD investigations using in plane and out of plane techniques indicate lack of salt spray 1% NaCl – 1% Na₂SO₄ solution influence on the phase constituents at 550 °C. Nevertheless, fabrication process of the Haynes based material influenced the oxide phase formation under high temperature exposure. The results correspond to kinetic findings and clearly indicate that the highest concentration of Cr₂O₃ and NiO oxides and the highest oxidation rate have been found to be in Haynes 282-CAST, whereas the lowest in HAYNES[®] 282[®] WROUGHT alloy. The highest mass gain of Haynes 282-CAST was associated with the formation of Cr₂O₃ and NiO corrosion products. In the HAYNES[®] 282[®] WROUGHT, chromium – cobalt – iron nickel matrix and aluminium chromium nickel matrix with tiny amount of Cr₂O₃ oxide has been found showing high resistance to oxidation. In Haynes 282-SINT, the percentage of Cr₂O₃ oxide was slightly higher with lower content of aluminium chromium nickel matrix and chromium cobalt iron nickel matrix.

3.4 Cross-sections

The presented cross-section images in **Fig. 6** performed in Backscatter Electron (BSE) mode under SEM revealed that all three alloys developed extremely thin and adherent oxide scales. It has been observed in this study, that HAYNES[®] 282[®] WROUGHT alloy showed no internal oxidation process. However, in two other alloys isolated oxides rich in Al and Ti have been observed in the grain boundaries, suggesting internal oxidation corrosion products formation (**Fig. 6B** and **Fig. 6C**). The formation of such products suggest the oxidation of (γ') gamma – prime Ni₃(Al,Ti) phase. The micrographic investigations using high resolution SEM coupled with EDS analyser showed no sulphur, chlorine or sodium in the oxide scale suggesting, no influence of salt solution.

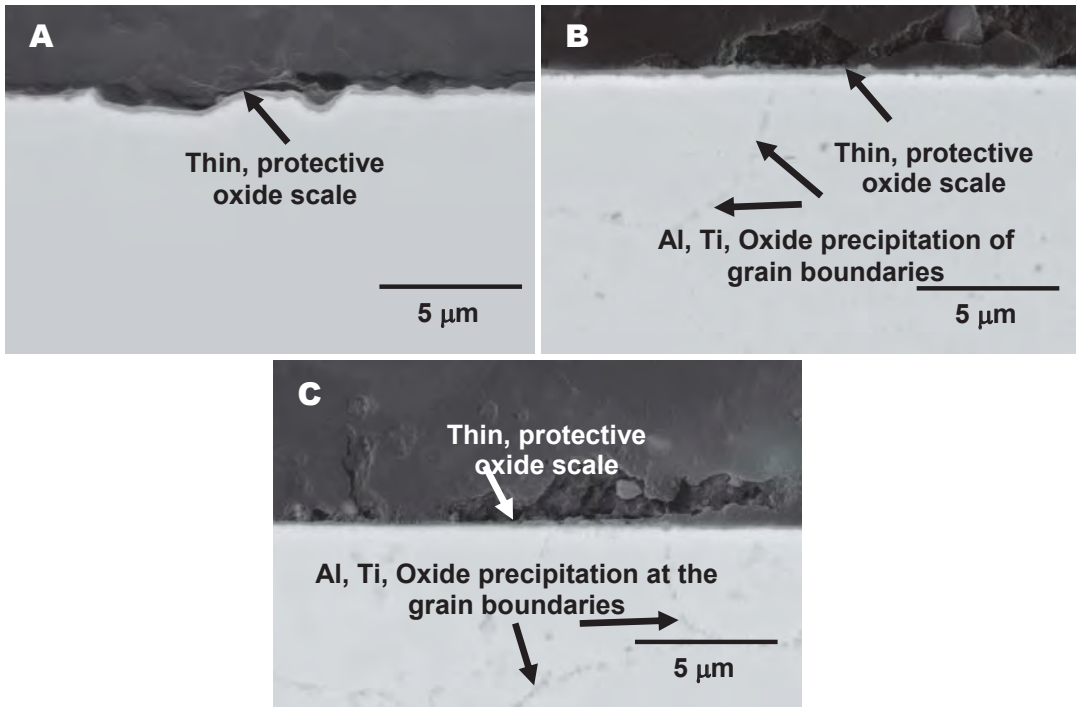


Figure 6 Cross-section morphologies of: A) HAYNES® 282® WROUGHT, B) Haynes 282-SINT and C) Haynes 282-CAST alloys after salt spray test containing 1% NaCl – 1% Na₂SO₄ salt solution for 500 hours at 550 °C

3.5 Thermodynamic consideration

Thermodynamic consideration can be used for explanation of oxide scale formation upon high temperature exposure of Ni based alloys fabricated using three different processes in 1% NaCl – 1% Na₂SO₄ salt solution for 500 hours at 550 °C. However, in current situation, despite of 1% NaCl – 1% Na₂SO₄ salt solution, the corrosion process involves more oxygen containing atmosphere than corrosion process influenced by the salt presence. Because, under certain condition only two corrosion products have been found Cr₂O₃ and NiO, thermodynamic considerations will be rationalised in the following way.

When clean, surface of the Ni based alloy is exposed to the salt spray of a 1% NaCl – 1% Na₂SO₄ rich oxygen solution. In the beginning at 100 °C water starts to become steam according to the following chemical reaction.



$$\Delta G_{\text{H}_2\text{O}} = 230.000 - 8.14T \cdot \ln(T) + 9.25T$$

Further, when rich in oxygen atmosphere surrounds the surface of the exposed alloys, immediately, the most active elements start to be oxidised according to the following chemical reactions:



For the reactions, the values of standard free energy formation (ΔG_T° , KJ/mole) with temperature (T in Kelvin) can be obtained from the following equations [10]:

$$\text{Cr}_2\text{O}_3(\text{s}) \quad \Delta G_T^\circ = -750790 + 171.4T \quad (4)$$

$$\text{NiO(s)} \quad \Delta G_T^\circ = -471790 + 172.1T \quad (5)$$

Also,

$$\Delta G_{\text{Cr}_2\text{O}_3, T}^\circ = -RT \ln K_{\text{Cr}_2\text{O}_3} = -RT \frac{a_{\text{Cr}_2\text{O}_3}}{(a_{\text{Cr}})^2 \cdot (p_{\text{O}_2})^{3/2}} \quad (6)$$

$$\Delta G_{\text{NiO}, T}^\circ = -RT \ln K_{\text{NiO}} = -RT \frac{a_{\text{NiO}}}{a_{\text{Ni}} \cdot (p_{\text{O}_2})^{1/2}} \quad (7)$$

Where $K_{\text{Cr}_2\text{O}_3}$, K_{NiO} , are the equilibrium constants for reactions 2 – 3 and R is the gas constant. Under certain oxygen partial pressure ($P_{\text{O}_2} = 0.21 \text{ atm}$), the minimum Cr and Ni, activities required to form Cr_2O_3 , and NiO can be calculated according to the above equations as shown in **Fig. 7**.

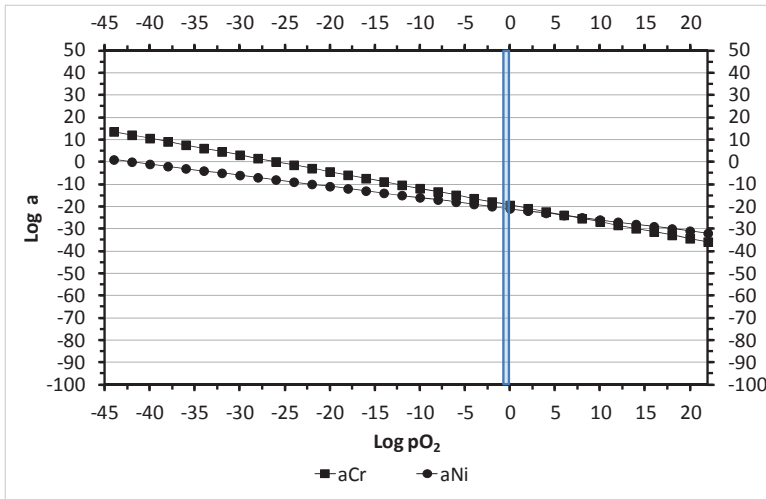


Figure 7 Minimum activities of Cr and Ni required to form Cr_2O_3 , and NiO at 550 °C

In present situation, thermodynamic calculations reveal that the minimum activities of Cr and Ni required to form Cr_2O_3 and NiO in the environment (assuming $p_{\text{O}_2} = 0.21 \text{ atm}$) are 10^{-17} and 10^{-16} respectively. These results indicate that both phases would form preferentiall.

In this work it has been found that three alloys showed development of Cr_2O_3 in different percentage, however only Haynes 282-CAST developed NiO and indicated the highest corrosion degradation. Therefore, in this manner, this section is divided in paragraph where HAYNES[®] 282[®] WROUGHT and Haynes 282-SINT corrosion resistance is described together and paragraph dedicated only to corrosion resistance of Haynes 282-CAST alloy.

3.5.1 HAYNES[®] 282[®] WROUGHT and Haynes 282-SINT alloys

The HAYNES[®] 282[®] WROUGHT and Haynes 282-SINT alloys showed no NiO formation under 550 °C exposure in 1% NaCl – 1% Na_2SO_4 water salt solution according to findings revealed by XRD investigations. Both alloys developed of tiny amounts of Cr_2O_3 showing high degree of corrosion resistance. Nevertheless, the alloy with higher percentage of Cr_2O_3 showed development of internal oxidation process where corrosion products of Al_2O_3 and TiO_2 have been observed. Based on this results it can be found, that the formation of oxide scale i.e. Cr_2O_3 in Ni based alloy lead to the development of internal oxidation zone underneath the formed oxide scale. The process may lead under longer exposures to failure due to development of oxides (corrosion products) inside the material at the grain boundaries [11]. The internal oxidation predominantly concerns around Al and Ti oxidation and has been described recently by Young and Deodeshmukh respectively [12, 13].

3.5.2 Haynes 282-CAST alloy

Only in Haynes 282-CAST alloy NiO phase besides Cr_2O_3 phase has been observed with the highest percentage. The results lead to conclusion that casting process influence hardly the formation of Cr_2O_3 and NiO. The Cr and Ni are the main constituents of Haynes[®] 282[®] (γ') gamma - prime alloy, oxidation of these two elements dominates over the other elements from the metal matrix as has been shown in this work. The formation of NiO during the exposure may be related to the decreased concentration of Cr in the alloy in comparison to HAYNES[®] 282[®] WROUGHT and Haynes 282-SINT alloys (Table 3). Lower corrosion resistance of CAST alloy can be associated with the evaporation of Cr during melting and casting process under reduced pressure [14,15,16]. The reduced Cr concentration compared to the other alloys in this study influenced the corrosion behaviour at 550 °C. At higher temperature i.e. 800 °C high temperature oxidation process can accelerates more rapidly. The findings are in good agreement with work presented by Robb et al. [17]. The author stated that 20 – 25 wt% of Cr is required to form continuous Cr_2O_3 scale in Ni based alloys, in other case when Cr concentration decreased below 20 wt% then mainly Cr_2O_3 -NiO scale form. Similar to this work where Cr concentration decreased below 20 wt% and formation of Cr_2O_3 – NiO oxide scale mainly with additional Mo, Co has been observed.

4. Conclusions

The aim of this study was to investigate phase development after salt spray corrosion test using 1% NaCl – 1% Na_2SO_4 salt solution at 550 °C for 500 hours. Based on the results achieved, the following conclusions can be made:

- 1) No influence of salt spray 1% NaCl – 1% Na_2SO_4 water solution at 550 °C on three differently fabricated Haynes[®] 282[®] alloys
- 2) The formation of Cr_2O_3 scale in HAYNES[®] 282[®] WROUGHT and Haynes 282-SINT alloys at 550 °C has been observed.
- 3) The formation of mixed NiO – Cr_2O_3 oxide scale in Haynes 282-CAST alloy has been observed.
- 4) Internal oxidation of Al and Ti from (γ') gamma – prime $\text{Ni}_3(\text{Al,Ti})$ phase has been observed in Haynes 282-SINT and Haynes 282-CAST alloys at 550 °C.
- 5) No internal oxidation process has been observed in HAYNES[®] 282[®] WROUGHT alloy.

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