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BEHAVIOUR OF COMPOSITE MATERIALS IN NICKEL-PHOSPHORUS-BORON DEPOSITS ON MILD STEEL

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ABSTRACT

The bath composition for deposition of nickel-phosphorus-boron has been standardized as reported earlier. The present study consists of incorporation of silicon carbide, tungsten carbide, boron-carbide and alumina in Ni-P-B alloy deposit on mild steel with and without current. The operating conditions like current density, temperature, pH and time to get uniform deposit were standardized. The physical properties of Ni-P-B plus composite coated mild steel panels were determined and the structure of the deposit was examined under SEM. The corrosion resistance of the coating was evaluated by electrochemical measurements and weight loss. It was shown that incorporation of particular material improves the corrosion resistance of Ni-P-B coatings on mild steel.

INTRODUCTION

Electrodeposition of amorphous nickel alloy deposits is a part of the metal surface amorphidization scheme for steel surfaces. In this endeavour, it has been seen that incorporation of more number of metalloids may substantially increase the corrosion and wear resistance, along with the hardness of these deposits. It was earlier shown that amorphous nickel alloy containing 13% phosphorus [1] or phosphorus and boron together [2] could be electrodeposited satisfactorily from the conventional plating baths.

However, the possibility of incorporation of these deposits with materials like alumina, boron carbide, tungsten carbide, silicon carbide, molybdenum, silicon, titanium carbide, titanium boride etc. for higher abrasion resistance in moving parts at elevated temperatures meets the demands for newer materials [3-9] in the sophisticated technological applications like aeronautical and space engineering, defence and nuclear engineering.

The codeposition [9] of alumina, polycrystalline diamond, silicon carbide and polytetrafluorethylene, has become commercially popular. These composite coatings are also finding applications in a variety of machinery parts in the textile industries, automobile, water pumps and chemical process pumps.

It has also been reported [9] that nickel-silicon carbide composite reveals better abrasion resistance characteristics above 300° C than nickel-diamond composites. Composites with silicon carbide showed deterioration above 400° C [9].

EXPERIMENTAL PROCEDURE

1. Pretreatment

Mild steel specimens (7.5 cm x 2.5 cm x 3 mm thick) are polished to −000 emery and degreased in trichloroethylene before electroless plating. For electroplating experiments, the specimens are cleaned in the alkaline solution comprising of sodium hydroxide (25 g/l) and sodium carbonate (35 g/l) at 50° − 60° C, using a current density of 4 to 5 A/dm^2. They are first treated cathodically for a few seconds and then anodically for one minute and washed thoroughly in running water. The specimens are subsequently dipped in dilute hydrochloric acid solution and then washed thoroughly in distilled water before the electroplating operation.

2. Preparation of plating bath

Both electroless and electrodeposited composite coatings are obtained on the mild steel substrates using a special type of glass container. The bath composition and operating conditions are given in Table I.

Table I: The bath composition and operating conditions

Nickel sulphate	150 g/l
Nickel chloride	45 g/l
Phosphoric acid	180 ml/lit
Sodium hypophosphite	135 g/l
Sodium borohydride	**6.5 g/l**
Particulate materials g/l	a) 15 SiC + 15 Al_2O_3 b) 5 SiC + 5 Al_2O_3 c) 5 SiC + 5 BC + 15 Al_2O_3 d) 5 SiC + 5 BC + 5 WC + 5 Al_2O_3 e) 5 SiC + 5 BC + 5 WC + 15 Al_2O_3

Operating condition	Electroless	Electroplating
Temperature	80° C	80° C
pH	3–4	3–4
Time	1/2 hr to 2 hrs	1 hr
Additive	d and e are used	only e is used
Current density A/dm^2	–	5–30
Thickness of the coating μm	10–12	31–45
Appearance	Dull bright	Grey

Mild steel as cathode is placed in between two nickel anodes. The electrolyte suspension is constantly stirred during deposition.

The electrolyte suspension is prepared by dissolving nickel sulfate, nickel chloride and sodium hypophosphite in distilled water followed by the addition of known volume of phosphoric acid. Then weighed amount of sodium borohydride is added slowly with stirring and the whole bath is allowed to age overnight to get a clear solution. The pH of the bath is corrected to 2 and afterwards to 3 – 4, either with phosphoric acid or freshly prepared nickel hydroxide. Finally, the weighed amount of particulate materials is added to the bath and thoroughly mixed.

3. Determination of physical properties

a) Thickness

Thickness of the plated mild steel panels is measured in six different points at random by using pencil type magnetic thickness meter and the mean value is reported.

b) Hardness

Hardness values are obtained on four different coated panels in V P N scale.

4. Electrochemical studies

The plated panels are immersed in 1.5 % NaCl solution. The change in potential of the plated panels with time are recorded with reference to S.C.E. using a microvoltmeter. An anodic current (2 mA – 100 mA) was impressed with the help of auxiliary electrode and the change in potential with reference to SCE was recorded.

5. Examination of the coated surface in the Scanning Electron Microscope

Electroless and electrodeposited Ni-P-B coatings with – particulate materials of SiC, BC, WC and Al_2O_3 are examined in the scanning electron microscope (JEOL/SEM 35 CE). The topographic and composition images are obtained with back scattered electrons. Secondary electron images of the surface texture are also taken.

6. Corrosion test

Specimens of identical shape, coated with bee's wax resin so as to expose 2.5 x 2.5 cm area are immersed in 10 % HCl solution for 2 hours. The corrosion rate values are obtained from the difference in initial and final weights after cleaning the corroded specimens.

7. Thermal stability of the composite coating

Both electroless and electrodeposited composite coatings on mild steel plates are subjected to soaking for a period of 45 minutes at 400°, 450°, 600°, 650° and 800° inside a muffle furnace. The plates are taken out from the furnace, after the stipulated period and visually observed.

RESULTS AND DISCUSSION

The Ni-P-B bath [2] was used for the composite electroless and electroplated deposits using particulate materials like SiC, BC, WC and Al_2O_3 in the ratio of 1 : 1 : 1 : 1 and 1 : 1 : 1 : 3. Table I (refer page no. 00) reveals the bath parameters like pH, time of plating temperature percentage of particulates as a function of the thickness, appearance and corrosion resistances of the coated specimens. It is seen that a pH of 3-4 and temperature of 80° results in an adherent dull bright deposit for both the particulate ratios mentioned earlier. Coating thickness increases with plating time. However, the electroplating experiments could not be conducted at current density above 30 A/dm^2 due to excessive boiling of the bath. At this current density, the coating thickness obtained is maximum.

Table II shows the physical properties of the composite coatings.

Table II : Physical properties of the coating. (Bath composition as in Table I)

No.	Additives in the bath g/l	Duration (hr)	Current density A/dm^2	Thickness (μm)	Hardness (V P N)	Corrosion rate (g/sq.mm) x 10^{-4}	Surface appearance
1.	nil	1/2	15	40	1200	1.33	Uniform, adherent, bright
2.	5 SiC 5 WC 5 BC 1.5 Al_2O_3 (Total particulates in the coating = 20 %)	1	15	31	1400	0.20	Uniform, grey colour
3.	– do – (Total particulates in the coating = 37 %)	2	Electroless	12	1400	0.33	Dull bright uniform, adherent
4.	5 SiC 5 WC 5 BC 5 Al_2O_3 (Total particulates in the coating = 33 %)	2	"	10	1000	0.55	Lustrous grey and adherent

It is seen that hardness value is increased with the incorporation of particulate materials. The electroless (and electroplated) composite coating from the high alumina bath reveals the presence of higher percentages of lower atomic weight species (e.g. boron, phosphorus etc) in comparison with that of low alumina bath. The distribution of composite materials in the matrix is more uniform in the deposit obtained from high alumina bath.

Table II shows the corrosion rate values of the coatings with and without particulates. It is seen that that the incorporation of particulate materials in the coating has decreased the weight loss corrosion rate values to a considerable extent. The electroplated composite coating reveals minimum corrosion rate values, although of lower particulates content. This may be attributed [10] to the total higher content of lower atomic weight species (phosphorus and boron) as shown in the composition profile.

Thermal stability of the electrodeposited composite coating remains unaffected up to 600°C, while electroless composite coatings fail at around 350°C. The variation of potential with time of the nickel composite immersed in 1.5% NaCl was recorded (Fig. 1).

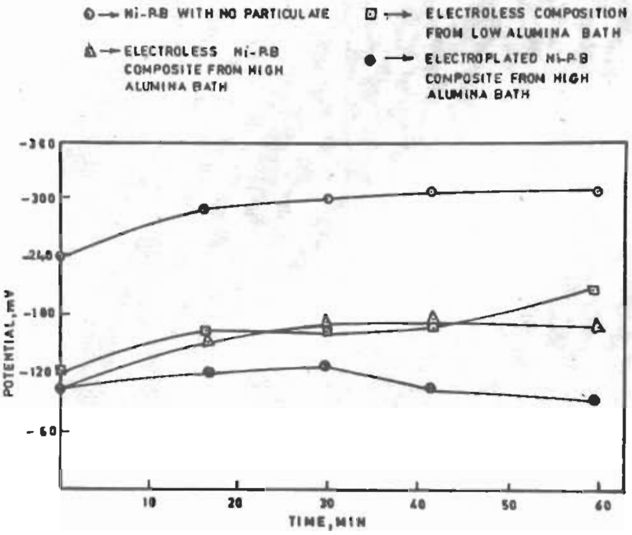

Fig. 1: Potential variation with time in 1.5% NaCl solution

It is seen from Fig. 1 that Ni-P-B deposit without particulate gave more negative potential than the composite coating. Addition of particulate in the deposit gave more positive potential. Anodic polarisation of the deposited metal in 1.5% NaCl is shown in Fig. 2.

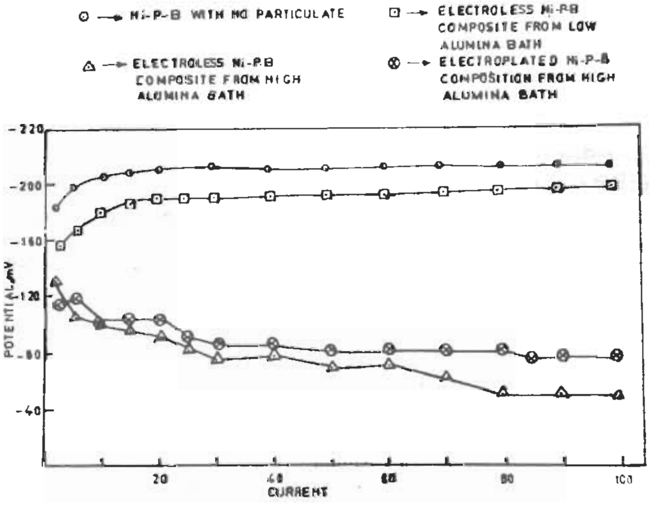

Fig. 2: Anodic polarization of composite nickel deposits in 1.5% NaCl solution

It is seen that in all the cases very little polarisation is observed. The composite deposit with high alumina content showed more positive potential than low alumina composite coatings.

The surface characteristics and composition profile have been studied in the Scanning Electron Microscope (Fig. 3–6). It is seen from Fig. 3 that crystal boundaries are not present there.

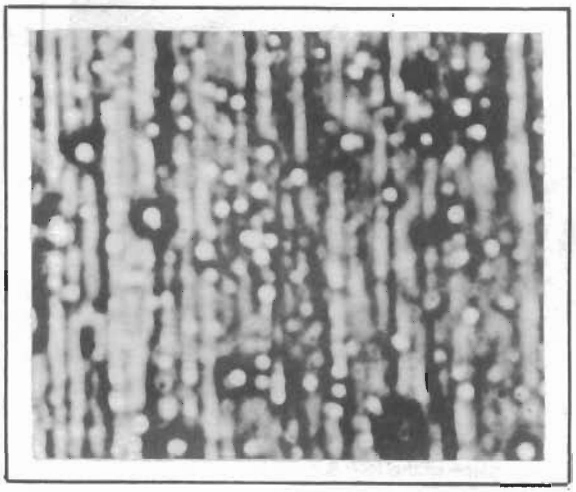

Fig. 3: SEM photograph of the Ni-P-B coated surface X 200

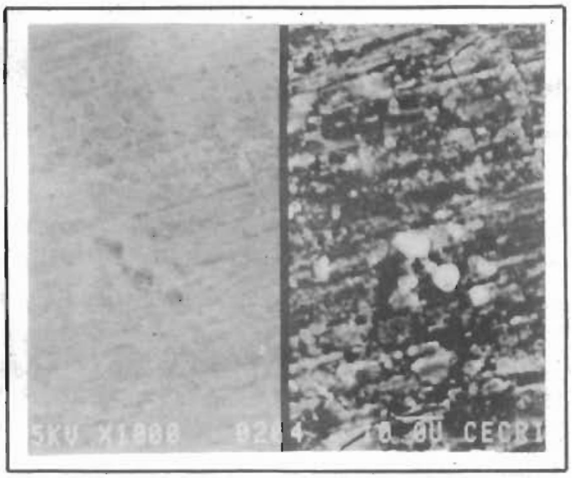

Fig. 4: SEM photograph of the Ni-P-B composite electroless surface from high alumina bath X 1000

In Fig. 4 it is seen that the roughness is considerably reduced. It also shows the presence of low atomic weight species (B, P) [10] in the composition profile. Fig. 5 reveals the topography and secondary electron image of the composite surface obtained from low alumina bath. The non-uniformity of the composite distribution is quite apparent. Fig. 6 shows the secondary electron image and the composition profile for the elctroplated composite coatings. It reveals the higher amount of low atomic weight species in comparison with Fig. 4 (Electroless coating).

CONCLUSIONS

It is concluded that incorporation of particulate materials in N-P-B coatings increases corrosion resistance characteristics. The electrodeposited composite coatings retain the resistance to high

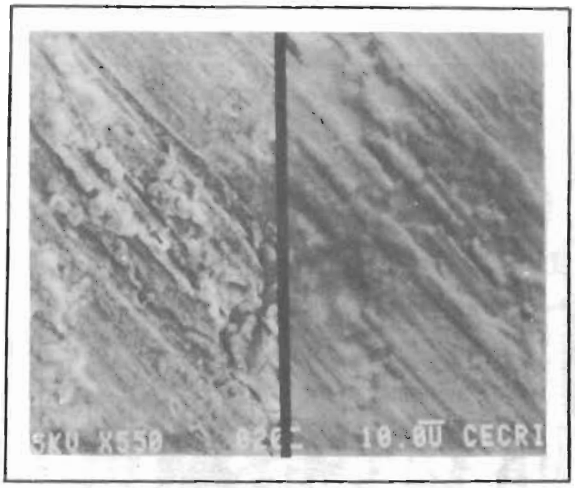

Fig. 5 : SEM photograph of the Ni-P-B composite (Electroless) from low aluminium bath X 550

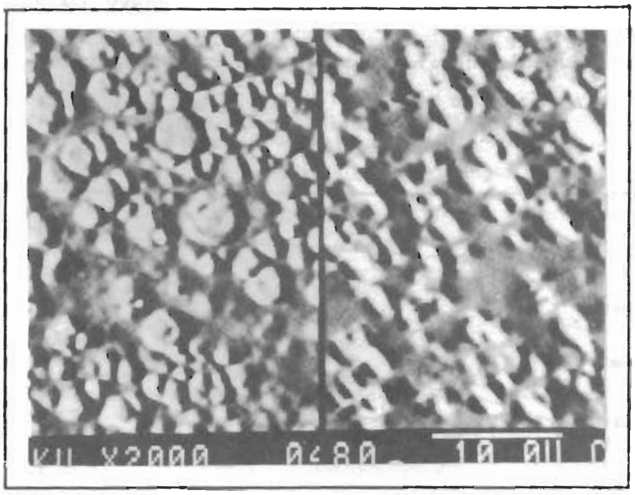

Fig. 6 : SEM photograph of the Ni-P-B composite (Electroplated) high alumina bath X 2000

temperature in comparison with that of the electroless coatings. The hardness of the composite coatings is also increased. The high alumina bath gave good uniform deposit.

Acknowledgement

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