Synthesis and Characterization of Ni-P Coated Hexagonal Boron Nitride by Electroless Nickel Deposition

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Electroless plating has been receiving a steady progress over the last decade on the modification of the surface properties of ceramic materials in order to produce composite coatings with unique characteristics for critical tribological systems. In this work, an electroless nickel deposition process was used to deposit nickel-phosphorous (Ni-P) coating on hexagonal boron nitride (h-BN) particles via hypophosphite-reduced acid bath solution. The substrate particles were initially subjected to series of pretreatment operation in order to ensure that the particles are cleaned and catalytically active prior to electroless plating. The characterization of the as-received and Ni-coated powder was studied through scanning electron microscopy (SEM), energy dispersive x-ray (EDX) spectroscopy and field emission scanning electron microscopy (FESEM). The result reveals that the pretreatment of h-BN powder provides substrate particle surfaces with coarse and roughened structures which are normally considered suitable for Ni-P deposition. Moreover, the result of the EDX analysis confirms the existence of nucleating agents and Ni-P coating on the surface of the treated h-BN powder. The cross-sectional microstructure of the coated powder shows that the h-BN particles were embedded in a continuous matrix layer of Ni-P deposit. The EDX mapping profiles further indicate that the deposited Ni-P alloy mass was uniformly distributed on the surface of the Ni-P codeposited h-BN particles (Ni-P-h-BN). The successful development of Ni coated h-BN powder will raise the potential of h-BN as a high-performance coating material.

Keywords: electroless deposition, h-BN particles, etching, Ni-P layer, surface modification.

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1. INTRODUCTION

Electroless nickel (Ni-P) plating process is gaining wide acceptance in tribology community as a surface treatment offering the possibility to produce a wide range of metal-coated engineering components [1, 2]. The potential of this technique has to be related to its unique quality of combining hardness and corrosion properties as well as capability to generate uniformly distributed deposit, regardless of geometrics, on various metallic and non-metallic surfaces as demonstrated in pure metals and alloys [2–3], plastics [4], ceramics [5–7], fibers [8] and powders [9–13]. The electroless nickel plating process basically involves the chemical reduction of aqueous nickel cation on a catalyzed substrate surface in the presence of a reducing agent [14]. On comparing with the conventional electroplating technique, the electroless plating process offers impressive advantages in terms of lower processing cost since no electricity is involved and the possibility of obtaining varying deposits with excellent properties by easy adjustment of the pH, the temperatures and the compositions of an electroless bath [2]. This breakthrough is probably accounted for the growing importance of electroless plating among the electrodeposition methods for the generation of newer electroless composite coating having outstanding attributes for critical tribological and mechanical applications [14–15].

Over the years, a wide variety of micro and nano hard particles (TiC, WC, SiC, Si_3N_4, TiO_2 etc.) have been successfully codeposited via the electroless nickel deposition process to achieve a superior wear-resistance [16–20]. Besides, some electroless codeposits containing soft lubricating particles such as polytetrafluoroethylene [19–22], molybdenum disulphide [5], tungsten disulphide [23–24], carbon nanotubes [6, 25] and graphite [9, 25] have been developed to obtain self-lubricating electroless composite coatings. Since most of these solid lubricant coatings are normally employed for high temperature lubrication, the survey of the available reports showed that the tribological performance of these coatings are limited within the temperature range of 350°C to 500°C. A further increase in wear temperature will result to unsatisfactory wear performance characteristics [2, 10, 14].

Hexagonal boron nitride (h-BN) is another important solid lubricant which has been proven to offer high thermal stability and chemical inertness combined with an excellent lubricating property.

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The h-BN lubricant is therefore a promising alternative candidate material for solid lubrication that involves high temperature, a vacuum environment and where it is difficult to use liquid lubricants and other traditional solid lubricants [26]. However, reports of relevant studies have shown that h-BN has been relatively the least explored in tribology for developing surface tailored composite coatings owing to the accounts of its poor wettability with metal/ceramic matrix as well as its inadequate thermo-oxidative performance [27–28]. Moreover, h-BN coatings have been demonstrated to exhibits poor frictional control at operating temperature below 400°C which restricts their proposed application to high-temperature lubrication [27]. Recent research made by Liu et al [4], indicated that incorporation of h-BN lubricant particles within matrix layer of active metal such as nickel could be explored to improve the wettability performance of h-BN. Based on this perspective, it is of interest to improve the physical and chemical behavior of h-BN via electroless nickel plating with a view to expanding its performance window for the development of an efficient tribo-composite coating system. Although a study by Du et al [26] related the use of Ni-coated h-BN for laser treated composite coating, a detail insights on the electroless deposition of nickel deposit on h-BN particles has remained a subject of vast interest.

In the present work, therefore, electroless plating technique has been explored to co-deposit h-BN particles with Ni-P coating. The deposition routes for the pretreatment of h-BN particles and electroless nickel deposition were reported in detail. Besides, the surface morphologies and composition of the Ni-P coated h-BN powder were investigated. It is believed that this nickel plating has the potential to improve the wettability of h-BN particles with metallic and ceramic matrices.

2. EXPERIMENTAL

2.1. Surface preparation of h-BN powder

The hexagonal boron nitride particles (98% purity, 5 µm) considered for electroless plating in this investigation were supplied by MK Impex Corporation, Canada. Table 1 provides the details of the properties of the h-BN lubricant particles. Since h-BN particles are non-conductive and lack catalytic surface, the as-received h-BN powder had to be activated through a series of pretreatment operation which includes cleaning, chemical etching, sensitization and activation. This step-by-step treatment was performed to make the surface of the powder clean and active before the electroless Ni-P deposition [10]. In this work, the pure h-BN powder was first ultrasonically soak-cleaned with detergents and filtered using a microfilter paper. The powder retained on the paper was thoroughly washed with distilled water to achieve a pH of 7.0, rinsed in ethanol and then dried in an oven at a temperature of 60°C for 2 hours. This surface cleaning step was considered in order to remove the dust and any oxide films present on the surface of the particles. Etching treatment was subsequently conducted by dispersing the cleaned powder in a strong oxidizing acid solution containing 40 ml/L hydrofluoric acid (HF), 80 ml/L nitric acid (HNO_3), 2 g/L ammonium fluoride (NH_4F) and distilled water. The operation was carried out under an ultrasonic wave stirring for 15 min at 40°C in order to produce particles with micro-roughened surfaces. The roughened h-BN particles were then rinsed with distilled water, which was then followed by filtering and drying in an oven at a temperature of 60°C for 2 hours. Finally, a two-step sensitization and activation processes was carried out at room temperature in order to render the lubricant particle surfaces catalytically active for the electroless nickel deposition. The chemically etched samples were then sensitized in an acid stannous chloride solution (2 g/L of $SnCl_2$, 40 ml/L of 3M HCl) followed by distilled water rinse. Duration of 15 minutes was adopted for the sensitization process. Afterwards, the sensitized h-BN particles were ultrasonically treated with palladium chloride solution (0.2 g/L $PdCl_2$, 40 ml/L of 3M HCl) for activating their surface up to duration of 15 minutes. The palladium activated h-BN particles were then filtered, rinsed with distilled water several times and allowed to dry in an oven at 60°C for 2 hours. The basic stages for the pretreatment of the h-BN powder are summarized as shown in Figure 1.

Table 1. Details of h-BN lubricant particles

Properties	Details
Colour	White
Chemical formula	BN
Crystal structure	Hexagonal
Density	2.3
Coefficient of friction (CoF)	0.15–0.7
Temperature stability	1000°C (air), 1400°C (Vacuum)
Thermal conductivity	0.08 cal/cm.sec.k

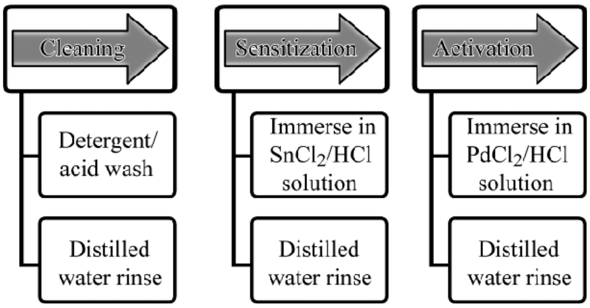


Fig. 1. Typical steps during pretreatment of h-BN powder.

2.2.Electroless plating of h-BN particles

After the pretreatment operations, palladium-activated h-BN particles were introduced into an acid electroless plating bath. The schematic representation of the electroless coating bath system is shown in Figure 2. The plating solution contains sodium hypophosphite as the reducing agent and nickel sulphate as the source of nickel ions. A combination of sodium citrate and glycine was added as complex agents in order to avoid spontaneous a decomposition of the bath by exerting buffering action and retarding the precipitation of nickel phosphites. Furthermore, drops of a cationic surfactant in the form of Cetyl-Trimethyl Ammonium Bromide (CTAB) were added to the bath solution in order to ensure a uniform dispersion of the suspended particles. The plating process was performed for the period of 3 hours and intermittently, it was agitated in the present magnetic stirring. During the deposition process, the bath solution was continuously monitored and kept at the temperature and pH of 85 (± 2°C) and 4.6 (± 0.1) by means of a Solotone pH meter and a thermometer. The low pH was realized through the addition of an aqueous solution of 1M H_2SO_4. After plating, the electroless h-BN coated powders were filtered, thoroughly, cleaned with distilled water and dried at a temperature of 60°C for 2 hours. All the chemicals used in this experiment were analytical of a reagent (A.R) grade. The details of the compositions and operating parameters used for the preparation of the N-P/h-BN codeposited powder are given in Table 2.

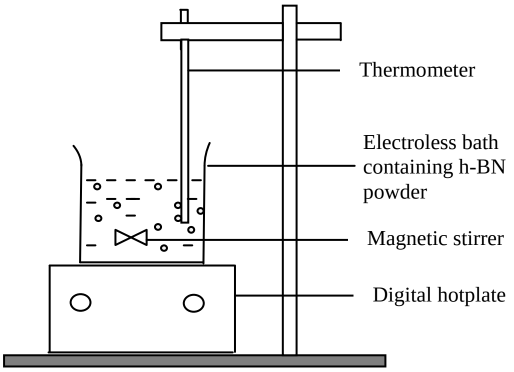


Fig. 2. Schematic representation of the electroless deposition bath.

2.3. Particles characterization

The surface morphologies of the as-received and the electroless coated powders were observed using a JEOL-JSM-5410 scanning electron microscope (SEM). The same equipment was also used to study the cross-sectional microstructures of the coated particles. The samples for the cross-section studies were sprayed on the epoxy, polished and gold sputtered to reveal the network of the Ni-P around the h-BN particles. The energy dispersive X-ray (EDX) spectroscopy was used to analyze the chemical compositions of the powders. The elemental mapping analysis of the Ni-P coated h-BN sample was observed by an EDX attached to a field emission scanning electron microscope (FESEM).

3.RESULTS AND DISCUSSION

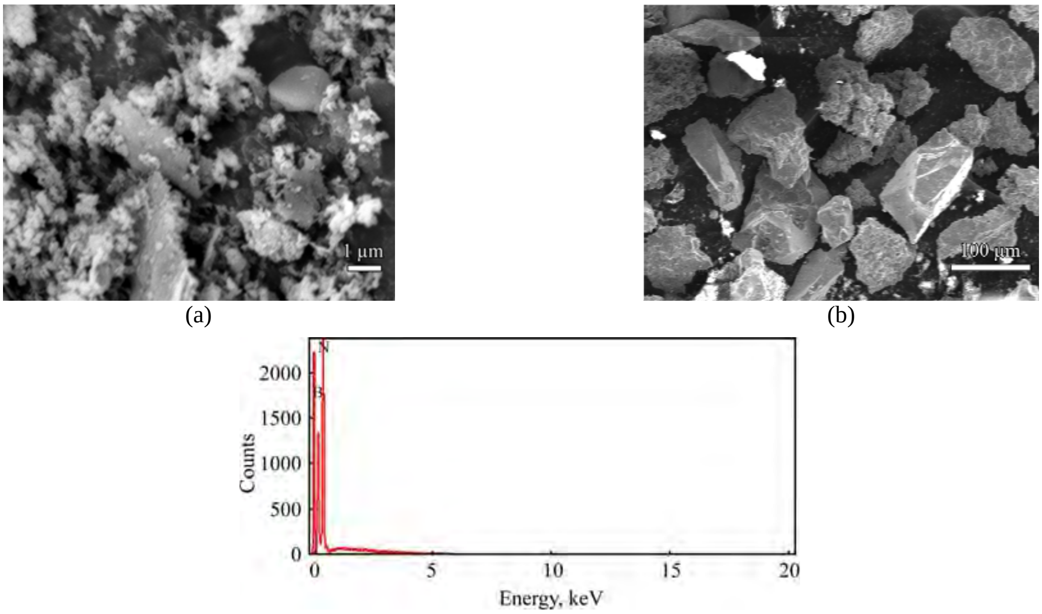
3.1. As-received and pretreated particles characterization

The surface morphology of the as-received h-BN powder examined by scanning electron microscopy is shown in Figure 3a. As can be observed from the micrograph (Fig. 3a), the as-received h-BN powder presents a smooth and near-spherical shaped particles. Figure 3b shows the SEM image of the pure h-BN after the cleaning treatment. It is evidenced from the SEM image that all the attached debris (such as dust, oil, oxide layer) has been removed when compared to the original powder. This feature was considered beneficial for the successful surface activation and plating processes which largely depends on the removal of all such defects from the substrate surface to ensure plated layer are absorbed into the surface of the substrate and not to the attached contaminations [13]. Figure 3c shows a typical energy dispersive X-ray (EDX) pattern of the untreated h-BN powder. The result of the EDX spectrum confirmed that the substrate particles mainly compose of boron and nitrogen, an indication that the ceramic particles used in this work are boron nitride.

The results of the h-BN powder after the micro-etching and activation treatments are presented in Figure 4. It was found that the morphology of the h-BN particles was changed from a spherical-like to irregular sheets after these treatments. The purpose of pretreatment operations was to achieve a lubricant powder surface with an increased surface area, plus good catalytic behavior. As evident from the micrographs in Fig. 4a, the surface of the pretreated powder became roughened through the creation of desirable pits which by this means exposed their surface area for more catalytic activities. Some researchers have observed that the presence of these large roughened surface areas on the pretreated powder would promote the adsorption process of the nucleating agents to the substrate as well as the continuous growth of the Ni-P layer on the substrate particles during the electroless plating process [12–13]. An EDX analysis of the tin and palladium treated h-BN samples were given in Fig. 4b,c respectively. The results revealed that the major components in the treated layers are tin and palladium and had no impurities. These finding clearly demonstrate that the cleaning and chemical etching stages were successful in allowing the surface

Table 2. Compositions and operating parameters of electroless Ni-P deposition bath

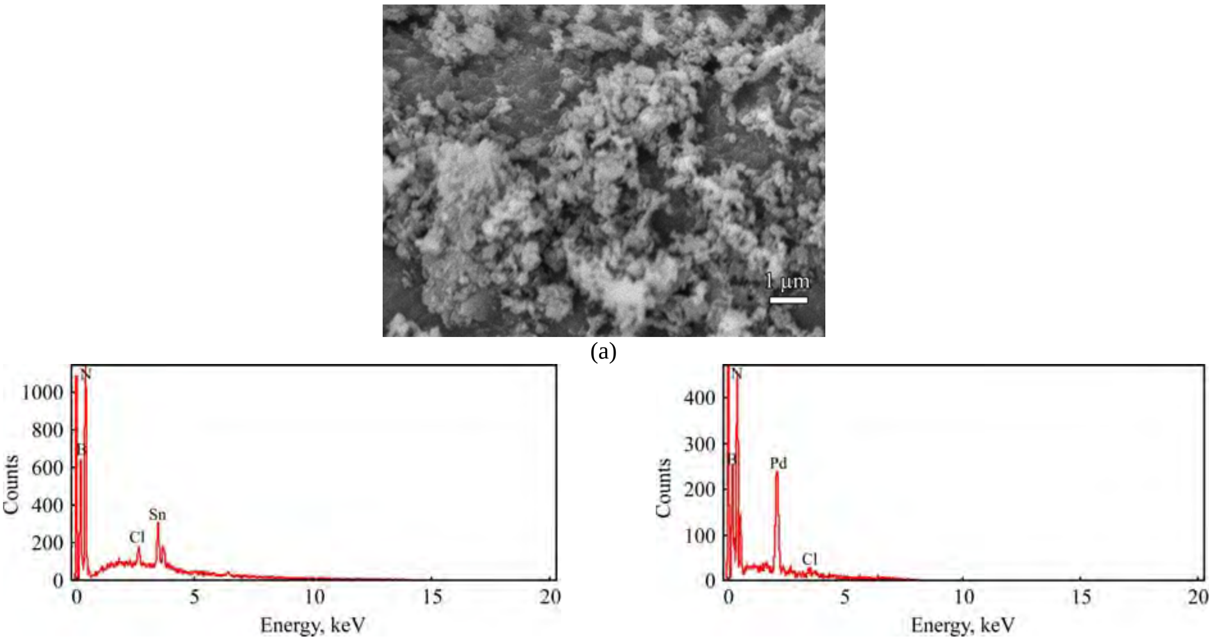
No	Bath Constituents	Components	Quantity (g/ltr)
1	Nickel sulphate, hexahydrate, $NiSO_4{\cdot}6H_2O$	Nickel ions source	30
2	Sodium hypophosphite, monohydrate, $Na_2H_2PO_2{\cdot}H_2O$	Reducing agent	20
3	Tri-sodium citrate, hexahydrate, $Na_3C_4H_6O_4{\cdot}6H_2O$	Complexing agent	50
4	Ammonium sulphate (NH_4SO_4)	pH regulator	20
5	Glycin,$C_6H_5Na_3O_7$	Complexing agent	10
6	Lead nitrate, $PbNO_3$	Stabilizer	0.02
7	Cetyl-trimethyl ammonium bromide, CTAB	Surfactant	0.3
8	Pretreated hBN powder content	Substrate powder	5
Operating conditions			
9	pH	4.6 ± 0.1	
10	Temperature (^oC)	85 ± 2	
11	Magnetic agitation rate (rpm)	300	
12	Duration (h)	3	



(a) (b)

(c)

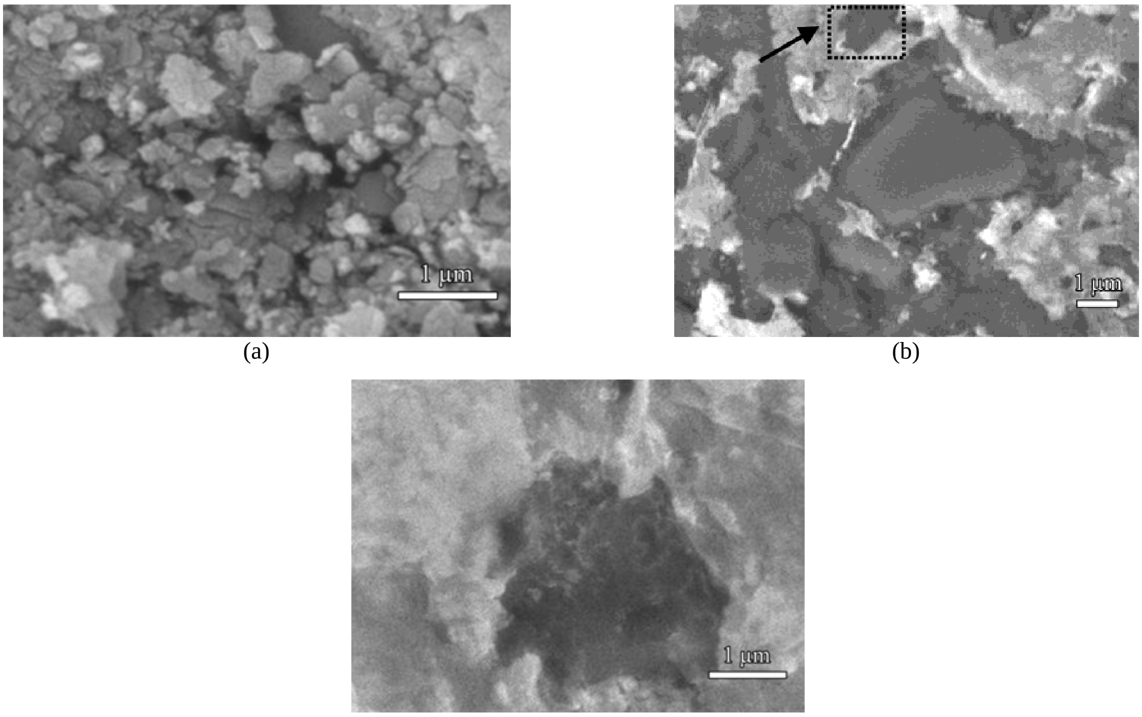
Fig. 3. Scanning electron micrographs of: (a) as-received h-BN powder before ultrasonic-assisted cleaning; (b) as-received h-BN powder after ultrasonic-assisted cleaning and (c) EDX of as-received h-BN powder.



(a)

(b) (c)

Fig. 4. Scanning electron images of: (a) as-pretreated h-BN sample; (b) EDX spectrum result of h-BN sample after sensitization treatment and (c) EDX spectrum result of h-BN sample after activation treatment.



(a) (b)

(c)

Fig. 5. Surface morphologies of h-BN particles after electroless nickel plating: (a) SEM micrograph of Ni-P deposited h-BN particles; (b) SEM cross-section micrograph of Ni-P coated h-BN particles and (c) High magnification of the marked region in (b).

adsorption of these nucleating agents on top of the substrate surface. The colour of the substrate powder changed from white to a brownish colour after the pretreatment process.

3.2. Characterization of Ni-P coated h-BN particles

Figure 5 shows the SEM morphologies of the h-BN lubricant particles after an electroless Ni-P plating process. The appearance of the electroless Ni-P coated particles in Fig. 5a reveals a uniform distribution of the Ni-P deposits around the surface of the h-BN particles indicating one of the best features of electroless nickel plating. The area marked with the arrow pointers in the micrograph (Fig. 5a) indicates there were traces of some Ni and P particles freely formed on the surface of the coated particles. A similar result has been observed by Luo et al. [13] in their electroless plating of Cr_3C_2 powders. During the electroless deposition, a very vigorous reaction was found to have occurred at the initial stage of the plating reaction in the electroless bath. This observation might be attributed to the evolution of hydrogen gas which resulted from the catalytic dehydrogenation of the hypophosphite reducing agent during the electroless reduction of Ni^{2+} species (from $NiSO_4$). The summaryof the overall reaction on the plating process could be expressed by Eq.1 and Eq. 2:

$$Ni^{2+} + 2H_2PO_2^- + 2H_2O \xrightarrow{\text{catalytic surface}} Ni^0 + 2H_2PO_3^- + 2H^{2+} + H_2, \quad (1)$$

$$H_2PO_2^- + 2H_2O + 2H^{2+} \xrightarrow{\text{catalytic surface}} 2P + 4OH^- + 2H_2. \quad (2)$$

As indicated in the reactions shown in equation (1), the catalytic reduction and deposition of nickel/phosphorous ions on the activated powder have been initiated by the adsorbed H^+ from the aqueous solution. The presence of Pd deposits on the pretreated powder surface serves as catalytic sites for the initial deposition of Ni-P layers. Owing to the self-catalytic nature of nickel particles, the just-deposited nickel films, then acted as a nucleation centre and catalyst for subsequent electroless deposition as the plating reaction continues based on the equation (1) & (2). In this case, the nickel-phosphorous layers continued to grow until the reaction was completed and thus formed a dense Ni-P coated layer on the h-BN powder.

The SEM cross-section morphologies of the electroless coated h-BN particles are shown in Fig. 5b,c. The surface analysis of the cross sectional view revealed that the coated particles were successfully isolatedwithin the island of cell-like structures which were analyzed to consist of Ni and P particles (Fig. 5a). It was observed from Fig. 5b (the high magnification image of a marked box in Fig. 5a) that the co-deposited particles are tightly bound to the Ni-P matrix layers forming a compact structure. Taking into account the fact that hBN particles exhibits poor wettability with most metallic and ceramic matrix to form an effective composite structure, this new finding therefore suggests that the electroless plating process was beneficial to improve the surface functionality of h-BN through the existence of a highly active nickel deposit on the substrate particles. The EDX pattern of h-BN particles after the electroless Ni-P plating is shown in Figure 6. The spectra depicting the peak of the x-ray for nickel and phosphorous were observed other than boron and nitrogen. This clearly confirms that the major elements of the as-deposited coatings are Ni and P. The quantification analysis of the coated powder surface was estimated to contain approximately 88 wt.% Ni and 12 wt.% P (as shown in the spectrum of Figure 6).

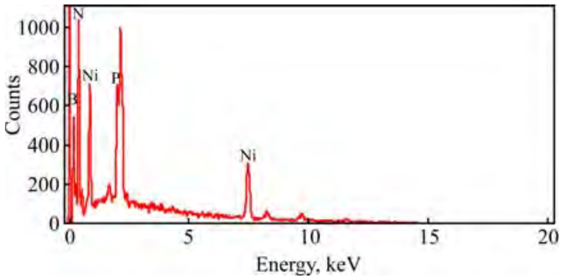


Fig. 6. EDX spectrum of Ni-P coated hBN powder.

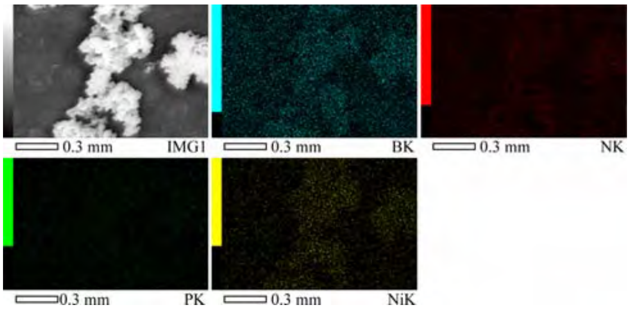


Fig. 7. Elemental mapping profile of Ni-P coated hBN particles.

The SEM-EDX mapping analysis obtained for the electroless Ni-P coated powder is given in Figure 7. The map profile shows the presence of B, N, Ni and P in the composite coated powder. It can be observed that these elemental constituents were uniformly distributed as measured from the respective colour intensities of each of the elemental constituent. After the electroless plating, a visual observation showed that the colour of the activated powder turned from brown to black.

4. CONCLUSION

Based on the results obtained in this study, it is possible to the following conclusions:

1. The step-like pretreatment processes was demonstrated to be effective in producing catalytic centres on the surface of h-BN particles favouring the adsorption of the nucleating agents as well as the nucleation and growth of Ni-P layer during the electroless plating.

2. Nickel-phosphorous coatings were satisfactorily obtained on the h-BN substrate. The Ni-P deposit was found to be dense and uniformly distributed on the h-BN lubricant particles.

3. The existence of Ni layer on h-BN particles would improve their wettability for efficient metal-ceramic adhesion.

4. The success of Ni-P/h-BN codeposition could be regarded as a stimulus to develop advanced tribocomposite materials suitable surface-reinforced coatings and powder metallurgical processes.

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Реферат

В течение последнего десятилетия наблюдался устойчивый прогресс применения химического осаждения покрытий для решения задач модификации поверхностных свойств керамических материалов с целью получения композиционных покрытий с уникальными характеристиками для критических трибологических систем. В этой работе был использован процесс химического осаждения никеля для нанесения никель-фосфорных (Ni-P) покрытий на частицы гексагонального нитрида бора (г-BN) из кислотного раствора ванны с пониженным содержанием гипофосфита. Частицы субстрата были первоначально подвергнуты ряду операций предварительной обработки для обеспечения очистки частиц и их каталитической активности перед химическим осаждением покрытия. Характеризация частиц порошка в исходном состоянии и с никелевым покрытием проводилась методами сканирующей электронной микроскопии (SEM), рентгеновской энерго-дисперсионной спектроскопии (EDX), автоэмиссионной сканирующей электронной микроскопии (FESEM). Результаты исследования показывают, что предварительная обработка порошка гексагонального нитрида бора обеспечивает получение частиц с поверхностями с грубой и шероховатой структурой, которые, как правило, считаются подходящими для нанесения покрытий Ni-P. Кроме того, EDX анализ подтверждает существование зародышеобразователей и Ni-P покрытия на поверхности обработанного порошка г-BN. Анализ микроструктуры поперечного сечения порошка с покрытием показал, что частицы г-BN были встроены в непрерывный матричный слой осадка Ni-P. Профили распределения элементов по EDX-анализу также показывают, что масса осажденного Ni-P сплава равномерно распределена на поверхности г-BN частиц с соосажденным Ni-P. Успешное развитие процесса получения порошка г-BN с Ni покрытием позволит повысить потенциал применения порошка гексагонального нитрида бора в качестве материала для покрытий высокого качества.

Ключевые слова: химическое осаждение, г-BN частицы, травление, Ni-P слой, модификация поверхности.