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## **Incorporation of Super Fine Particles in Ni-P Deposits on Low Carbon Steel**

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**Abstract** Electroless nickel-phosphorus (EN) coating is a chemical reaction process results in a reduction of the metallic ion. It depends on catalytic action. In this work, several samples of base metal were coated with (Ni-P) deposit. Then nano-sized (40nm SiC) and micro-sized (11.22 $\mu$ m SiC) particles were added separately to the bath to be incorporated into the coating layers. Coated samples were annealed in a vacuum furnace at a temperature at (400 °C) for a period of one hour. Several tests were conducted such as, XRD, SED, microhardness and wear test. Coating layers were uniform and microcrack-free. Significant improvements in hardness and wear resistance were observed. Heat treatment introduce further increase in hardness. The morphology of the hard compound (Ni<sub>3</sub>P) deposits was global and spherical.

**Keywords** Electroless nano-composite coating; microhardness; wear resistance.

### **1. Introduction**

Electroless plating is a chemical reduction process, which depends on the catalytic reduction of a metallic ion from an aqueous solution containing a reducing agent, and the subsequent deposition of the metal without the use of electrical energy [1]. Electroless nickel-phosphorus is widely used in chemical, mechanical and electronic industries because of its corrosion, wear resistance and its inherently uniform coating thickness. In recent years the electroless nickel process has been used in a wide variety of applications in aerospace, automotive, electronics, computers, machinery, nuclear, oil, gas production and various industries [2]. Brenner and Riddell invented electroless Ni plating in 1946 [3], rather accidentally when they observed that the additive (NaH<sub>2</sub>PO<sub>2</sub>) caused apparent cathode efficiencies of more than 100% in a nickel electroplating bath [4]. Other outstanding characteristics of EN coatings include the ability to be applied to a variety of substrate materials and the ability to be plated uniformly on intricate part geometries. The phosphorus content of deposit defines the physical, mechanical and corrosion resistance properties of the coating [5]. There are three types of EN coatings available based on the weight percentage of phosphorus: (i) low phosphorus (2 to 5%), (ii) medium phosphorus (6 to 9%) and (iii) high phosphorus (10 to 13%). Based on the presence of phosphorus content the structure may be microcrystalline, amorphous or a combination of both [5-6]. EN coating takes special interest function to the best or fine wear and corrosion resistance, thermal conductivity and high electrical. Several researches emphasize that the properties of lubricity and anti wear of substrate material improved by a co-deposit coating in the presence of fine ceramic particles, such as Al<sub>2</sub>O<sub>3</sub>, SiC, and diamond or PTFE, graphite and MoS<sub>2</sub> within an electroless nickel matrix [7-8]. One of the widely used coating materials is SiC, a high band gap semiconductor which is also a high strength ceramics. Most of its applications are connected to its good resistance against temperature and chemical effects [9]. In the present study, a high phosphorus Ni-P was coated on low steel. The mechanical properties and wear behaviour was studied. Incorporation of nano-particles in electroless Ni-P alloy coating would be significant for broadening the scopes of the coating in engineering, because various types of



nano- particles have special natures much different from that of bulk counterparts and could endow the coating with special functionality [10].

The present work aims to investigate the mechanical effects of incorporation of nano and micro-sized particles in Ni-P coatings deposited on low carbon steels.

## 2. Experimental Method

### 2.1 Materials

Samples used were low carbon steel sheets (11.78) mm diameter and (2-2.5) mm thick, Table (1).

**Table 1:** Chemical composition (weight %) of base metal

C	Si	P	Mn	Fe
0.207	0.265	0.017	0.843	Bal.

### 2.2 Bath composition and operating conditions

**Table 2:** Composition of the used bath

Composition	Concentrations
NiSO <sub>4</sub> .6H <sub>2</sub> O	20 g/L
NaH <sub>2</sub> PO <sub>2</sub> .H <sub>2</sub> O	24 g/L
C <sub>3</sub> H <sub>6</sub> O <sub>3</sub>	10 ml/L
C <sub>3</sub> H <sub>6</sub> O <sub>2</sub>	5 ml/L
Super-Fine SiC	12 g/L

The pH value of the as prepared bath was between 4-6. Adjustment of the pH was carried out by addition of NaOH. Reaction temperature was maintained thermostatically between 85-90°C.

### 2.3 Surface pretreatment

Specimen surface was carefully grinded and polished with emery paper (tungsten oxide paper). Specimens were then immersed in alkaline solutions (10 % NaOH) for 2 min at 75 °C. The electrolyte was used magnetic stirring for stirring to remove any dust and oil from the surface of the metal. Specimens were washed with distilled water. Further treatment was carried out by 30 % hydrochloride acid at 20-25 °C for 2 min. Specimens were then dried and measured.

### 2.4 Heat treatment

Specimens were heat treated in a vacuum furnace at 400 °C for a period of 1 h. Scanning electron microscope was used for metallographic examination.

### 2.5 Mechanical Tests

#### 2.5.1 Hardness Test

Vickers microhardness was measured by using TH-717 Vickers hardness tester and, it was used to determine the hardness of the coating layers. The applied load was 50 g and the duration time was 15 sec.

#### 2.5.2 Wear Test

Pin-on-disc technique used to investigate the wear behaviour of the coating layers. The weight loss per time was recorded every period of 5 min. The applied load was 1 N and the rotating speed was 100 rpm, disc of stainless steel, of hardness (800 HV).

## 3. Results and Discussion

### 3.1 Surface morphology and Microhardness behaviour

Incorporation of super-fine particles nano-SiC and micro-SiC particles were embedded separately in the coating layer. In both case a value of 12 g was added to the bath. However, the EDS analysis gives Ni (69.16 %), P (12 %) and Si (18.84 %) mass fraction, in the coating layers (figure 1).



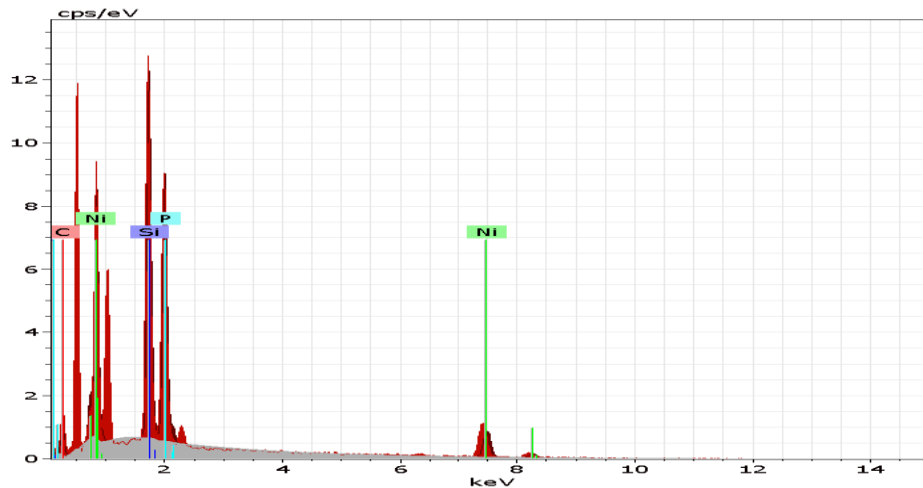


Figure 1: EDS spectrum of electroless (Ni-P-SiC) composite coating

Substantial improvement in microhardness of the coating layer was observed compared to that of the base metal (table 3). Nano-sized composites caused higher increase in hardness compared with the micro-sized particles.

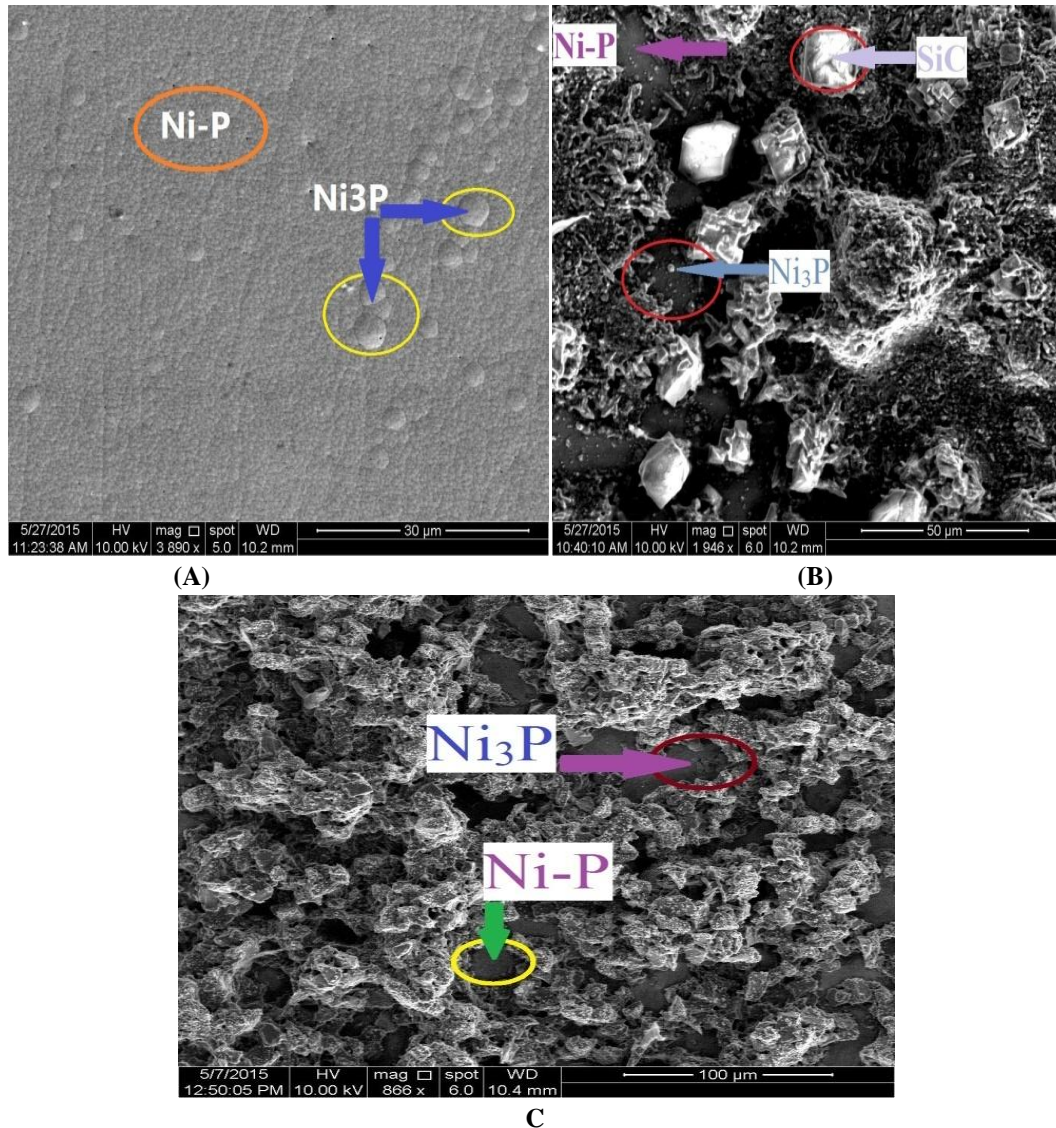


Figure 2: Ni-P and SiC observed by SEM.



**Table 3:** Shows of results of microhardness test

Type of Coating	HV	HV	$(H_C / H_A) \%$
	Before Heat Treatment	After Heat Treatment	
Low Carbon Steel	230	---	---
Ni – P	683	887	385.6
Ni - P – micro SiC	786	904	393.04
Ni - P – nano SiC	1024	1196	520

**A = carbon steel, C = coating layer**

### 3.2 Heat treatment

It was carried out at 400 °C for a period of 1 h. Metallographic examination and XRD shows the precipitation of the hard compound deposits of ( $Ni_3P$ ). As shown in figure 2, 3 and 4, it appears as global spherical deposits. A significant further improvement in microhardness was observed (table 3). This increase in hardness is attributed to dispersion hardening produced by the incorporated super-fine particles and to the precipitation of the hard compound ( $Ni_3P$ ).

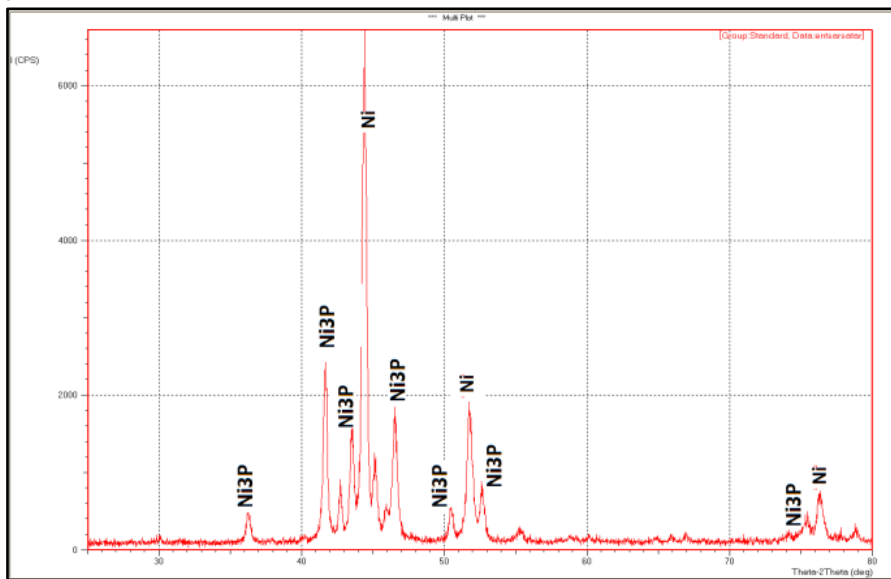


Figure 3: XRD patterns of (Ni-P) coating.

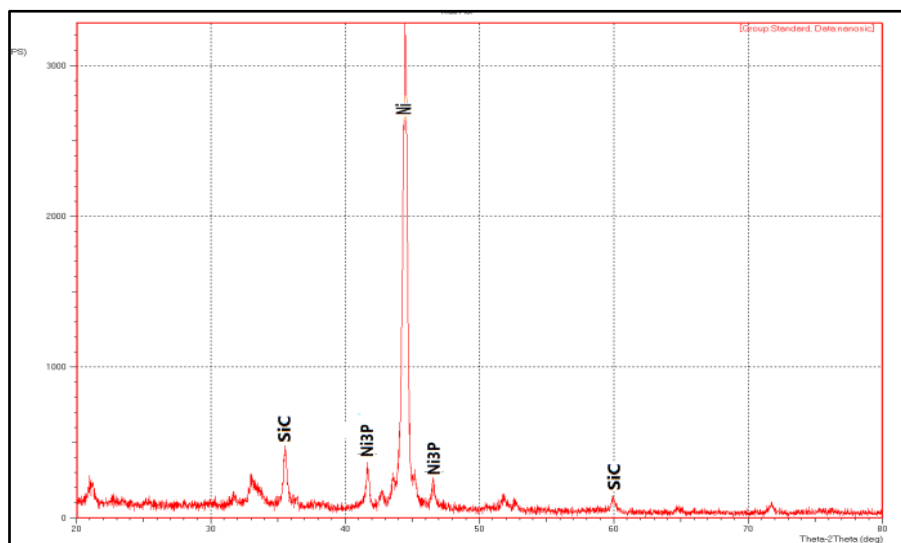


Figure 4: XRD patterns of (Ni-P-SiC) composite coating.



### 3.3 Wear behavior:

The variations of weight loss after different periods of time for the coated sample is shown in figure 5 and 6. It is clear from the figure 5, before heat treatment, the weight loss of superfine and micro-sized particles are gradually decrease with the increase in test times. From tribological point of view, this behavior is expected due to the process of asperity smoothing in the initial stages of contact associated with work hardening and eventually fatigue fractured. SiC nano-particle incorporated in the deposits gave higher wear resistance, than both Ni-P and Ni-P-micro SiC. The weight loss of the (Ni-P) coatings appears to increase with time. This means the coating was soft enough to be easily eroded by the hard disc. Examination conducted after the test supported this conclusion on the surface of the hard carbide disc. In spite of this the wear resistance of (Ni-P) coating has a lower weight loss than the base metal (low carbon steel).

The effect of heat treatment on the coating layer is shown in figure 6. (Ni-P) deposits increased with time, then level out after 15 min. The reason stands behind this behaviour is already stated. It was due to lack of uniforming and adhesion. In spite of this fact, the hardness is higher than the untreated layers. The variation of wear behavior of the heat treated (Ni-P) is almost similar to the untreated on, figure 5, but it levels out after 15 min. The observed high wear resistance of the heat treated Ni-P-SiC nano-particle (figure 6) is due to dispersion strengthening of the matrix by these added carbide particle and the formation of (Ni<sub>3</sub>P) hard phase. Hardening depends on size, amount, and area occupied by these hard particles. The incorporation of these hard carbide particles reduced the friction coefficient. This reduction is then reflected as a less loss during wear test. The nano-sized SiC-particles caused lower weight loss. This is because they distribute themselves more uniformly among the matrix and hence, the nano-particles are more effective in enhancing coating layer formation.

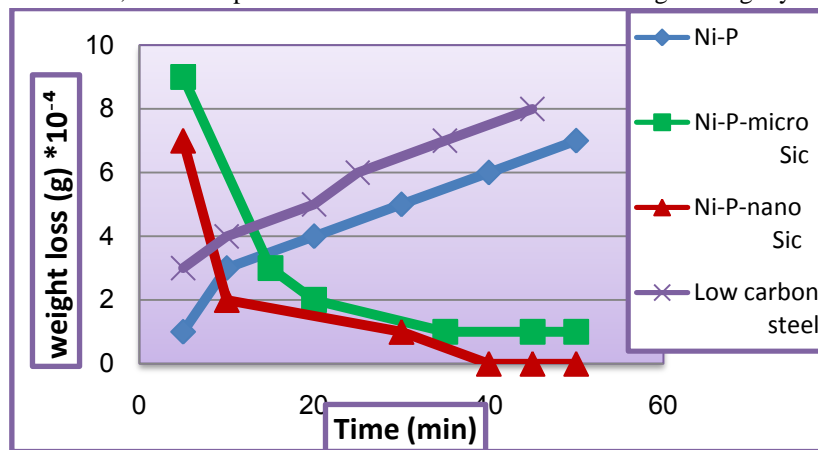


Figure 5: Effect of testing time on weight loss prior to heat treatment

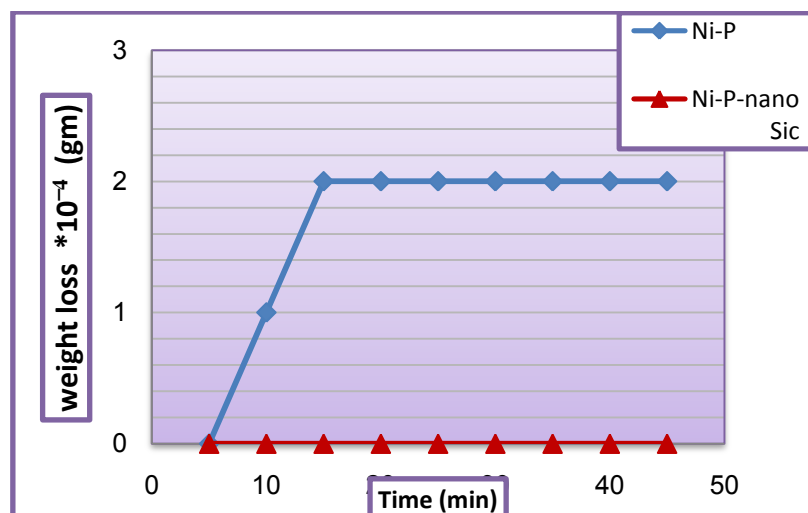


Figure 6: Effect of testing time on weight loss post to heat treatment.



#### 4. Conclusions

Heat treatment of (Ni-P) deposits on the base metal increased microhardness from 230 HV to 887 HV. Incorporation of nano-sized particles in (Ni-P) heat treated coating layers increased the microhardness from 230 HV to 1196 HV. The weight loss was almost (zero) during wear test period. Embedding SiC-micro particle increased the microhardness to (904 HV). Coating layers containing SiC particles developed global spherical deposits of (Ni<sub>3</sub>P) compound phase.

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