

Study of Wear, Corrosion and SEM of Ni-Al₂O₃ Composite Coated Specimens

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Abstract— Electrodeposited composites are picking up significance for their points of interest including minimal effort, straightforwardness and effortlessness of operation. Composite electroplating is a technique for co de-positioning fine particles of metallic, nonmetallic mixes or polymers in the plated layer to enhance material properties, for example, wear resistance, hardness and erosion resistance. Electrodeposition has been distinguished to be a mechanically practical and for some applications, financially unrivaled procedure for the creation of Nano crystalline materials. With expanding accessibility of micron-sized particles of Al₂O₃, there is developing enthusiasm for the electrolytic and electro less co-affidavit of these particles. In this examination work an endeavor is made to create electrodeposited Ni – Al₂O₃ covering on a M S substrate by electroplating process. By altering the plating parameters, for example, current density, pH estimation of the electrolyte shower, measure of Al₂O₃ particles, fomentation pace and temperature of the shower, electroplating is done for the wear, SEM and corrosion examinations.

Key words: Mild Steel, Nickel, Aluminium oxide

I. INTRODUCTION

The metallic materials with better surface properties are required for the feature technology. We cannot meet the requirements by using single metal alone.

As a result many researchers have been giving importance to the composite materials with new structures and special functions.

Composites are prepared by different coating techniques some of them are given below [1].

- Thermal spray method.
- Physical vapor deposition.
- Chemical vapor deposition.
- Electro co-deposition.
- Electro less deposition etc.,

Composite coatings are coated in micro and Nano levels and some of the classifications of Nano coatings are [2].

- Metal matrix.
- Ceramic matrix.
- Polymer matrix.

From the above coating techniques electro co-deposition method is one of the widely used technique because when compared to other techniques it includes precise control, low energy requirement, uniform deposition, low cost in production of large area samples, good reproducibility, high production rate and reduction of waste [3].

The purpose of choosing this electro co-deposition technique is because it is one of the best methods to obtain metal matrix composite coatings in new technologies [4] Nickel is one of the best coating materials in the field of

marine since it enhances wear, corrosion and hardness [5] To prepare matrix of nickel by using Electrodeposition a wide range of ceramic particles have been used such as oxides like Al₂O₃, SiO₂, Cr₂O₃ and ZrO₂ [6-11], Carbides like WC [12], TiC [1]etc. Electrodeposition has been recognized to be a mechanically doable and for some applications, financially unrivaled method for the generation of Nano crystalline materials. The target of giving a covering to a substrate is to upgrade Engineering and physical properties of the substrate material or to acquire covering innovation has been utilized widely as a part of numerous assembling territories, for example, coatings for motor chambers, Textile factory rollers, Small size cams and apparatuses. In this work an attempt has been made to get a composite specimen of M S (mild Steel) coated with Ni-Al₂O₃ by considering some parameters such as pH of the electrolyte to 4, current density of 2 amps, electrode distance of 10 cm, stirrer speed of 250 rpm and amount of Al₂O₃ (3, 6 and 9 g).The main motivation behind this project is to get enhanced wear and corrosion properties of the M S specimen coated with Ni-Al₂O₃.

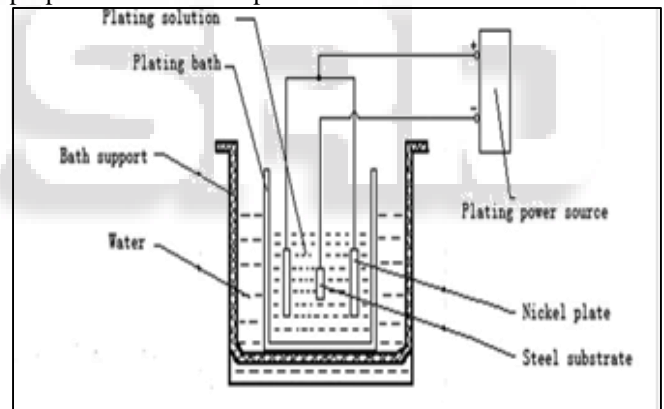


Fig. 1: Schematic diagram of the electro-co-deposition technique

A schematic diagram of a basic electro-co-deposition cell is shown in Fig1. Electro-co-deposition refers to the electrolytic process by which reduced metal ions together with ceramic or organic particle are deposited on a substrate, forming a composite structure of metal matrix dispersed by the particles. The purpose of electro-co-deposition process is to improve the surface properties such as corrosion protection, lubricity, wear resistance and coating thickness.

II. EXPERIMENTAL DETAILS

A machine is used for the deposition of Ni-Al₂O₃ on to the mild steel which is called as Electro deposition machine. It consists of following parts.

- Borosil tank of volume 4-5 liters with 2-3 mm of wall thickness which can withstand temperature of 800C to use a plating cell.

- Jacketed tank, made out of 2mm ss304 material in which the glass tank will be placed and hot water will be circulated in the SS jacketed tank to maintain the temperature of the plating solution in the tank by indirect heating method.
- An instant electric geyser of volume 2-3 liters to heat up the water in the above jacketed tube.
- Digital temperature controller with a PT-100 probe placed in the process tank to control and monitor the process temperature.
- Rectifier of capacity 6 volts x 10 Amps CC and CV control provision digital voltmeter and digital ammeter with a current resolution of 0.1 Amps.
- Electrical stirrer with non-metallic impeller to agitate the plating solution.

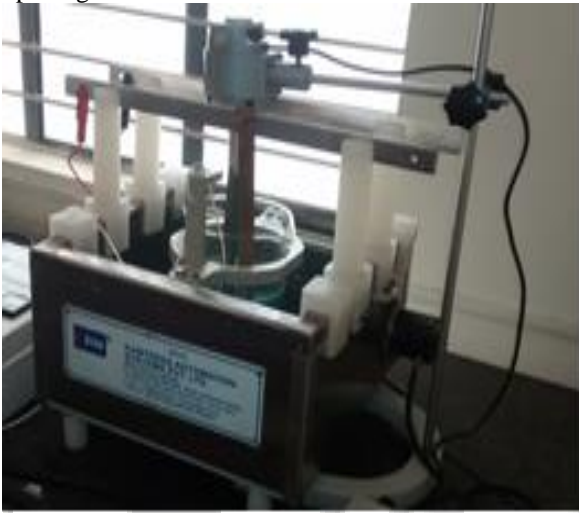


Fig. 2: Electro co- deposition setup.

III. PREPARATION OF SAMPLES

The specimens of mild steel material of cylinder pins are prepared for wear test of length 25mm and diameter of 10mm according to the ASTM G-99 standards by using by using a lathe machine.

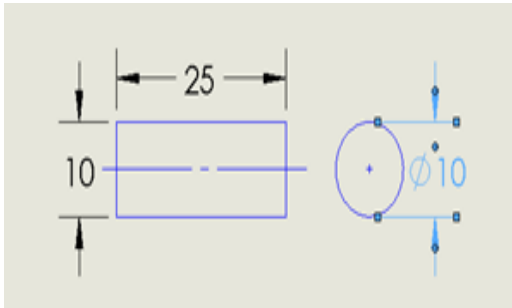


Fig. 3: wear test MS specimen

These pins are fitted to a pin on disc wear testing machine. The specimen's surface is first flattened by using facing operation on a lathe machine, and then by using emery paper smooth finish is given to the specimen. Followed by the machining operations the wear specimen is cleaned with soap water to remove any oily substances on the specimen followed by cleaning in an ultrasonic cleaner to remove small dirt particles which are settled on the surface of the specimen. The specimen is then cleaned with acetone to remove water particles, further at room temperature the specimen is rinsed in 5% sulfuric acid for 5 minutes. Then finally the specimen

is rinsed in distilled water for 5 minutes. Before stating the coating the initial readings of the specimens are noted down.

IV. PREPARATION OF BATH SOLUTION

At first the above mentioned chemicals are weights and added in to a beaker and then the solution is stirred by an electric stirrer for one hour for homogenous mixing of the chemicals later the beaker is kept in the container in which water is present which can be heated by using a heater through which the electrolyte gets heated by means of an indirect heating and the pH of the electrolyte is maintained at 4 by using equal proportions of Boric acid and sodium hydroxide and at required temperatures of the electrolyte the coatings are performed.

Chemical	Volume
Distilled water	1.5 liters
Nickel sulphate	375 grams
Nickel chloride	52.5 grams
Boric acid	60 grams
Aluminium oxide	3, 6 and 9 grams
NaoH	110 ml

Table 1: Electro co-deposition coating bath composition of Ni-Al₂O₃ composite coatings

V. COATING METHODOLOGY

The coating in this project is performed at the followed conditions which are as shown in the below table 2

Parameter	condition
Electrode distance	10cms
pH	4
Temperature	40°C
Aluminium oxide	3,6 and 9 grams
Stirrer speed	250 rpm
Current density	2 A
Time taken for coating	1 hour

Table 2: Conditions for considered for performing the coating operation.

After achieving the required temperature of the electrolyte the electrode which has nickel at its end is made as an anode and the other electrode which has mild steel wear specimens will act as a cathode. The specimens of the cathode are connected in pentagon shape by means of a copper wire and attached to the cathode. The fig of the same is as shown in the fig below.



Fig. 3: M S specimens for coating

Then by maintaining the above mentioned parameters in the table 2 the coating is performed for a life span of one hour. After the coating the cathode is removed on which the Nickel with aluminium oxide is deposited and cleaned with distilled water and dried in sun for 15 min to get good coating. This test is conducted at different volumes of aluminium oxide Say 6 and 9 grams and the wear test and corrosion tests are performed on these specimens and compared with base material.

VI. WEAR TEST

The wear test is done on a pin on disc machine which consists of a rotating part and a digital display. At first a disc is fixed on the rotating part of the machine and then initially weighed specimen is attached on the disc at a certain length (L=300mm). Then setting the time to 3 min and rotating speed of the machine to 300rpm and making the frictional force to zero and further applying a load of 10N, 20N, 30N, 40N. The test has been conducted and the weights of the specimens after wear and frictional force are tabulated. The fig of the pin on disc wear machine is as shown below.



Fig. 4: Pin on disc wear machine.

Assumptions that have been considered to conduct this test are as follows

- Sliding distance (SD) = 300mm.
- Speed of the rotating disc (N) = 300rpm.
- Diameter of the pin (D) = 100mm.
- Length of the pin (L) = 25mm.

Formulae used for this test:

Time considered to conduct wear test is given below

$$t = \frac{SD}{\pi \cdot D \cdot N} = \frac{300}{\pi \cdot 0.1 \cdot 300} = 3.18 \text{ min.}$$

$$\text{Wear rate} = \frac{\text{volume loss}}{\text{Load} \cdot \text{SD}}$$

Where, Volume loss = Weight difference / Density.

VII. CORROSION TEST

In this method pH of the solution prepared is maintained below 7. The chemicals used for this test are liquid sodium chloride (NaCl) and Sulphuric acid (H₂SO₄). In this project we are considering acidic corrosion method and maintaining a pH of the solution at 2. The specimens to be tested are cleaned with Clark's solution to remove any unwanted foreign particles on the surface of the coated specimens and initial weights of each specimen are tabulated. The cleaned coated specimens are kept in small cups which are shown in the fig, and then the acidic solution is poured on it and with a time intervals of 1 day, 2 days, 3 days and 4 days the specimens are removed and cleaned with Clark's solutions and their weights is tabulated. Then by using below formulae the corrosion rate is calculated.

$$\text{Corrosion rate} = \frac{534 \cdot W}{D \cdot A \cdot T} \text{ (Area in inches) or}$$

$$\text{corrosion rate} = \frac{87.6 \cdot W}{D \cdot A \cdot T} \text{ (Area in cm}^2\text{)}$$

Where,

W = Weight difference in mg.

D = Density in gram/cc.

A = surface area of the specimen.

T = Time in hours.

Corrosion rate will be in mm/year.

VIII. SCANNING ELECTRON MICROSCOPE (SEM)

Scanning Electron Microscopy (SEM) investigations the surfaces of materials, particles and filaments with the goal that fine subtle elements can be measured and surveyed through picture examination. SEM gives a way to industry to determine pollution issues, explore segment disappointment, recognize obscure particulates or study the association amongst substances and their substrates. It can likewise give an abundance of data to bolster exploration of materials, chemicals or natural specimens. The procedure of deciphering SEM pictures is not generally clear and direct. In errands, for example, the translation of surface setting on metal segments the ID of particulates, or the investigation of physical and substance qualities of material, SEM turns into a genuinely effective procedure if fitting specimen readiness strategies are utilized and experienced microscopists perform the examination.

IX. RESULTS AND DISCUSSIONS

A. Wear Test Results and Discussions

1) Ni- Al₂O₃ (3g) coated M S specimen

Initial weight = 14.1g

Final weight = 13.9g

Force in N	Time in min	Wear rate in mm ³ /N-m
10	3	0.0092
20	6	0.0046
30	9	0.0031
40	12	0.0023

Table 3: Wear in Ni- Al₂O₃ (6g) coated M S specimen

2) Ni- Al₂O₃ (6g) coated M S specimen

Initial weight = 16.2g

Final weight = 16.1g

Force in N	Time in min	Wear rate in mm ³ /N-m
10	3	0.0040
20	6	0.0020
30	9	0.0013
40	12	0.0010

Table 4: Wear in Ni- Al₂O₃ (6g) coated M S specimen

3) Ni- Al₂O₃ (9g) coated M S specimen

Initial weight = 15.2g

Final weight = 15.1g

Force in N	Time in min	Wear rate in mm ³ /N-m
10	3	0.0043
20	6	0.0021
30	9	0.0014
40	12	0.0011

Table 5: Wear in Ni- Al₂O₃ (6g) coated M S specimen

4) Non coated M S specimen

Initial weight = 14.8g

Final weight = 14.4g

Force in N	Time in min	Wear rate in mm ³ /N-m
10	3	0.0177
20	6	0.0088
30	9	0.0058
40	12	0.0044

Table 6: Wear in Ni- Al₂O₃ (6g) coated M S specimen

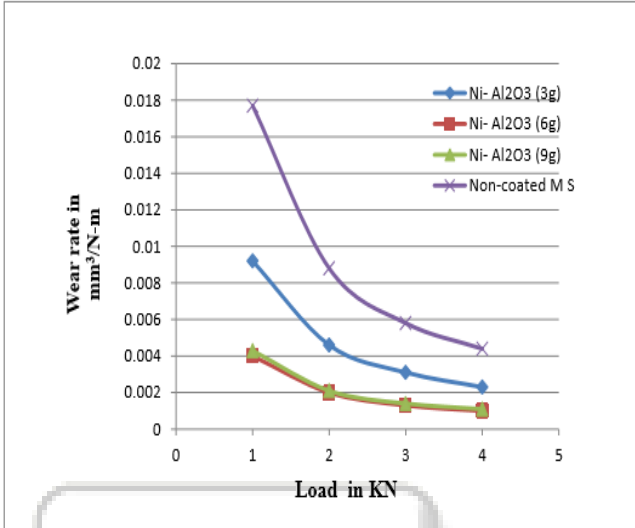


Fig. 5: Graph for comparison between non coated and coated specimens

From the above graph 5 it is clear that the wear rate is minimized by coatings i.e. when compared with non-coated M S and coated M S specimen and is better for 6 and 9 g of Al₂O₃.

Corrosion test results and discussions

Material	pH	Initial wt.	Wt. after 24 hrs.	Wt. after 48 hrs.	Wt. after 72 hrs.
Non-coated	2	14.330	14.324	14.304	14.221
Ni-Al ₂ O ₃ (3g)	2	15.549	15.544	15.534	15.515
Ni-Al ₂ O ₃ (6g)	2	15.218	15.215	15.211	15.207
Ni-Al ₂ O ₃ (9g)	2	15.317	15.314	15.310	15.306

Table 7: Corrosion test observations.

Corrosion rate in different coated M S specimen

Material	Corrosion rate after 24hrs in mm/year.	Corrosion rate after 48hrs in mm/year.	Corrosion rate after 72hrs in mm/year.
Non-coated	14.324	14.304	14.221
Ni-Al ₂ O ₃ (3g)	15.544	15.534	15.515
Ni-Al ₂ O ₃ (6g)	15.215	15.211	15.207
Ni-Al ₂ O ₃ (9g)	15.314	15.310	15.306

Table 8: corrosion rate in different coated m s specimen

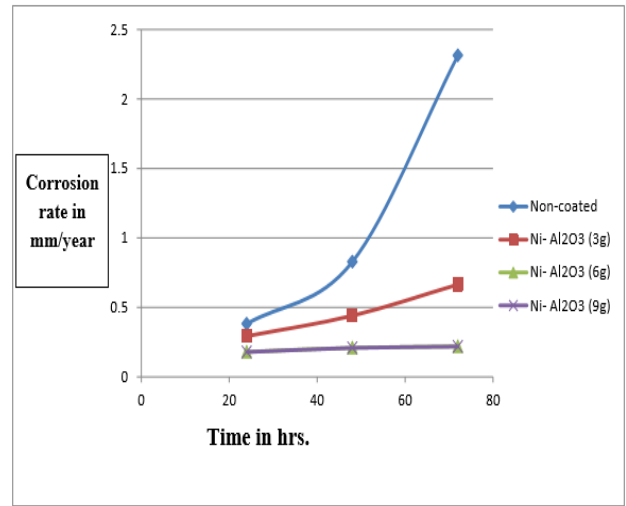


Fig. 6: Graph for comparison between non coated and coated specimens of corrosion test

From the above graph it is clear that the rate of corrosion has been minimized and we can observe that, with the increase in the amount of Al₂O₃ the corrosion resistance is more.

X. SEM RESULTS AND DISCUSSIONS

A. SEM Results for Ni- Al₂O₃ (3g) Coated M S Specimen

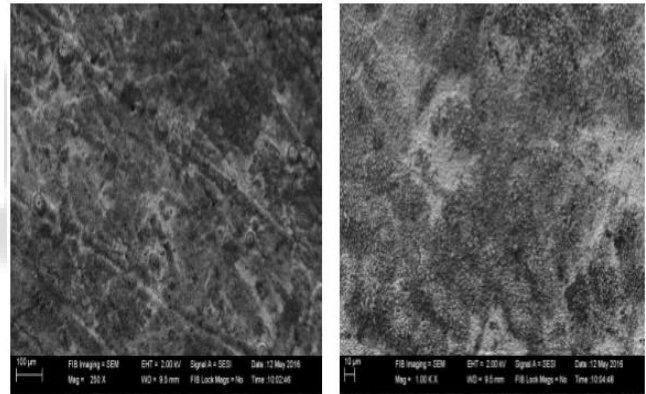


Fig. 7: SEM image of Ni- Al₂O₃ (3g) coated specimen 100µm and 10µm magnification

For the Ni- Al₂O₃ (3g) coated m s specimen the rate of uniform deposition is poor it can justified from the above fig 8.

B. SEM Results for Ni- Al₂O₃ (6g) Coated M S Specimen

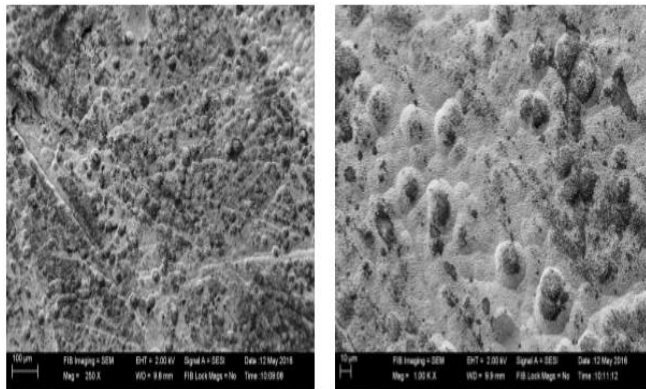


Fig. 8: SEM image of Ni- Al₂O₃ (6g) coated specimen 100µm and 10µm magnification.

For the Ni- Al₂O₃ (6g) coated M S specimen the rate of uniform deposition is better when compared to Ni- Al₂O₃ (3g) coated M S specimen and it can justified from the above fig 9.

C. SEM Results for Ni- Al₂O₃ (9g) Coated M S Specimen

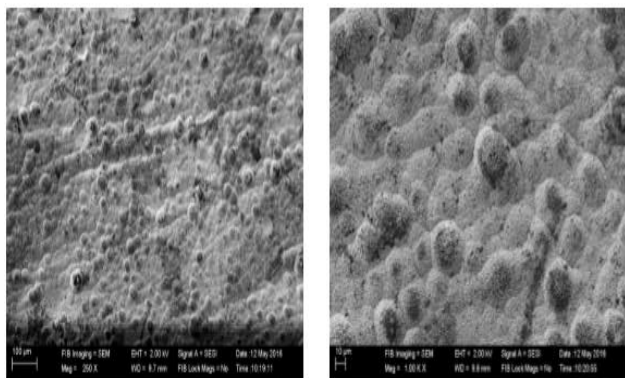


Fig. 9: SEM image of Ni- Al₂O₃ (9g) coated specimen 100µm and 10µm magnification.

For the Ni- Al₂O₃ (9g) coated M S specimen the rate of uniform deposition is good when compared to other coatings and it can justified from the above fig 10.

XI. CONCLUSION

From this project I came to a conclusion of the following things

- The wear resistance is more for the coatings where addition of aluminium oxide content is more.
- The corrosion rate is more for the base material and it has been minimized by NiAl₂O₃ (6 & 9g) coating on M S specimen.
- Uniform coatings have been achieved for Ni-Al₂O₃ (6g & 9g) coated M S specimens.

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