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Microscale Hardness of Acidic Electroless Ni-P/Ni-P-SiC Nano Composite Depositions

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Abstract
The ELNi-P along with Ni-P-SiCnano-composite coatings were plated on mild steel (AISI1040) substrates effectively. The ultrafine SiC particles with nano-size range of 50-70 nm as second phaseparticles were co-incerted intoan acidic electroless Ni-P bath. The SiC nano-particles of 4 gpl concentration were used for co-deposition into an EL Ni-P bath. The sodium hypophosphite particularly is selected as a reducer in chemical reactions. After two hours completion of platings, the as-coated EL Ni-P/Ni-P-SiC platings were heat treated at 400^{o}C in argon (Ar) environment for lone one hour period. As coating time increases thickness of coating also increases. The thickness of coating for all samples is found in between of range 20 to 25 micrometer. Further the micro-structure along with micro-hardness of as-deposited and heated coupons and were investigated for surface morphology by FESEM and EDAX respectively. The randomly dispersal of SiC nano-particles into the EL Ni-P-matrix is shown for 4gpl by micro-structural results. Insertion of ultrafine SiC particles into Ni-P matrix, it appears that size of Ni/Ni-P grainthroughout plating process reduced and introverted the grain growth for the period of the heat treatment. The micro-hardness at HV_{30} scale was improved significantly by adding up of especially SiC nano-particles into acidic Ni-P electroless bath after heated at 400 ^{o}C for time of one hour. The increment in hardness of Ni-P-SiC nano-composite coatings against Ni-P coating is explained.

Keywords- Coatings, Electroless, Ni-P-SIC, SEM, EDAX, Hardness.

1. Introduction

The electroless nano-composite platings are equipped by scattering impenetrable ceramic or metallic nano powders in an electroless bath. The immediate particulate insertion and the metal phase form composite coatings which further results in an enhancement of tribological and physical properties. The electroless plating is a very simple set-up and has controlled chemical reduction process and also there is consistency in composition in addition to depth of the coating (Agarwala and Agarwala, 2003). The uncontaminated Ni metal and metallic twofold alloysNi-P (Agarwala, 1987), Ni-B (Brenner and Riddell, 1946; Datta et al., 1991; Srivastava et al., 1992), Co-P (Brenner and Riddell, 1947; Agarwala and Agarwala, 2003)

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and Co-B (Brenner and Riddell, 1947; Agarwala and Agarwala, 2003) etc. were plated effectively and successfully by electroless deposition technique and analysed for their tribological and corrosion resistance properties (Mallory and Hajdu, 1990).The significance has been droop towards co-plating of succeeding segment particles in EL Ni-P milieu to transform their tribological and corrosion resistance properties for copious industrial applications. There are many numbers of particles, which already have been included in electroless Ni-Pmilieu (Mallory and Hajdu, 1990). The prospects of co-platation of second phase (X) particles into Ni-P milieu rely ahead on applications. The malleable particles as MoS_2, PTFE, CNT, BN (h), WS_2 and graphite (C) provide good lubrication when built-in into the electroless Ni-P matrix (Sudagar et al., 2013). These soft/lubricating particles encompass potential to stop the linkage between twobuddy surfaces beneath un-lubricated surroundings. Furthermore when prominent hardness as well aswear resistance properties aremost important necessity then the hard particles such as, ZrO_2, SiC, WC, W, Al_2O_3, TiO_2,Si_3 N_4 etc., are preferred for the co-deposition (Apachitei et al., 2002; Sharma, 2002; Huang et al., 2004; Jiaqiang et al., 2006). Among the above inclusions of soft and hard particles in EL Ni-P medium, it is found that SiC is individual very important ceramic material in favour of structural along with electronic appliances. The SiC particles have high hardness and wear resistance, very good chemical corrosion confrontation as well as thermal stability at lofty temperature, because of its covalent bonding character (Wang et al., 2003). Therefore, authentication of silic one carbide (SiC) nano-particles in acidic Ni-P electroless bath resting at mild steel material has been conceded out. The SiC nano-particles are purchased of Aldrich Company and reinforced into EL Ni-P-matrix from outside. For relevance of Ni-P-SiC nano-composite platings their characterizations and micro-hardness resistance properties have been carried out especially at an eminent temperature.

2. Experimental

2.1 Materials with Methods

The mild steel (MS) having quality AISI 1040was utilized like a basic substance for EL deposition and further elemental concerto of coupons are presented in Table 1.

The mild steel (MS) specimens of size (10 mm×05 mm× 04mm) was cleaned with a solution of acetone in addition to after it dirt-free through 5 % aqueous NaOH solution to eradicate several slippery stuff and corrosion merchandise and further followed bathe by de-ionized water. After this the sample is pursued by sensitization through 1% $SnCl_2$ (Loba Chemicals) along with subsequently activation with 0.05 % $PdCl_2$ (Merck) for one minute duration. For the duration of sensitization procedure, tin (Sn) ions were set down onto coupon surface to which palladium (Pd) ion was put down into establishment stage to afford nucleation or catalytic location to smooth the progress of the deposition procedure. The operating settings as well as the bath composition intended for coatings are given in Table 2. The chemicals in electroless bath were employed in as-obtained from exclusive of in the least refinement.

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Table 1. Chemical composition of mild steel sample

S. No.	Salt/Compound chemical formula	Quantity in gram (g) for 100 ml)	Function of chemicals
1	($NiSO_4$) Nickel sulphate	03.65 g	Ni^{2+} ions Source
2	Tri-sodium citrate	04.84 g	Work as a complexing negotiator, put off unrestrained discharge of Ni^{2+} ions
3	Sodium acetate	02.30 g	An acidic buffer to sustain the pH value
4	Sodium hydroxide/ Acetic acid 10% solution	supplementary drop wise	To uphold pH of solution ~5.5
5	Sodium hypophosphite	02.38 g	Work as a reducing agent, supply electrons to Ni^{2+} ions which on compliant electrons get reduced to Ni^0 and deposited on catalytic surface
6	Sodium dodecyl sulphate	00.01g	Increase wettability and surface charge of sample
7	Lead acetate	00.10mg	Work as a stabilizer for solution
8	SiC nano-particles	00.40 g	A reinforcement into EL Ni-P matrix
9	Bath operating conditions	-	Temperature 80-85°C; constant stirring is required, pH 5.5;
10	Annealing temperature	Upto 400 ^{0}C	To understand the consequence of heat behavior on corrosion resistance

Table 2. Chemical composition and operational parameters of the (EL) electroless bath

Elements	C	Cu	Mn	S	P	Si	Fe
weight%	0.081	0.049	0.33	0.021	0.007	0.033	balance

2.2 Characterization of Coatings and Hardness Methods

The exterior morphology of fine particles along with platings was scrutinized by SEM technique at Wadia Institute of Technology (EHT 20kV, Mag 5 KX, I probe 102pA, WD 13.5mm) Dehradun. Further the qualitative fundamental investigation of platings was carried out by means of an EDAX connection with FESEM equipment. The Vickers hardness (VHN) of platings was premeditated by a micro-hardness tester. The Coating thickness is calculated by way of following formula. Coating thickness in µm is measured by W x 10^4/D x A. Here 'W' put down for weight increase (g), 'D' is density of deposits (7.75 g/cm^3) and 'A' is surface area of deposition (cm^2).

3. Results along with Discussion

3.1 Depositions of Ni-P and Ni-P-SiC via Electroless System

In the electroless bath deposition procedure was carried out for total 2 hours duration, at constant temperature (85 ± 3°C). Primarily, a Ni-P coat was coated for 30 minutes duration (to avoidevery porosity in plating) and after that SiC nano-particles (~50-70 nm size) were commenced with fixed rousing in anidentical bath for following 90 minutes for co-plating of SiC nano-particlesin Ni-P milieu (Figure1). In end results, plain EL Ni-P platings lackingof SiC nano-particles was also arranged. Subsequent to deposition, coated coupons were taken out, sluiced with deionised water, after that air dried further more accumulated in vacuity desiccators. The heating of Ni-P as well as Ni-P-SiC depositions was carried out in an argon (Ar) ambience at 400^{0}C for one hour duration. The well-known piece of information is that, after taking place heat treatment of depositions a segment revolution from amorphous/micro-

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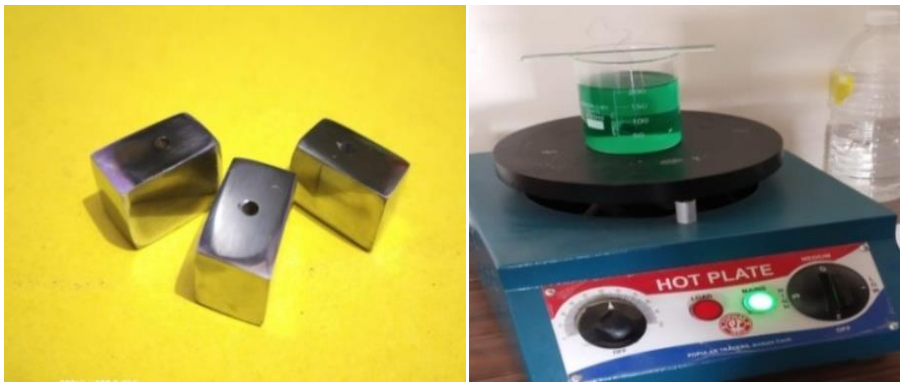
crystalline to crystalline segment takes place for the reason that of precipitation of Ni_3P, which enhance sticking togetherness of plating to substrate (Wang et al., 2003; Huang et al., 2004; Sudagar et al., 2013) in addition to get better the properties (hardness in addition to wear resistance) with respect to as-plated samples.



(a) (b)

Figure 1. (a) Polished coupons (b) Deposition process of Ni-P-SiC nano-particles introduced into electroless bath

3.2 Morphology of ELNi-P/ Ni-P-SiC Depositions

The FESEM micrographs of heat treated as well as of as-coated Ni-P along with Ni-P-SiC platings are given away (Figures 2). The SEM images of as-plated and heated coupons of EL Ni-P-SiC display the distribution of ultrafine SiC nano-particles into the EL Ni-P matrix. The SiC nano-particles illustrate an indiscriminate allocation in Ni-P milieu along with entity clustering character come into view 0.8 micro-metersin size. This size can be set up in extremely magnified images. Further some note worthy transformation in topography of the platings is experienced subsequent to heat handling at 400^0C. The microstructure images of Ni-P plating illustrated characteristic sphere-shaped bulbous consistent constitution even though morphology of Ni-P-SiC nano- composite plating is unlike on or after Ni-P plating.This behaviour may be due to deposition of SiC nano-particles resting upon facade of Ni-P plating. It is also found middling grain proportions are 0.7micro-meters in diameter and 3-5 micro-meter in length. While in support of the as-plate damalgamated samples with ultrafine SiC nano-particles, average grain size reduces to 70-80nm.It can be over and done with that ending size of Ni-P grains in composites is restrict edowing to a superior nucleation density and inclusion of SiC nano-partcles into Ni-P matrix changes the shape of grains. The estimated stoicheo-metric proportion of elemental analysis (wt. %) dogged through EDAX is specified in Table 3. The EDAX study, in case of heat treated Ni-P-SiC depositions, suggest a decrease in concentration of Ni on addition of SiC (4 gpl) and an increase in concentration of P, C, Si and Fe %.It is beingac counted (Balaraju et al., 2005; Feldsteen et al., 1999; Sharma et al, 2014) that elemental allocation in plating influences plating properties furthermore forms an inter-diffusion deposit. The occurrence of note worthy quantity and consistent allocation of P, C, Si and Fe revealed by EDAX analysis, creditedto dissemination of plating

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elements en route for boundary of plating and mild steel material which is responsible for sky-scraping hardness of heated Ni-P-SiC coupons.

Table 3. EDAX values of EL Ni-P -SiC as-plated and heated coupons

Element	Weight %	
	Ni-P-SiC (As-Coated)	Ni-P-SiC (Heated)
Ni K	80.41	77.45
P K	10.26	10.71
Fe K	02.34	03.54
C K	03.17	03.29
Si K	03.82	04.98
Total %	**100.00**	**100.00**

Figure 2. Ni-P-SiC coated heated coupon

3.3 Micro-Hardness Investigation of Ni-P/Ni-P-SiC Platings

The (VHN) micro hardness of Ni-P as well as Ni-P-SiC nano-composite platings in as-coated and heat treated environments were dogged by resources of micro-hardness tester with dwelling time of 15second under a 30 gf load (Table 4). The micro-hardness values of Ni-P coveredsegmentas well as Ni-P-SiC plated samples are in subsequentexpress as Ni-P-SiC(Heated)>Ni-P-SiC (as-plated)>Ni-P(Heated) >Ni-P (as-plated) > mild steel (MS).Gades of microhardness recommend that accumulation of SiCnano-particles intooutside layer does add significantly to the micro-hardness of the sample as SiCnano-particles are hard ceramic carbides and also some inter-metallic's of Ni-P, Ni-Fe,Ni-Si (crystalline structure) etc. are formed.

Table 4. Micro-hardness of electroless nano-composite platings

Test Samples	Value of Micro-hardness (HV_{30})
(MS) Mild steel	338
Ni-P (as- plated)	440
Ni-P (heated)	482
Ni-P-SiC (as-plated)	534
Ni-P-SiC (heated)	677

4. Conclusions

In this experimental work, aless black hazy and uniform nano composite platting of Ni-P-Si Cupon the mild steel token is pragmatic. Further the FESEM/EDAX studies reveal that SiC nano-particles into electroless Ni-P milieu were co-deposited and randomly distributed Figure 3. The coating thickness increases as coating time is increasing. The small Ni-P grains are

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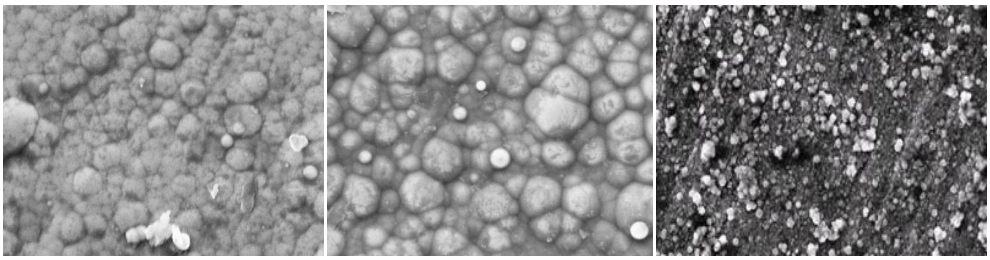
created in province among nano ceramic particles, which demonstrate the proportions to a large extents lighter than that of grains size. Further, importantly additions of ceramic particles make available the formation of miniature Ni grains all through juncture of electroplating in addition to slow down grain augmentation of Ni-P into thermal handling. The Ni-P/Ni-P-SiC coated coupons were also heated to accomplish crystalline nature of depositions. The EL Ni-P-SiC plating show signs of good quality adherence upon mild steel surface. A very good step up in micro-hardness ethics for Ni-P-SiC nano-composite platings is observed as put side by side to Ni-P and MS coupons Figure 4.



(a) **(b)** **(c)**

Figure 3. FESEM micrographs (a) Ni-P heated (b) Ni-P-SiC as-coated and (c) Ni-P-SiC heated coupons

Figure 4. Micro-hardness tester

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