CORROSION BEHAVIOUR OF Ni PTFE and Ni Al2O3 COATING

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Abstract

The Objective Of The Present Work Is To Study The Corrosion Behaviour Of Ni Al$_2$O$_3$ Coating Comparing With Other Types Of Coatings Such As Ni PTFE, Nickel Coatings, Nickel Phosphorous Coatings, Chromium Coatings, Nickel Cobalt Coatings And The Bare Metal. The Different Thickness Of Coatings Was Obtained By Adjusting Current Density And Deposition Time. The First Corrosion Test Conducted Is Salt Spray Corrosion Test. The Weight Loss Was Calculated. Among The Electro Deposition Process, The Ni Al2O3 Coatings Have Shown The Highest Hot Corrosion Resistance Followed By Ni-PTFE, Cr, Ni-Co, Ni-P, Ni Coatings And The Bare Metal. Then The Optimum Thickness For Ni Al2O3 Was Obtained.

Key words: Corrosion, Corrosion Behaviour, Coating, Electro Deposition Process

I. INTRODUCTION

Degradation by hot corrosion and erosion are the main failure modes of components in the hot sections of gas turbines, boilers, industrial waste incinerators, metallurgical furnaces, petrochemical installations, etc. Super alloys have been developed for high temperature applications, but they are not able to meet the requirements of both the high-temperature strength and the high-temperature erosion corrosion resistance simultaneously. One possible way to overcome these problems is the use of thin high-temperature erosioncorrosion and wear-resistant coatings. Coatings can add value to products by allowing the mechanical properties of the substrate materials to be maintained while protecting them against wear or corrosion. Although the Ni-based super alloys possess adequate strength at the turbine operating temperatures, they often lack resistance to the combustion environments. In such environments, protective coatings on the surface of super alloys are frequently used. "Lot of research is being carried out and show that the composite coating is a technique that produces a wide range of coatings for high temperature applications."

A. Nickel and Ni-Co Coatings

Nickel coatings are utilized in large number of applications due to its advantages such as strength, toughness and little resistance to corrosion/wear. Its mechanical properties can be varied by altering the electrolyte composition and operating parameters. Ni and Ni-Co coatings show moderate wear resistance and they do not withstand high temperature environment.

B. Ni-P Electroless Coatings

Electroless NiP coating has received widespread acceptance as it provides high hardness and moderate resistance to wear and corrosion.

C. Hard Chromium Coatings

Hard chrome plating are currently used in gas turbines to provide improved corrosion and oxidation resistance, and thereby extend component life.

Chromium electroplating is related to the reduction in the fatigue strength of base metal. The hard chromium electroplating decreases the fatigue resistance of a component, which is attributed to high residual tensile stress and micro cracks density contained into the coating.

D. Ni Al$_2$O$_3$ and NiPTFE Composite Coatings

Inert particles (SiC, WC, Ni Al$_2$O$_3$, PTFE, etc.) suspended in an electrolytic bath can be co-deposited during electrode position. Composite coatings produced by this technique enhance physical and mechanical properties such as wear and corrosion resistance as compared to the pure metal coatings. This technique involves no high-temperature or high-pressure process. Furthermore, the concentration and spacing of the particles in deposits can be controlled precisely by this method.

Ni Al$_2$O$_3$ and PTFE particles have many superior properties, such as low price, good chemical stability, high micro hardness and wear resistance at high-temperature. Because of its non-fouling, the deposition of NiPTFE composite coating on heat exchanger surfaces and heating elements can prevent the formation of lime scale.

This resulted in substantial cost savings. NiPTFE composite coating is usually prepared by electroless plating or traditional electroplating with very low deposition rate. The agglomeration of particles in coatings would result in poor mechanical properties to composite coatings. To solve the above problem, the use of fine particle of size 1 µm is used in my experimental work. Although many investigations have revealed that the addition of Al_2O_3 and PTFE particles on Ni-P matrix can promote the wear and high temperature corrosion resistance, the influence of these particles on the Ni matrix was not studied. Therefore, an attempt had been made to investigate the influence of the concentration of Al_2O_3 and PTFE particles on the Ni matrix and to improve the property of metal matrix through heat treatment.

II. EXPERIMENTAL WORK

Here, the general experimental set up of electro deposition process is shown in Figure 2.1

A. Experimental Set-Up of Electro Deposition System

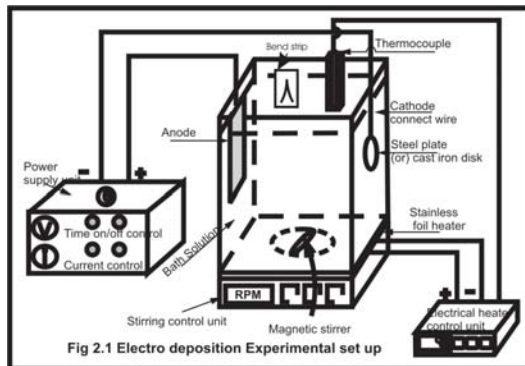Fig 2.1 Electro deposition Experimental set up

Fig. 1 Electro deposition Experimental set up

The bath solution is contained in the 10 mm thick Poly-glass with 5 mm thick FRP lining tank for the purpose of Electro-deposition with provision of anode and cathode. The size of the tank is 2.5m length,1m width and 1m height. The amount of bath solution is 2000 lit. The different bath composition and operating conditions are maintained according to the type of coating.

Anode and Cathode are connected with the rectifier by proper wire connection for electro-deposition purpose. The current input can be varied using dimmer stat in the control panel. Stainless foil heater is mounted at the bottom of the tank to heat the bath solution to attain the required temperature. A thermocouple is inserted into the bath to measure the bath solution temperature and hence maintain it depending on the type of coating. A copper bend strip unit is inserted into the bath solution to measure the internal residual stress of the coating.

Magnetic stirrer is needed for agitation to obtain uniform thickness of the coating. The speed of the magnetic stirrer is controlled by stirring control unit. The part to be electro deposited is connected with the cathode wire.HCL acid dip tank of six mm thick M.S. sheets with five mm thick FRP lining is used for pickling purpose ie each specimen is dipped in this tank for the removal of oil, grease, dirt etc., Water swill tank of six mm thick Poly-Glass with five mm thick FRP tank is used for washing and rinsing purpose after it is dipped in the HCL tank. For 2 µm thick coating, a current density of 10 A/dm2 can be used for 10 minutes. Hence, for getting 8 µm thick coating, 40 A/dm2 can be used for 10 minutes or 10 A/dm2 can be used for 40 minutes. By varying current density or the duration of coating, the coating thickness can be varied.

B. Specimen Preparation

Mild Steel was taken as specimen. Different samples of varying sizes were taken for the analysis. The samples are shown in Figure 2.2
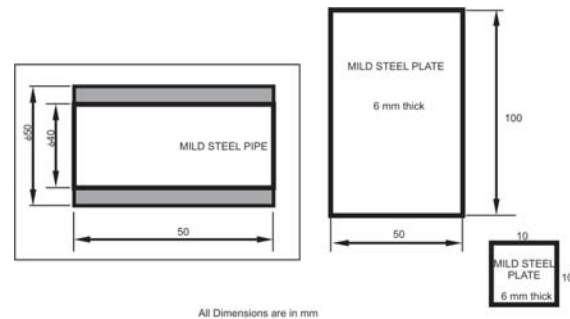

All Dimensions are in mm

Fig. 2. Sample Specimen

TABLE 1. BATH COMPOSITIONS AND OPERATING PARAMETERS

Ni deposition solution	
Nickel Sulphate $NiSO_4$	360 gm/l
Nickel Chloride $NiCl_2$	50 gm/l
Boric Acid H_3BO_3	45 gm/l
Temperature	55 ± 2°C
PH	4.6
Hard Chromium deposition solution	
Chromic Acid CrO_3	250 gm/l
Sulphuric Acid H_2SO_4	2.5 gm/l
Temperature	55 ± 2°C
pH	4.6
Ni – P (Electroless Nickel) Deposition Solution	
Nickel Sulfamate $Ni(NH_3SO_3)_2$	320 gm/l
Nickel Chloride hexahydrate $NiCl_2$	10 gm/l
Boric acid H_3BO_3	40 gm/l
Phosphorous acid P_3BO_3	10 gm/l
Temperature of bath	80°C

pH	5
Ni – Co Deposition Solution	
Nickel Sulphamate Ni (NH$_3$ SO$_3$)$_2$	300 gm/l
Nickel Chloride NiCl$_2$	6 gm/l
Cobalt (as sulphamate),	20 gm/l
Boric acid (buffer) H$_3$BO$_3$	30 gm/l
Sodium dodecyl sulphate [CH$_3$(CH$_2$)10CH$_2$OSO$_3$Na], (surfactant).	0.2gm/l
Temperature	55 ± 2°C
pH	4.5
Ni–PTFE solution	
NiSO$_4$6H$_2$O	25 gm/l
NaH$_2$ PO$_2$	30 gm/l
H$_3$C$_6$H$_5$O$_7$ 6H$_2$O	18 gm/l
NaCH$_3$ COO	18 gm/l
PTFE(60 wt.%)	40 gm/l
(NH$_2$)$_2$ CS	1 ppm
C$_{20}$ H$_{20}$F$_{23}$N$_2$O$_4$l (FC-4)	0.5 g/l
Temperature	60 ± 2°C
pH	4.8
Ni - Al$_2$O$_3$ Metal Matrix Composite electro-deposition Solution	
Nickel Sulphate NiSO$_4$	360 gm/l
Nickel Chloride NiCl$_2$	50 gm/l
Boric Acid H$_3$ BO$_3$	45 gm/l
Alumina particles size 1 μm	90gm/l
Temperature	55 ± 2°C
pH	4.5

C. Salt Spray Corrosion Test

In salt spray corrosion test, metal thickness loss was occurred corresponding to the corrosion scale formation and scale thickness loss. Degradation of the specimens was assessed by measuring the weight change per unit area as the extent of corrosion. The weight change was consisting of weight gain owing to the formation of oxide scale and weight loss due to the suspected spalling and fluxing of the oxide scale. The net weight change represented the combined effects of these two processes.

This investigation was made to evaluate the performance of the electro deposition coatings in NaCl salt environment. This knowledge of the Behaviour of these coatings under this most aggressive environment once evaluated would be helpful in choosing the suitable coating for the given environment.

The cyclic hot corrosion behaviour of these coatings was studied in NaCl salt at 900°C. The internal oxidation and cracking of scale was observed during the testing. The maximum protection was observed in case of Ni - Al2O3 and Ni-PTFE coating and was a minimum protection in case of bare specimen and then Ni coating.

D. Hot Corrosion Studies

Hot corrosion studies were conducted at 900°C in the laboratory using silicon carbide tube furnace, calibrated up to the variation of ±5°C. The uncoated samples were subjected to mirror polishing, whereas coated samples were subjected to wheel cloth polishing for 5minuites. The coated specimens were mirror polished down to 1 nm alumina on a cloth polishing wheel before the corrosion run. Thereafter, the samples were heated in the oven up to 250°C and then taken out. The salt mixture of NaCl (5 wt.% NaCl, pH of 7 at 35oC)dissolved in distilled water was coated on the hot polished samples(250oC) with the help of salt sprayer. The amount of the salt coating applied was 4mg/ cm2. The coated samples were then dried at 110°C for 34h in the oven to remove the moisture and then weighed.

Hot corrosion studies were carried out for 50 cycles. Each cycle consisted of 1h heating at 900°C followed by 20min cooling at room temperature. The weight of samples was measured at the end of each cycle by cleaning and removing spalled scale. The weight change measurements were taken at the end of each cycle using an electronic balance machine with a sensitivity of 1 mg. The spalled scale was removed at the time of measurement of the weight change to determine the total rate of corrosion.

The weight loss/unit area results for the six types of coating namely Ni,Ni-P,Cr,NiCo,Ni-PTFE,Ni-Al2O3 and bare specimen exposed to the NaCl salt mixture at 900oC for 50 cycles are found. The Ni-Al2O3 coating showed the minimum weight loss, whereas bare specimen showed the maximum weight loss and then Ni coating showed the maximum weight loss among all the coatings.

III. RESULTS AND DISCUSSION

A. Salt Spray Corrosion Test

Hot corrosion studies were conducted on bare as well as coated specimens after exposure to a NaCl salt environment at 900°C for specified time intervals. Here studies were conducted for 50 cycles - each cycle consisting of 1 hour heating in 900oC and 20 min cooling in Room temperature as per the ASTM standard B 117.

After washing with acetone, bare metal and coated metals were heated to 250°C in a muffle furnace and taken out. The sodium chloride salt was mixed with distilled water with a concentration of 500 gm/ lit. The salt solution was sprayed on all the specimen with the range of 3 5 mg/cm². Each salt coated specimen was placed on different crucibles. The weight of crucible and specimen were measured together with the help of Electronic Balance Machine Model 06120 (Petit) with a sensitivity of 1 mg. The crucibles along with specimen were kept in the furnace and heated to 900°C. The heated specimens were kept at 900°C for 1 hour followed by 20 min cooling at room temperature. This completes one cycle. After 5 cycles, the specimen were taken out and completely cleaned by special brush and weighed again along with crucibles by the same Electronic Balance Machine and the data was noted. Similarly, at every 5 cycles interval, the weight loss was measured. The data was collected for 50 number of cycles. The spalled scale was removed at the time of measurements of weight loss to determine the total rate of corrosion.

The studies were conducted for uncoated as well as coated specimens for the purpose of comparison. The overall weight change was nearly gradual. The overall weight changes after 50 cycles of hot corrosion for bare as well as Ni, Cr, Ni-P, Ni-Co, Ni-PTFE and Ni Al2O3 were found to be 13.438, 12.015, 8.021, 8.699, 8.261, 7.481 and 7.021 mg/cm² respectively. Evidently, the bare specimen lost the maximum weight during the observation. From the weight loss data, it can be inferred that all the coatings provided necessary hot corrosion resistance under given conditions. Among the electro deposition process, the Ni Al2O3 coatings have shown the highest hot corrosion resistance followed by Ni-PTFE, Cr, Ni-Co, Ni-P, Ni coatings.

The following tables show the weight loss data for different metal, composite coatings.

TABLE 3. SALT SPRAY CORROSION TEST- I

Number of cycles – 5		
Sl. No.	Resisting coating	Weight loss in mg/cm²
1	Bare	1.956
2	Ni	1.801
3	Cr	1.561
4	Ni P	1.702
5	Ni-Co	1.432
6	Ni-PTFE	1.450
7	Ni-Al$_2$O$_3$	1.045

TABLE 4. SALT SPRAY CORROSION TEST-X

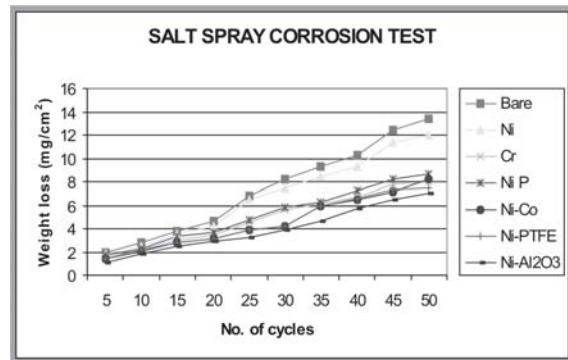| Weight loss after 50 cycles		
Sl. No.	Resisting coating	Weight loss mg/cm²
1	Bare	13.438
2	Ni	12.015
3	Cr	8.021
4	Ni-P	8.699
5	Ni-Co	8.261
6	Ni-PTFE	7.481
7	Ni-Al$_2$O$_3$	7.021

Fig. 3 Salt Spray Corrosion Test

of (weight loss/area) vs. number of cycles. It was observed from the graph that all the coatings followed a nearly parabolic rate law and thus they had a tendency to act like diffusion barriers to the corroding species. The results of salt spray corrosion test after 50 number of cycles were given in the Fig 4
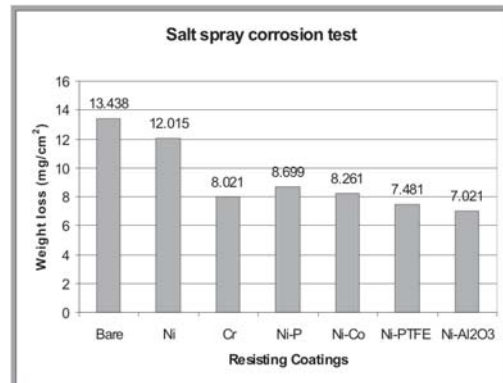

Fig. 4.Salt Spray Corrosion Test

The weight loss/unit area results for the six types of coating namely Ni, Cr, Ni-P, NiCo, Ni-PTFE, Ni-Al2O3 and bare specimen exposed to the NaCl salt mixture at 900oC for 50 cycles are shown in Fig 3.2. The comparison of weight loss for different types of coatings after 50 number

of cycles in the salt spray corrosion test can be done using figure 3.2. The Ni-Al2O3 coating showed the minimum weight loss, whereas bare specimen showed the maximum weight loss and then Ni coating showed the maximum weight loss among all the coatings. The composite coatings in all cases showed a much lower weight loss than the uncoated specimens in the given molten salt environment.

IV. CONCLUSION

The corrosion studies were conducted for uncoated as well as coated specimens for the purpose of comparison. The overall weight change was nearly gradual. Evidently, the bare specimen lost the maximum weight during the observation. From the weight loss data, it can be inferred that all the coatings provided necessary hot corrosion resistance under given conditions. Among the electro deposition process, the Ni-Al2O3 coatings have shown the highest hot corrosion resistance followed by Ni-PTFE, Cr, Ni-Co, Ni-P, Ni coatings. The Ni-Al2O3 coating shows the minimum weight loss, whereas bare specimen shows the maximum weight loss and then Ni coating shows the maximum weight loss among all the coatings. From the graph of (weight loss/area) vs. number of cycles, it was observed that all the coatings followed a nearly parabolic rate law and thus they had a tendency to act like diffusion barriers to the corroding species.

The composite coatings in all cases showed a much lower weight loss than the uncoated specimens in the given molten salt environment. During the course of the study, it was observed that the uncoated specimen underwent much spalling as well as sputtering. Sputtering of uncoated specimen occurred continuously during the period of cooling or even while keeping the sample inside the furnace. A rust-coloured compact and dense continuous scale was observed to be formed on the hot corroded Ni and Ni-P coatings and no spallation or peeling off of the scale was noticed. Hard chromium electroplating process yielded deep micro cracks. It was also well known that the increase in thickness enhanced the hard chromium protection to the salt spray corrosion.

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