Hot Corrosion Studies of Cold Sprayed NiCrAlY Coating on Nickel-based Superalloy at 900°C

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Abstract—The cold spray process is new emerging technology to deposit the dense coatings by avoiding or minimizing many deleterious shortcomings of traditional thermal spray methods such as high-temperature oxidation, evaporation, melting, crystallization, residual stresses, and gas release without any change in the phase. In the present research work an attempt has been made to study the hot corrosion behavior of cold sprayed NiCrAIY coating on a Ni-based superalloy in the molten salt environment of Na₂SO₄–10%NaCl at 900 °C under cyclic conditions for 50 cycles. The thermogravimetric technique was used to establish the kinetics of corrosion. The XRD, SEM/EDS and X-ray mapping techniques were applied to evaluate the corrosion products. The NiCrAIY coating have shown good hot corrosion resistance and protected the underlying substrate. The poor performance of bare superalloy as compare to coated one is due the active oxidation of bare superalloy. But in case of coated superalloy the active oxidation did not occurred due to the formation of protective oxides of chromium and aluminum which did not allow the chlorine to penetrate.

Keywords: Cold Spray, NiCrAlY Coating, Hot Corrosion

INTRODUCTION

Hot corrosion of the metallic hot section components of waste to energy plant is very critical due to the presence of chlorine based environment. In the presence of chlorine the active oxidation takes place which consume the metallic components at unpredictable high rate. Lu et al [1] reported that the chlorine based energy to waste plant are running still at low efficiency around 25% as compare to energy plants with non chlorine environment which are running at around 47% efficiency. It has been learnt from the literature that a small change in temperature in the chlorine based environment affects the corrosion be behavior of metals/alloys. With advancements in materials, many new Ni based materials with high chromium have been developed for high temperature applications. But these materials even cannot sustain for longer period of time in chlorine based aggressive environment. From last three decades coatings are being used to protect the underlying metallic components. Coatings are considered as economical viable solution to protect the metallic components from hot corrosion. According to Matthews et al. [2] the coating can enhance the life of components which is equal to the 10 times the cost of producing coatings. A number of researchers [3-8] investigated the hot corrosion behavior of thermal spray coatings and they found that use of thermal spray coatings significantly increases the life of materials.

The NiCrAIY coatings had been deposited with different thermal spray processes by numerous researchers [9-12] and they observed that these coatings provided the good protection to underlying substrate. A few studies on hot corrosion behavior of NiCrAIY coatings deposited with cold spray process are available in open literature. Cold spray process is high velocity low temperature process. It is 100 percent solid state spraying process. The basic principle of cold spray process is very simple and shown in Fig. 1. The spraying particles are

accelerated through convergent divergent nozzle with a velocity in the range 300-1200 m/sec but the temperature always below than the melting point of spraying particles. Due to high impact velocity the spraying particle plastically deform on impact and form splats, which bond together to produce coatings. Due to high velocity and low temperature of the spraying particles in cold spray process many harmful effects of traditional thermal spray process such as high-temperature oxidation, evaporation, melting, crystallization, residual stresses, gas release and any change in the phase can be avoided or minimizes. Moreover very dense coatings with low porosity can be produced with cold spray than other thermal spray technique which is highly desirable for hot corrosion resistance.

The objective of present research work is to evaluate the corrosion behavior of bare superni 600 and NiCrAlY cold sprayed Superni 600 in chlorine based environment. The hot corrosion behavior was studied in molten salt of Na₂SO₄–10%NaCl at 900 °C under cyclic conditions for 50 cycles.



Fig. 1: Schematic Diagram of Cold Spray System

EXPERIMENTAL PROCEDURE

Substrate Material

The Superni 600 superalloy (Midhani grade Ni based superalloy) was used as substrate material in the present

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study. The material was provided by Mishra Dhatu Nigam Limited, Hyderabad (India) in the rolled sheet form.

The composition along with some application of superalloy as per catalog of manufacture is provided in Table 1.

Grade	UNS No.	Fe	Ni	Cr	Ti	Al	Mo	Mn	Si	Cu	С	Applications
Superni	N 06600	10.0	Bal	15.5	-	0.6	-	0.5	-	-	0.2	Furnace muffles in oxidizing atmosphere.
600												High temperature springs, Heat exchanger
												tubes, Chemical and food processing
												equipment,
												Nuclear parts viz. feed values, combustion
												chambers.

Table 1: Chemical Composition, UNS Number and Application of Superni 600 as per the Catalog of Manufacturer

Coating Powder

Commercially available Ni-22Cr-10Al-1Y power from Sulzer Metco (USA) was used as feedstock material for coatings. A SEM micrograph of NiCrAlY showing the microstructure of the powder has been shown in Fig.2. The particle size of the powder varies from 5 to 20 μ m and shape of powder is also varying spherical to oval. The EDS analysis shows Ni (66%wt), Cr (4%wt), Al (9%wt) and Y (1%wt) which is close to designated composition.



Fig. 2: SEM Micrograph of NiCrAlY Powder

Formulation of Coatings

The bare superalloy specimens were cut from the sheet with dimension of 20x15x3mm. Polishing of specimen was done using SiC emery papers 100, 220, 400, 600 grit size. Then specimens were grit blasted with alumina powders (grit 45) before development of the coatings by cold spray process.

Cold spray Kinetics 4000 system was used for depositing the NiCrAlY coating. Coatings were developed at ASB industries, Inc., Barbeton Ohio (USA) with Kinetics 4000 from CGT technologies, Germany using Helium as process gas. Standard spray parameters, as mentioned in Table 2 have been used while coating the specimens. All the process parameters were kept constant throughout the coating process. Table 2: Standard Spray Parameters

Parameter	Value			
Gun Temperature	400°C			
Gun Pressure	38 bars			
Powder feed rate	40 gm/min			
Process gas flow rate	150 m3/sec			
Carrier Gas	Nitrogen			
Flow rate of gas	4 m3/h			

Hot Corrosion Studies

Hot corrosion studies were carried out in silicon carbide tube furnace at 900°C with temperature variation of \pm 5°C.The studies were conducted for 50 cycles in molten salt environment of Na2SO410%NaCl. Each cycle consisted of 1h heating at 900°C followed by 20min cooling at room temperature. The samples were subjected to mirror polishing before the corrosion run. Thereafter, a coating of Na₂SO₄.10%NaCl salt was applied on the pre heated samples at 250°C with the help of a camel hair brush. The coating thickness of Na₂SO₄10%NaCl salt was kept as uniform thickness with 3 to 5 mg/cm^2 . Then the samples were dried in oven at 110°C temperature for 3-4 hours to remove the moisture. The weight of samples was measured after each cycle of study. The samples were visually examined at the end of each cycle for any change in the color, luster, adherence of scale to the substrate and spalling tendency. After 50 cycles of studies, the corrosion products on the surface were studied with XRD and SEM/EDS analysis. For crosssectional analysis the samples were sectioned and mounted in epoxy along cross-section. The mounted specimens were prepared by polishing, using SiC emery papers of 220, 400, 600 grit and subsequently 1/0, 2/0, 3/0, and 4/0 grades. Fine polishing was carried out using a 0.3 µm diamond paste. Then prepared specimens were analyzed with SEM/EDX and X-Ray mapping.

RESULTS

Visual Examination

The macrograph of hot corroded bare Superni 600 superalloy after 50 hours of cyclic exposure in molten salt Na_2SO_4 -10%NaCl environment at 900°C is shown in Fig. 3a. During first cycle of the study, the colour of Superni

600 turned light brownish grey which changes to dark brownish grey during subsequent cycles of exposure. Also some greenish tinges were observed after 7^{th} cycle onwards. A macro crack appeared in the scale after 6^{th} cycle of hot corrosion. Some peeling as well as spallation of the scale was observed after 10^{th} cycle of the study. The scale formed on the surface of bare Superni 600 was found to be fragile and irregular.

The macrograph for the hot corroded surfaces of cold sprayed NiCrAIY coated Superni 600 superalloy after 50 cycles of study is shown in Fig. 3b. During the initial cycle of study the colour of coated superalloy changes to greenish brown from dark brown. The colour of scale changes to gray with greenish shade during 5-8th cycle of hot corrosion. Then scale almost becomes greenish shade on grey background. Some spalling at the edges and corner was observed. The crack was observed in the scale of NiCrAIY coated Superni 600 on 26th cycle, thereafter the colour shade in the crack changes from grey to blackish shade. The spalling of scale was not noticed during study. The scale formed on the surface dense continuous and adherent.

Corrosion Kinetics

The weight gain data for the hot corroded bare and coated Superni 600 has been shown in Fig. 4. The coated Superni 600 shows a much lower weight gain as compared to bare Superni 600 in the given molten salt environment. The weight gained by NiCrAlY coated specimen is about 58% of the weight gain by bare Superni 600 after 50 cycles in the given molten salt environment. The parabolic rate constant k_p was calculated by a linear least-square algorithm function in the form of $(\Delta W/A)^2 =$ k_p t, where $\Delta W/A$ is the weight gain per unit surface area (mg/cm^2) , t is the hot corrosion time in seconds.. The parabolic rate constant, kp for bare Superni 600 calculated on the basis of 50 cycles data is found to b 16.934 $x10^{-10}$ g²cm⁻⁴s⁻¹, where k_p for NiCrAlY coated Superfer 600 is 5.806 $\times 10^{-10}$ g²cm⁻⁴s⁻¹. The value of k_p reported by Yuan [14, 13] for NiCrAlY coatings at 1000°C in air oxidation was 4.08 $x10^{-13}g^2cm^{-4}s^{-1}$ The coated superalloy has shown the parabolic behavior up to 50 cycles, whereas bare superalloy shows significant deviation from the parabolic path.







Fig. 4: Weight Gain vs. Number of Cycles Plot for the Coated and Bare Superni 600 Exposed to Hot Corrosion for 50 Cycles in Na2SO4-10%NaCl environment at 900°C

X-Ray Diffraction Analysis of Corroded Surface

X-Ray Diffraction analysis was carried out to see the different phases formed on the surface of specimens during hot corrosion in the given environment. The X-ray diffraction patterns for the NiCrAlY coated Superni 600 after hot corrosion for 50 cycles at 900°C indicate the formation of NiO as main phase along with Cr_2O_3 and $NiCr_2O_4$ as minor phases (Fig. 5). The XRD patterns of corroded bare Superni 600 indicate the formation of Cr_2O_3 , NiO and Al_2O_3 phases along with $NiCr_2O_4$.



Fig. 5: XRD Patterns after Hot Corrosion for 50 Cycles in Na₂SO₄-10% NaCl Environment at 900°C: (a) Bare Superni 600 (b) NiCrAlY Coated Superni 600

SEM/EDS Analysis

Surface Analysis

The SEM micrographs showing surface morphologies along with the EDS analysis of the corroded bare and NiCrAlY coated Superni 600 have been shown in Fig.6. The SEM image of bare Superni 600 shows a rough irregular surface. The EDS analysis reveals that the top scale of bare Superni 600 mainly consists of the oxides of Ni as the major phase along with some oxides of Cr and Fe (Fig. 6a).

The SEM image of NiCrAlY coated Superni 600 shows granular structure of the scale on surface (Fig. 6b). The structure appears like a cloud where black region are surrounded by white boundaries. The scale formed on NiCrAlY coated Superni 600 has Al and Cr as a principal phase along with Ni. Sufficient amount of O is also present to form oxides of these elements.

Cross-section Analysis

The SEM (BSE) image and elemental variation across the cross-section for the hot corroded cross-section of bare and NiCrAlY coated Superni 600 after 50 cycles of exposure are shown in Fig. 7.



Fig. 6: SEM/EDAX Showing Surface Exposed to Hot Corrosion for 50 Cycles in Na₂SO₄-10%NaCl environment at 900 °C (a) Bare Superni 600 (b) NiCrAlY Coated Superni 600

The EDS analysis at point 1 and 2 of bare Superni 600 superalloy shows the presence of Ni rich scale at top with some amount of Cr as shown in Fig. 7a. At the point

2 Cr increase as compared to point 1. The EDS analysis at point 3 reveals the Cr_2O_3 as the main phase of scale. Hence a layer rich chromium oxide is present under NiO rich scale at the top. Oxygen has penetrated up to point 4.

The SEM micrograph for the corroded cross-section of NiCrAIY coated Superni 600 indicates the white color thin streak of scale at the top (Fig. 7b). The EDS analysis on this thin streak at point1 reveals the presence of high concentration of Al with some amount of Cr and sufficient amount of oxygen indicating a very thin layer of Al₂O₃ with some Cr_2O_3 at the top. Beneath the top layer a continuous thick streak of partially oxidized coating is present. The EDS analyses at point 2 on this streak indicate the formation of NiO as major phase. The oxygen is present at the point 1 and 2 whereas it shows its absence at point 3 and 4 indicating the protective behavior of coating. So the top scale indicates the oxides of Al as the main phase beneath a thick layer of partially oxides coating having Ni as major phase.



Fig. 7: Cross-Sectional EDS of Bare and Coated Superalloy Exposed to Hot Corrosion for 50 Cycles in Na₂SO₄-10%NaCl Environment at 900 °C. (a) Bare Superni 600 (b) NiCrAlY Coated Superni 600

X-ray Mapping

The elemental X-ray maps at the cross-section of bare and NiCrAIY coated Superni 600 after cyclic hot corrosion in Na₂SO₄-10%NaCl environment at 900°C for 50 cycles are shown in Fig. 8. The BSE image of bare Superni 600 after 50 cycles of hot corrosion in the given molten salt environment show different layers (Fig. 8a). Element X-ray mapping reveals NiO rich scale at the top. Beneath layer contains the Cr as major phase along with small quantity of Ni and Fe. Therefore corroded specimen have Cr_2O_3 rich scale beneath NiO rich scale at the top.

The BSE image of coated corroded specimen shows compact dense and continuous scale at the top (Fig. 15).

Element mapping analysis of NiCrAIY reveals an Al enriched thick streak at the top of corroded specimen with oxygen and some amount of Cr which indicate Al_2O_3 rich scale at the top with some Cr_2O_3 . Just blow the top layer Ni is present as major phase along with small quantity of Cr, Fe, Al and oxygen indicating partially oxidized coating.

DISCUSSION

The bare Superni 600 superalloy under study has shown more weight gain as compared to the coated superalloy in the molten salt environment indicating the protective behavior of the coating. The weight gain of the bare superalloy was observed throughout the study, but more weight gain was found during initial cycles of study. This higher weight gain during initial might be attributed to the rapid oxidation of the surface elements to form oxide scale. Tiwari and Parkash [14], Kalsi et al. [15] and Sidhu et al. [16] had also reported the high weight gain during initial cycles of hot corrosion. The weight gain plot shows the deviation from the parabolic rate law due to the spalling and sputtering of the oxide scale. The higher weight gain in case of bare Superni 600 may be attributed to the formation of Ni rich oxide scale which is less protective in nature as compared to chromium oxides scale formed on the surface of coated Superni 600. The NiO was detected as major phase on the surfaces of corroded Superni 600 with EDS, XRD and X-Ray mapping analysis.

The cumulative weight gain coated Superni 600 was 10.1980 mg/cm² (Fig. 4). The coated superalloy follows the parabolic rate law in weight gain (Fig. 4) with minor deviations. The reason for this may be development of inhomogeneous oxides during hot corrosion. Similar results were also reported by Choi *et al.* [17], Liu *et al.* [18].

The first crack found on coated superfer 800H might be due to the different values of the thermal expansion coefficients of the oxides and stresses produced by cyclic oxidation. The better performance NiCrAlY coating might be due to the formation of protective Al₂O₃ layer at the top with some Cr_2O_3 . The formation of this layer is found with the EDS and XRD analysis and further confirmed with element X-Ray mapping analysis. In the initial cycles of hot corrosion the oxygen is penetrated through the pores and splat boundaries. The formation of protective oxides on the surface, in the pores and splat boundaries act as a barrier to the corrosive species. So it delays the further oxidation and prevents the hot corrosion. The low porosity of the cold spray coating is also responsible for lower rate of corrosion as the degrading species penetrates through pores. The presence of Fe in coating and scale (Fig. 8) indicates the diffusion of Fe particles from the substrate to the coating during hot corrosion cycles. Similar results of the inter-diffusion of elements between substrate and coating during hot corrosion was reported by Shifler and David [19]. Na, Si and Cl particles are also shows their existence all over.



Fig. 8: BSEI and X-ray Mapping of Bare and Coated Superalloy Exposed to Hot Corrosion for 50 Cycles in Na₂SO₄-10% NaCl Environment at 900 °C. (a) Bare Superni 600 (b) NiCrAIY Coated Superni 600

The presence of Na and Cl indicates the presence of these salt elements even after 50 cycles. Good hot corrosion resistance by NiCrAlY may be partially attributed to the small amount of Yttrium present in scale. According to Gurappa [20], yttrium dissolved either in the alloy or in the coating prevents segregation of sulphur present in the environment to the alumina scale/coating interface. It thereby prevents the harmful effect of reduced chemical bonding between the scale and the coating. Hanyi [21] studied the effect of addition of Yttrium on hot corrosion in Na₂SO₄-25% NaCl environment at 900°C and found that it improves the resistance of coating against hot corrosion.

Therefore NiCrAlY coating formed by cold spray on superfer 800H can be used in aggressive environment due to its low porosity and high hardness and can enhance the life of components such as power plants, gas turbines working in sulfate-chloride environment.

The EDS analysis of surface indicates the elements like Cl, S and Na which reveals the presence of these even after 50 cycles of the study. Probably during initial cycles of study, these element salts deposited on the surface form the molten salt. At high temperature the molten deposits take part in fluxing and hence causing dissolution of the protective oxide scale. The dissolution of Cr_2O_3 is generally takes place through basic fluxing of oxide in chlorine based environment, as reported [22]. $Cr_2O_3 + 3SO_3 + 2O^{2-} = 2CrO_4^{2-} + 3SO_2$

In the molten salt of NaCl the hot corrosion could be accelerated. Cr_2O_3 is very susceptible to attack in the presence of molten NaCl. The mechanism of the corrosion process for Cr_2O_3 scale can be explained as per the following reaction [23]:

8NaCl (l) + 2Cr₂O₃ (s) + 5O₂ (g)
$$\rightarrow$$
 4Na₂CrO₄ (S) + 4Cl₂ (g)

The Cl_2 produced penetrates through oxide scales along the cracks or pores and react with Cr in the coating and form volatile chlorides:

$$Cr(s) 3/2Cl_2(g) \rightarrow CrCl_3(g)$$

These chlorides may diffuse outward to the outer surface through pores and crack. The chlorides on the surface may deoxidize to form Cr oxides on the surface.

 $2CrCl_3(g) + 3/2O_2(g) \rightarrow Cr_2O_3(s) + 3Cl_2(g)$

The Cl_2 formed in above reaction can partially be penetrated back into the scale and repeat the reaction. So a little amount of chlorine is sufficient for the above said chlorination reaction. This is known as active oxidation. The oxides of other elements, such as Ni and Al can also follow the above reactions.

For the bare alloy, it is not possible to form a protective Al_2O_3 scale or Cr_2O_3 scale due to its low content of Al and Cr. This results in formation of loose non-protective scale layer on the uncoated superalloy as shown in BSE image of Fig. 8. The phase diagram of Ni/ Cr/Fe–Cl–O system in Fig.9 indicates that the formation of Cr₂O₃ takes place at lower partial pressure of oxygen as compared to oxides Ni. The EDS, XRD and X-ray mapping of corroded bare superalloy in the present study indicates the formation of NiO at the top layer and beneath Cr_2O_3 at surface close to the scale/alloy interface. The results are in confirmation with the phase diagram. The structure of the scale layer formed on the uncoated superalloy confirms that the scale formed by via oxychlorination reactions.



Fig. 6: Phase Diagram of Ni/Cr/Fe-Cl-O System [22]

In case of coated superalloy, the Cr and Al contents are sufficient to form Al_2O_3 and Cr_2O_3 oxide at the top. In the present study a continuous protective Al_2O_3 rich scale formed at the top of corroded coated specimen did not allow the O, S or Cl to penetrate. More over threshold concentration of Cr required for the formation of Cr_2O_3 is around 15 wt% and of Al for Al_2O_3 is 6 wt% [22]. The wt% of Al and Cr in the coating is 10 wt% and 22% respectively in the present study which is enough for the formation of protective oxide layer.

CONCLUSION

- Strongly adherent scale formed on NiCrAlY coated superalloy remains intact with the coating after 50h of cyclic exposure, results in successful hot corrosion protection.
- The cold sprayed NiCrAlY coating protect the substrate with the formation of Al₂O₃ rich scale at top in Na₂SO₄-10%NaCl molten salt environment at 900°C for 50 cycles.
- The weight gained by NiCrAIY coating is 58% of the weight gain by bare Superni 600 after 50 cycles in the given molten salt environment. The coating is able to reduce the weight gain and it follows the parabolic rate law of oxidation.
- The highly protective behavior of the coating is partially due to the dense coating structure obtained by the cold spray coating.
- The bare Superni 600 experiences active oxidation.

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