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Effects of Heat Treatment on Morphology and Hardness of Electroless Ni-P Coating: Impact of Temperature and Concentrations

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ABSTRACT

This study examines the effects of heat treatment on the morphology and micro-hardness of Electroless Nikel-Phosphorus (Ni-P) coating. The coatings were deposited at various levels of Nickel source concentration, reducing agent concentration and bath temperature. The coated samples were considered for heat treatment at 300 ℃, 400 ℃ and 500 ℃ for one hour. The hardness of the samples was evaluated with a Vickers microhardness tester and the morphology of the samples has been studied with scanning electron microscopy (SEM). Energy dispersive spectroscopic (EDS) analysis for elemental mapping was done by the coating delamination of the substrate. The samples deposited at conditions of low Ni source concentration and high P concentration and high bath temperature condition, showed the maximum microhardness when heat treated at 300 ℃. A sample with high Ni source concentration and low P concentration at low bath temperature conditions showed a gradual reduction in micro-hardness with an increase in heat treatment temperature. SEM observed the least agglomeration at a heat treatment temperature of 300 ℃. The coatings deposited at low P conditions showed severe cracks at 500 °C

Keywords: Electroless Ni-P coating; Heat treatment; Microhardness; Morphology

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INTRODUCTION

Electroless nickel plating is a process for depositing a nickel alloy from aqueous solutions onto a substrate without the use of an electric current. Electroless nickel plating is a chemical process that reduces nickel ions in solution to nickel metal by chemical reduction. The electroless process is an autocatalytic method in which the reduction of the metallic ions in the solution and the film deposition can be carried out through the oxidation of a chemical compound present in the solution itself. The procedure necessitates that electrons from the surface of a metal substrate or the catalysts used to start the deposition decrease a cation of the metal to be deposited. In turn, the reductant oxidizes this surface by delivering electrons there.

Brenner and Riddell [1, 2] developed a process for plating the inner walls of tubes with nickel-tungsten alloy using an insoluble anode bringing out the unusual reducing properties of the Hypophosphite. This process was eventually covered by a patent [3], While the initial acceptance of the chemically deposited coating was slow, subsequent development of the process on the improvements of both composition and techniques has increased its industrial applications. The approach employed by Bretean and Rouse [4] to deposit nickel differs from the electroless plating method in that the latter's reaction was complete and spontaneous, whereas Brenner and Riddell's method was catalytic. In this instance, deposition was limited to activated surfaces submerged in the bath environment. Since the electroless method did not provide a suitable way to control the phosphorous level, an electrolytic method was developed to produce the alloys when it was determined that the deposited metal was an alloy of nickel and phosphorous [5, 6]. The physical, mechanical, and chemical properties of electrodeposited nickel-phosphorous alloys are compared with electroless deposits shaving a similar phosphorous content. Such deposits were to be amorphous and comprise a mixture of metallic nickel and

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nickel phosphide (Ni_3P). Reactions occurring in electroless nickel deposition with hypophosphite ion as the reducing agent may be represented as

$$\mathrm{Ni^{2+} + H_2PO_2^- + H_2O \xrightarrow{\text{Catalytic active surface}} Ni + 2H^+ + H(HPO_3)^-} \quad (1)$$

$$\mathrm{HPO_2^- + H_2O \xrightarrow{\text{Catalytic active surface}} H(HPO_3)^- + \tfrac{1}{2}H_2} \quad (2)$$

Apachite et al. [7] developed an opportunity to increase materials performance for different applications to protect them by coatings. Electroless nickel deposition represents an alternative method to obtain coatings on various substrates (metallic and nonmetallic). As a result, a linear relationship between coating thickness and time usually occurs. To increase the hardness and abrasion resistance of electroless Ni-P deposits heat treatment was performed, so that maximum hardness was achieved after heat treatment at 400 ˚C for 1 hr and hardness increased up to 500-1100 HV. Yildiz et al. [8] studied the effect of heat treatments for electroless deposited Ni-B and Ni-W-B Coatings on 7075 Al Alloy. Electroless Nickel- Boron (Ni-B) coatings have a wide range of usage areas including aircraft and automotive applications as well as 7075 Aluminum alloys.

Maximum hardness values for both substrate and coatings are determined when the coated system was heat-treated at 150°C for 10 hours. SEM micrographs have shown that Ni-B coated specimen has a cauliflower-like microstructure after heat treatments which enable the coated system to perform lower wear rates. Micro-hardness values are increased which procures lower wear rates and give rise to be at crystallographic condition.

Zarebidaki et al. [9] studied the effect of heat treatment on the properties of electroless Ni-P carbon nanotube composite coating. Electroless Ni-P carbon nanotube composite coating was deposited on API-5L X65 steel substrates. The coating was vacuum heat treated at a temperature of 200˚C for 2h,400 ° C for 1 hr, 600˚C for 15 min. XRD results indicated that as plated coating had either nanocrystalline or a mixture of amorphous and nanocrystalline structures. Heat treatment of the coatings above 400˚C produced a mixture of polycrystalline phases. The highest micro-hardness was achieved for the samples heat treated at 600˚C for 15 min. The lowest corrosion current density value was obtained for the coating at 400 ° C for 1 hr. Wang et al. [10] studied the influence of heat treatment on the coating of nickel plating on hollow glass beads. Ni-plated hollow glass beads (GBs) were firstly prepared by pd-activation and electroless plating, then Ni-plated GBs were heat treated at 450˚C for 1h, Ni-plated GBs/PVC composite was fabricated by using polyvinyl chloride (PVC) adhesive. The results show coatings prepared by electroless plating were uniform, the nickel element in the coating was higher than 95.71% (mass fraction) with heat treatment, the surface roughness of the coating was greater and the reflectivity descended apparently the D-value was 1dB at the frequency of 15 GHz but the influence of heat treatment for heat insulation of Ni-plated GBs was not great. Xi et al. [11] studied on effect of phosphorous content on suspension of Ni-P coated diamond (NPCD) and wear resistance of electrodeposited Ni-Co-Mn/NPCD composite coatings. The results demonstrated that lower phosphorus contents in NPCD yielded better suspensions, and contributed to higher volume fractions in the composite coatings. The wear resistance of Ni-Co-Mn/LP-NPCD composite coatings was found superior to other NPCD samples and untreated diamonds. However, the latter was weakened as the phosphorus content increased in NPCD. The addition of a third metal to form ternary alloys like Ni-Co-Mn, combine the advantages of binary alloys with additional superior hardness when compared to Ni-Co and Ni-Fe, as well as lower frangibility than Ni-Mn and Ni-P. Kaissi et al. [12] studied on influence of sodium acetate on electroless Ni-P deposits and effect of heat treatment on corrosion behaviour. The optimization of the operating parameters made it possible to obtain a stable Ni-P alloy deposition formulation. To understand the reaction mechanism of the deposition process, a kinetic study was performed by cyclic voltammetry and by electrochemical impedance spectroscopy (EIS). The coatings obtained have a very high corrosion resistance in a very aggressive acid medium which increases with the heat treatment. Franco et al. [13] studied the effect of reinforcement and heat treatment on elevated temperature sliding of electroless Ni-P/SiC composite coatings. The investigation is focused on the wear behaviour at elevated test temperature of composite Ni-P/SiC deposit, with varying concentration of the reinforcing SiC particles. The phase evolution measured by X-ray diffraction suggests slight crystallisation during wear testing at 200°C. In coating without reinforcing particles, adhesive wear is accompanied by micro-cracks. The thermal heat generated and the cyclic loading could have induced subsurface micro-cracks. Owing to the effective matrix-ceramics system in composite coatings, fine grooves, abrasive polishing and uniform wearing are observed. Reinforcing particles in the matrix hinder micro-crack formation and significantly reduce the wear rate. Triboxidation is confirmed from energy-dispersive X-ray spectrometry.

Huber et al. [24] examined the microstructure and phase composition of Ni-P and Ni-P-Re layers that were electroless plated on copper substrate and had middle phosphorus contents of 8.8 and 8.2 weight % of P and 6.8 weight % of Re, respectively. The phases of Ni-P-Re plating were Ni, Ni_3P, $Ni_{0\cdot21}Re_{0.79}$, Ni_5P_2, and NiP, according to XRD data; TEM analysis verified the existence of Ni_3P, NiP, and Ni_5P_2.

Chuhong [25] studied on indentation and fracture behavior of electroless Ni-P-based composite coatings. Electroless Ni-P coatings have smooth and uniform surfaces with strong interfaces. The addition of nano-titanium particles increases surface roughness. As-deposited Ni-P coatings have an amorphous structure and the microstructure changes from amorphous into crystalline nickel and Ni3P after annealing to 400°C. As the annealing temperature increased, the percentage of nickel phase increased while that of Ni3P decreased. The addition of nano-titanium particles does not change the amorphous structure of as-deposited coatings. Crystalline nickel and nickel phosphide phases are also identified in annealed Ni-P-Ti coatings, and Ni3Ti was observed in Ni-P-Ti coatings after 600 and 800°C annealing. Annealing increases the interface strength between the coating and the substrate. Anijdan et al. [26] studied the effect of electroless bath temperature and heat treatment on the properties of Ni-P and Ni-P-Cu composite coatings. The addition of Cu particles reduced the hardness of Ni-P coating (from 482.4 to 351.2 VH in 1 gm/lit Cu) within Ni-P-Cu composite coatings the hardness of Ni-P-Cu composite coating increased from 351.2 to 380.7 VH by increasing the Cu particles from 1 to 7 gm/L. The structure of the coating was crystalline during the heat treatment making the coating harder with increasing the pH of the solution from 4.5 to 7 and then to 9 weight % of P and the particles of Cu were reduced. Gadhari et al. [27] studied the effect of annealing temperature and Alumina particles on mechanical and tribological properties of Ni-P-Al2O3 composite coatings. The micro-hardness, wear resistance and corrosion resistance of the composite coating improved significantly after heat treatment (400˚C) and in the presence of alumina particles. The composite coating deposited with alumina particle concentration of 10 g/L in an electroless bath and heat treated at 400˚C displayed excellent results compared to Ni-P, as-deposited Ni-P-Al2O3 coating and coatings heat treated at different annealing temperatures (200˚C, 300˚C and 500˚C). Microstructure changed and composition of the composite coatings due to the incorporation of alumina particles and heat treatment. Kundu et al. [28] studied the tribological behaviour of electroless Ni-P Deposits under different temperatures. The results obtained were compared among each other and with that of the room temperature (RT) tests of the coating. It was found that the friction coefficient (COF) and wear rate of coatings mostly increased with increased in load for all the test temperatures. On the other hand, the wear rate and COF exhibit a reversal of trend in the change of sliding velocity. The in situ heat treatment that the as-deposited samples experienced throughout the test may be the reason for their lower wear rate, especially at high temperatures. Better results are obtained with as-deposited coatings, particularly when the test temperature is maintained at or above the coating's phase transformation temperature. In the as-deposited phase, the coating is amorphous and exhibits a nodular shape. The coating becomes crystalline when heated. At increased temperatures, the EN coatings exhibit a wear mechanism that combines abrasive and sticky elements. Micro-cracks accompanies adhesive wear. Utilising energy-dispersive X-ray spectroscopy, Triboxidation is validated.

Mukhopadhyay et al. [29] studied the effect of Heat Treatment on the Characteristics of electroless Ni-B, Ni-B-W and Ni-B-Mo Coatings. The as-deposited coatings present a dense nodular surface morphology in the case of Ni-B and Ni-B-W coatings whereas Ni-B-Mo coatings reveal a cauliflower-like morphology with aggregated clusters of nickel nodules and visible surface cracks. Heat treatment results in grain growth and visible grain boundaries in the case of all three electroless deposits. Energy dispersive X-ray analysis reveals that the Ni-B deposits are boron-rich. The boron content decreases with the co-deposition of W in the EN matrix. From X-ray diffraction analysis it is concluded that the coatings are primarily amorphous in their as-deposited condition and turns crystalline on heat treatment. The micro-hardness of Ni-B-W coatings is the highest amongst the three in as-deposited condition and up to a heat treatment temperature of 400°C. While Ni-B-Mo coatings show higher thermal stability. Matik [30] studied the structural and wear properties of heat-treated electroless Ni-P alloy and Ni-P-Si_3N_4 composite coatings on iron-based PM compacts. The structural and wear properties of as-plated and heat-treated electroless Ni-P alloy and Ni-P-Si_3N_4 composite coatings on iron-based powder metal (PM) compacts were investigated. The coatings were prepared using hypophosphite-reduced alkaline electroless nickel bath. The synthesized submicron α-Si_3N_4 particles (10 g/l) were added into the Ni-P bath to develop Ni-P-Si_3N_4 composite coatings on the PM compacts.

The study has examined the coated compacts that were heat-treated at 300, 400 and 500 °C for 1 hour and the effect of heat treatment on the coating performance was studied. The chemical composition of the coatings was analysed by EDX. Phase transformation behaviour has been studied by differential scanning calorimetry (DSC). The wear properties have been studied using a pin-on-disc tribometer under unlubricated sliding conditions under a normal load of 40 N, sliding speed of 1ms^{-1} and sliding distance of 1000 m. The incorporation of submicron Si_3N_4 particles in the deposit significantly improves the hardness and wear resistance of composite coatings. The hardness and wear resistance of Ni-P and Ni-P-Si_3N_4 coatings have been analyzed heat-treated at 400 °C had the maximum hardness and wear resistance.

EXPERIMENTAL PROCEDURE

The specific parameters (temperature, duration, cooling rates) for both the coating procedure and heat treatment procedure will depend on the coating material, substrate, and the desired properties.

Coating procedure

Take two 250ml beakers and clean them with distilled water and mix nickel sulphate with 100ml of distilled water and take sodium hypophosphite into the second beaker and mix it with 100ml of distilled water. Two beakers are placed on the magnetic stirrer cum heater so that obtain a proper mixing of the salts and the temperature rises to 50-55℃. These two solutions are mixed in another 250ml beaker and finally, the solution becomes 200ml. This solution which is at 55 ℃ is placed in a pH meter and measured the pH value.

Table 3 1 : Range of process parameter

SN	*Process parameters*	**Range**	
		Low	*High*
(a)	Ni Source concentration (g/ l)	15	20
(b)	Reducing agent concentration (g/l)	20	30
(c)	Temperature (°C)	70	90

Table 3 2: Variation of Ni-Source concentration

Run no.	Ni-Source concentration (g/l)	Reducing agent concentration (g/l)	Temperature (°C)
1	15	30	90
2	20	30	90

Table 3 3: Variation of reducing agent concentration

Run no.	*Ni-Source concentration (g/l)*	*Reducing agent concentration (g/l)*	*Temperature (°C)*
3	15	20	90
4	15	30	90

Table 3 4: Variation of bath temperature

Run no.	*Ni-Source concentration (g/l)*	*Reducing agent concentration(g/l)*	*Bath temperature (°C)*
5	15	30	70
6	15	30	90

This solution is placed on the heater and the temperature rises to the required value and the temperature constant by using the controlling resistance of the heater. Activator solution (palladium chloride) is kept in another beaker and heated up to 55°C. The cleaned copper samples were immersed in to the activation solution for 10-15 sec. Activation samples are rinsed into the distilled water and then placed in a 200ml electroless bath which is kept at a steady temperature. The temperature of the solution is continuously measured with the help of a thermometer which is placed in the bath after one hour, the samples are taken from the solution and cleaned in the distilled water, after one hour of deposition, the coating obtained a bright thin layer of Ni-P on the copper samples.

Range of Process Parameter

The range of process parameters is provided in Table 3.1 below.

Experimental runs

Variation of Ni-Source concentration

The variation of Ni-Source concentration is provided in Table 3.2 below.

Variation of reducing agent concentration

The variation of reducing agent concentration is provided in Table 3.3 below.

Variation of temperature

The variation of temperature is provided in Table 3.4 below.

Heat treatment procedure

Heat treatment of the coated samples was carried out in the structure lab which is situated in the Civil Engineering Department, NIT Jamshedpur. First, it took the muffle furnace for heat treatment and the capacity of this muffle furnace is up to 1000 °C. It is cleaned the samples from the ethanol to remove the dust and impurities. I have 12 samples and each sample are varying parameters, samples having (high Ni), (low Ni, high P, high temperature), (low p), and (low temperature.) are varying parameters. Cleaned samples are kept in the muffle furnace which is hanging from the copper wire in the arrangement. The muffle furnace door is closed and heat treatment begins. Four samples have varying parameters and are held in the muffle furnace per trial. For the first time, heat treatment takes place at 300 ˚C for 1 hour and the heating rate is 10 °C /min and cooling will be done in the furnace. The second time, heat treatment takes place at 400 ˚C for 1 hour and the heating rate is 10 °C /min and cooling will be done in the furnace and in third time, heat treatment takes place at 500 °C for 1 hour and heating rate are 10 ˚C/ min and cooling will be done in the furnace. Finally, all the samples are heat treated and then after check the hardness and surface morphology.

RESULTS AND DISCUSSION

Microhardness of high Ni and low Ni-containing samples at different heat treatment temperatures

Microhardness for high Ni and low Ni samples at un-heat-treated condition and after heat treatment at different temperatures is presented in Fig. 4.1. For the High Ni sample, a gradual reduction in hardness can be observed. However, for the low Ni containing sample maximum microhardness was observed at heat treatment temperature of 300 °C, beyond which a gradual reduction was observed.

Microhardness of high P and low P containing samples at different heat treatment temperatures

Microhardness of high P and low P containing samples at un-heat-treated and heat-treated condition are presented in Fig. 4.2 for the high P containing sample, maximum microhardness was observed at 300 °C beyond which a drop in microhardness was observed. However, for the low P containing sample a drop in microhardness was observed with increasing heat treatment temperature.

Microhardness of samples deposited at high and low bath temperatures and heat treated at different temperatures

Microhardness of samples deposited at high and low bath temperatures at un-heat-treated condition and after heat treatment at different temperatures is presented in Fig. 4.3. The sample deposited at high bath temperature registered the maximum microhardness when heat treated at 300 °C. However, at higher heat treatment temperatures, the hardness reduced, the minimum being at 500 °C.

Morphological study of the coated samples

It may be observed that there is a general trend of reduction in microhardness of the coated samples with increase in heat treatment temperatures. Also, it may be noted that the coatings corresponding to low Ni (Run No. 1), high P (Run No. 4) and high bath temperature (Run No. 6) were deposited under the same deposition conditions (Table 3.2, 3.3 and 3.4). These three samples therefore showed the same trend in variation of microhardness with maximum microhardness at 300 °C heat treatment temperature (Fig. 4.1, 4.2, 4.3). All the other samples i.e. high Ni (Run No. 2), low P (Run No. 3) and low bath temperature (Run No. 5) registered similar trend of reduction in hardness with increase in heat treatment temperature.

The maximum microhardness for the samples corresponding to low Ni (Run No. 1), high P (Run No. 4) and high bath temperature (Run No. 6) after heat treatment at 300 °C, possibly occurred due to formation of some crystalline phase of Ni. Further investigation by X-ray diffraction is necessary to confirm this hypothesis. The scanning electron microscopic images of a reference sample (corresponding to low Ni, high P and high bath temperature) in un-heat-treated condition and after heat treatment at different temperatures are presented in Fig. (a, b, c, d). In comparison to un-heat-treated sample, the agglomerate grain size of the sample heat treated at 300 °C was finer which may be attributable for increase in microhardness. After heat treatment at 400 °C, the agglomerate grain size again increased slightly possibly because of adatom mobility. At the highest heat treatment temperature of 500 °C, the agglomerate size further increased. Some micro-cracks were also visible which possibly propagated during indentation, resulting in lower microhardness.

As mentioned earlier, similar trend of reduction in microhardness has been observed for the samples corresponding to high Ni (Run No. 2), low P (Run No. 3) and low bath temperature (Run No. 5). Therefore, morphological study of the sample corresponding to low P (Run No.3) has been conducted only which will also reflect on the trend of variation of microhardness of the other two samples viz. high Ni (Run No. 2) and low bath temperature (Run No. 5).

The scanning electron microscopic images of the sample corresponding to low P (Run No.3) in un-heat-treated

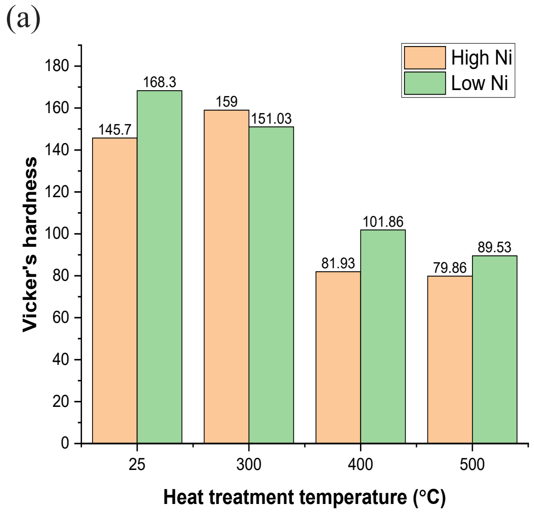


Fig. 4.1: Variation of micro hardness for high Ni and low Ni containing samples

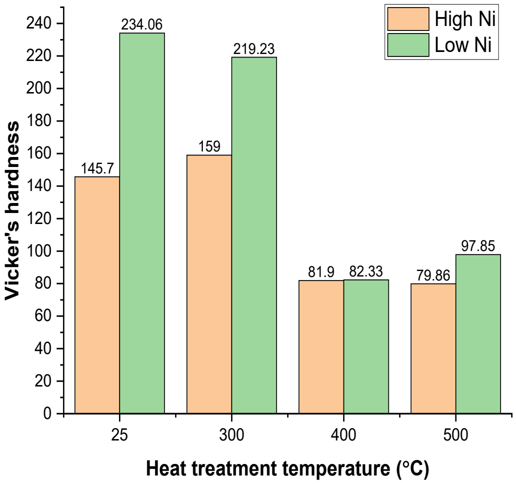


Fig. 4.2: Variation of microhardness for high P and low P containing samples

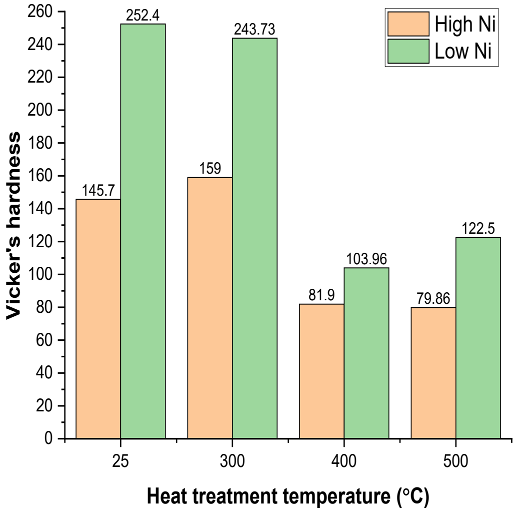


Fig. 4.3: Variation of microhardness samples deposited at high and low bath temperatures

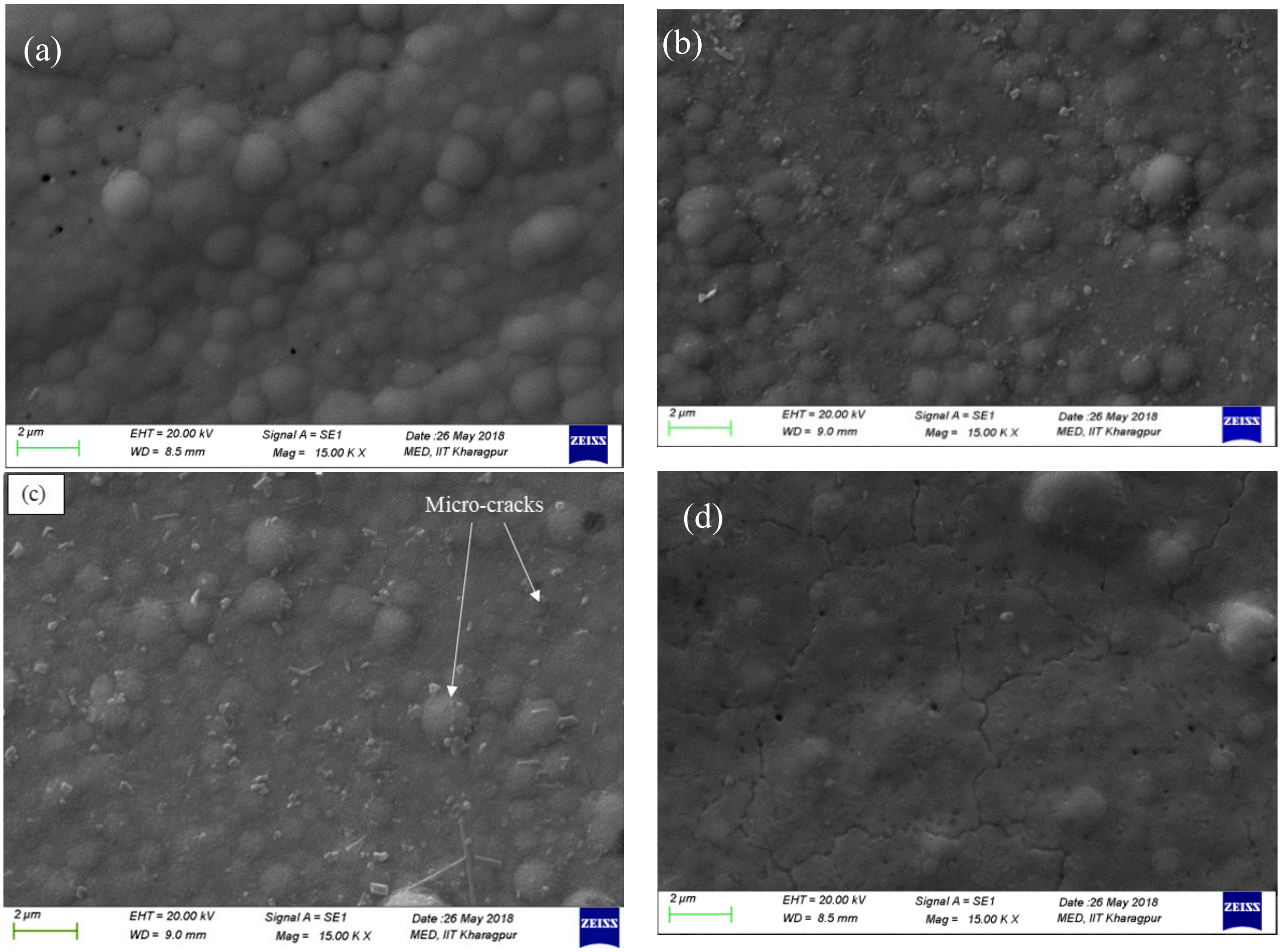


Fig. 4.4: Morphology of Electroless Ni-P coating at (a) Un-heat-treated condition and heat treated at (b) 3000C, (c) 4000C and (d) 5000C

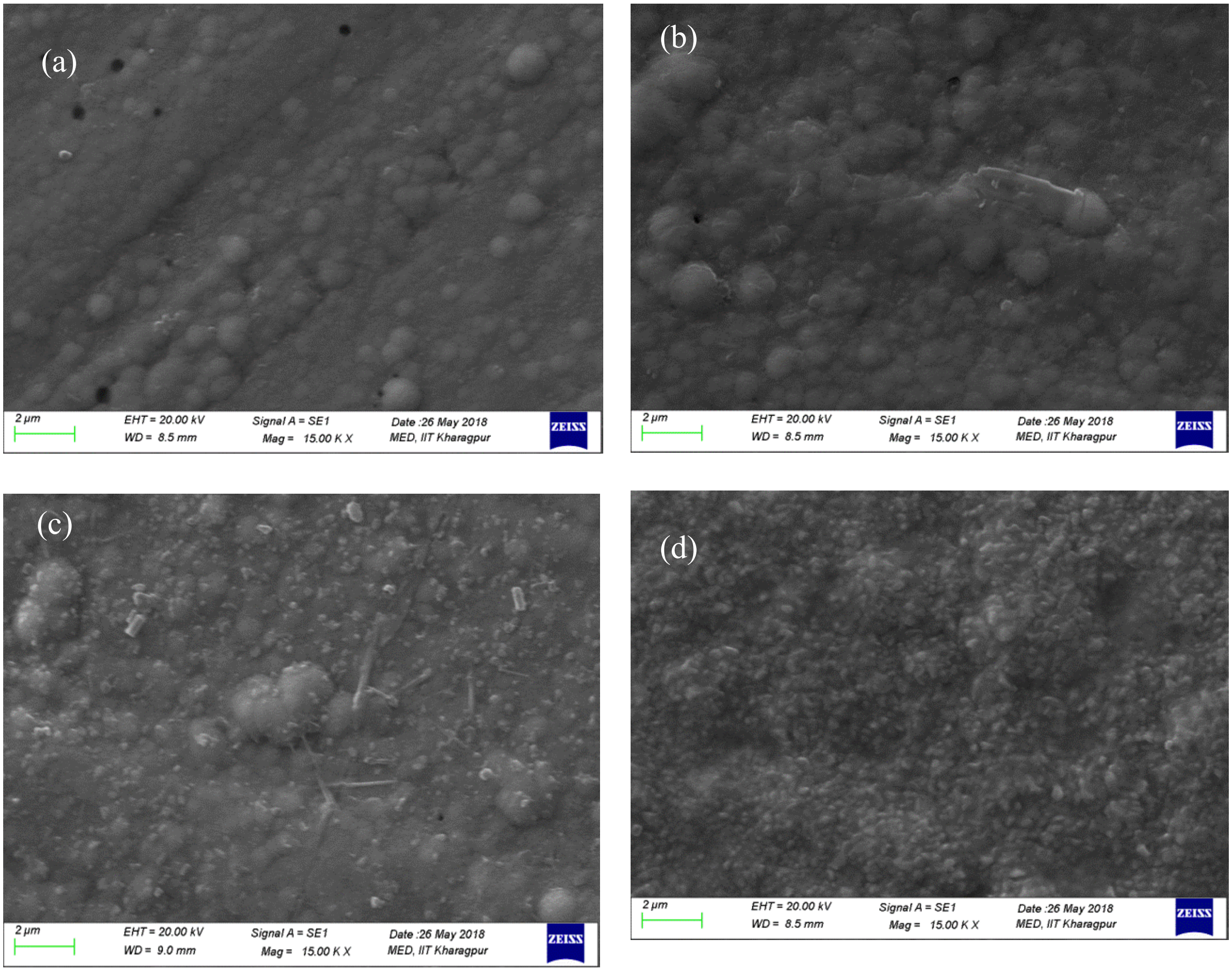
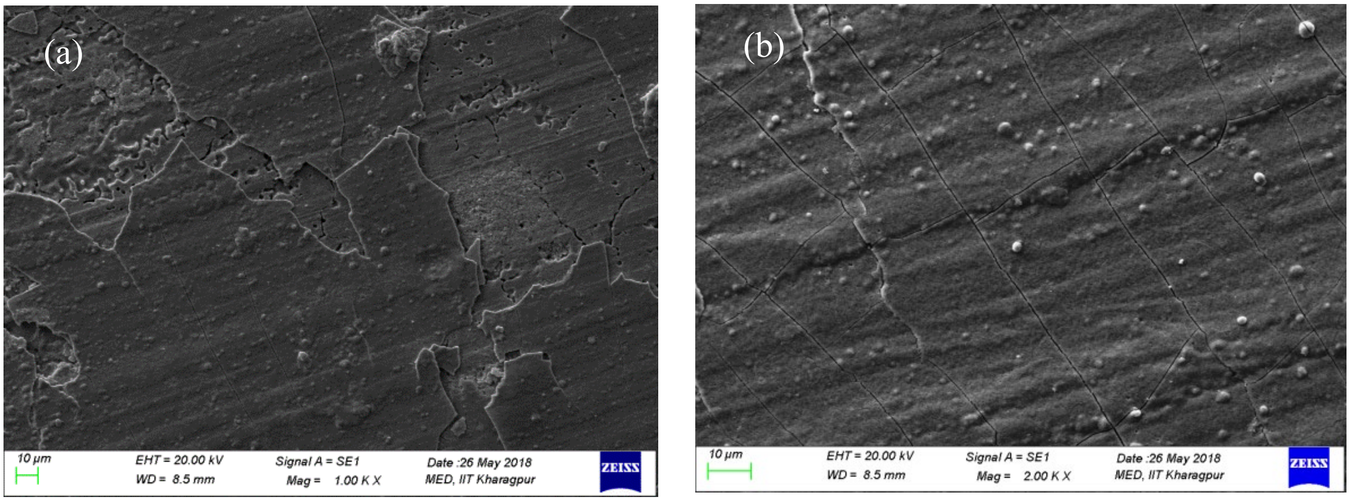


Fig. 4.5: Morphology of Electroless Ni-P coating deposited corresponding to low P (Run No.3) at (a) Un-heat-treated condition and heat treated at (b) 300^{0}C, (c) 400^{0}C and (d) 500^{0}C



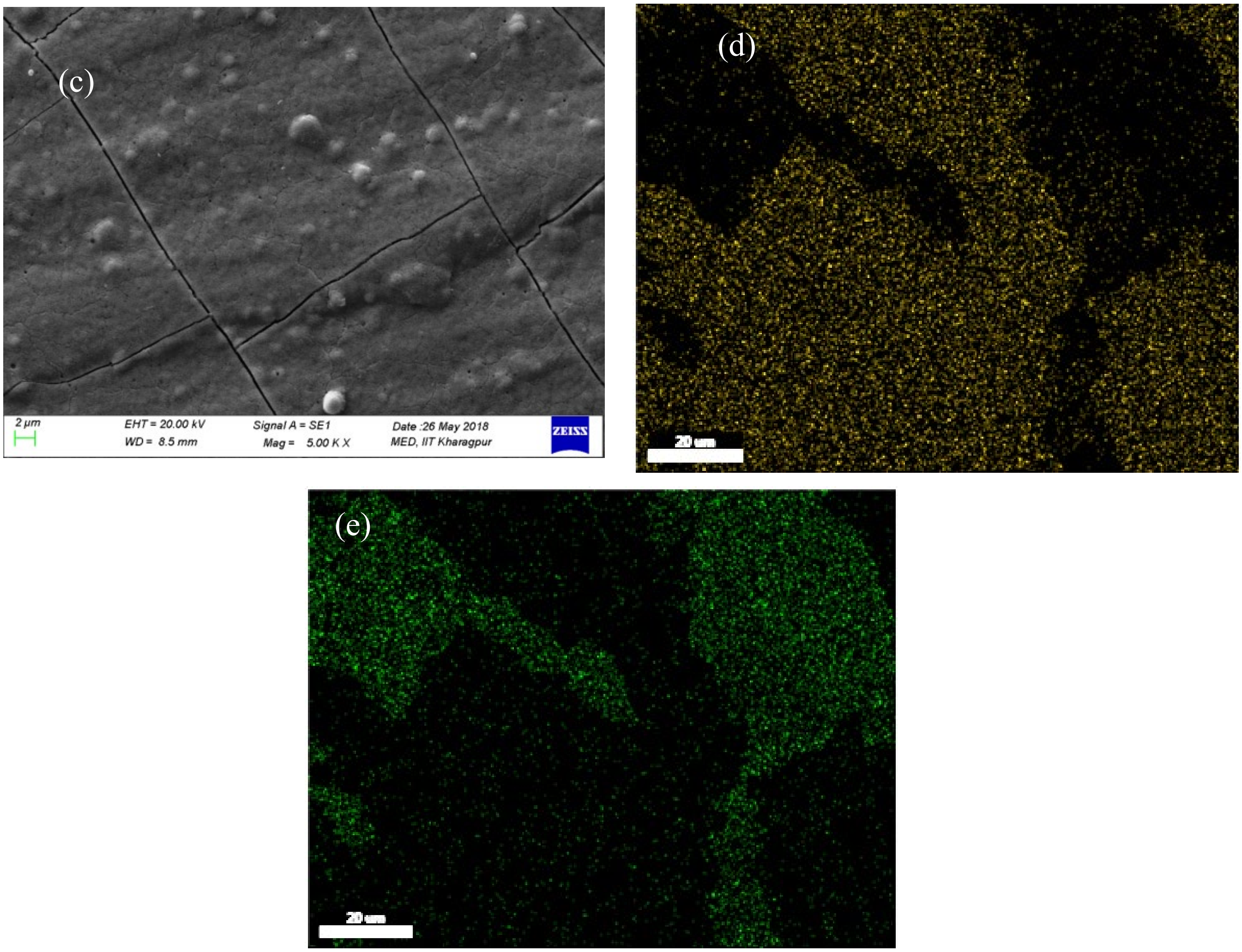


Fig. 4.6: (a–c) Severe cracking and coating delamination from substrate after heat treatment at 500^0C for the sample corresponding to low P (Run No.3) and distribution of (d) Ni and (e) Cu corresponding to area shown in (a)

condition and after heat treatment at different temperatures is presented in Fig. (a, b, c, d). As compared to the un-heat-treated sample, the agglomerate size scaled with heat treatment temperature which possibly resulted in a drop in microhardness.

At the highest heat treatment temperature of 500^0 C, severe cracking, and delamination of the coating (sample corresponding to low P (Run No.3) from the substrate as shown in Fig. (d). The higher magnification images clearly reveal the cracks produced in the coating.

Energy dispersive spectroscopic (EDS) elemental mapping of the region shown in Fig. (a-c) clearly reveals the delamination of the coating and exposure of copper substrate.

Conclusion

The following conclusions can be drawn from the work.

- The samples corresponding to low Ni, high P and high bath temperature showed maximum microhardness when heat treated at 300 ℃. At further higher heat treatment temperature of 400 ℃ and 500 ℃, the hardness reduced. Minimum agglomerate size was also observed at 300 ℃.
- The samples corresponding to high Ni, low P and low bath temperature showed a reduction in hardness with increase in heat treatment temperature. The agglomerate size scaled with heat treatment temperature.
- At highest heat treatment temperature of 500 ℃, severe cracking and delamination of the coating from the substrate were observed.

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