Manufacture and Properties of Ni-B-Fe$_2$O$_3$ Composite Nano-coatings by Electrodeposition

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Abstract

Present study deals with electrodeposition of binary Ni-B and ternary Ni-B-Fe$_2$O$_3$ alloy coatings using DMAB as a source of boron. The coatings were characterized for elemental composition, phase composition and surface topography using FE-SEM, XRD and Atomic Force Microscopy (AFM). The phase stability of coatings was studied using differential scanning calorimetry (DSC). Subsequently, the mechanical and electrochemical corrosion studies were carried out using nano indentation and potentiodynamic polarization studies respectively. The results indicate that addition of Fe$_2$O$_3$ in Ni-B alloy coatings results in 52% improvement in hardness and 3 times improvement in corrosion protection efficiency over Ni-B coatings. Ni-B-Fe$_2$O$_3$ coatings can be suitable economical option towards conventional electroless Ni-B alloy.

Keywords: Electrodeposition; Ni-B; Corrosion; AFM; Ni-B-Fe$_2$O$_3$; Hardness

Terminology
Ni-B-X: Alloy of Ni, B containing X element or oxide
Ra: Roughness Average of a surfaces measured microscopic peaks and valleys DMAB: Dimethylamine borane (Source of boron)
ICP-AES: Inductively coupled plasma atomic emission spectroscopy (is an analytical technique used for the detection of chemical elements)
EDX: Energy-dispersive X-ray spectroscopy (is an analytical technique used for the elemental analysis or chemical characterization of a sample)
FE-SEM: Field emission scanning electron microscopy (FESEM) provides topographical and elemental information at magnifications of 10x to 300,000x, with virtually unlimited depth of field
AFM: Atomic force microscopy (AFM) or scanning force Microscopy (SFM) is a very-high-resolution type of scanning probe microscopy (SPM), with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction limit
DSC: Differential scanning calorimetry is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature
FCC: Face centred cubic crystal structure
η: Corrosion protection efficiency

Introduction

The mechanical, chemical, electrochemical and thermal interaction of a material in a given environment initiates from its surface. So, the surface of a material is the most important engineering part. For the past few decades, among the standards which are developing continuously for production and manufacturing process, the use of surface modification/finishing technologies in the manufacturing process is indispensable. Surface finishing is used in various industrial fields to modify the surface properties of materials like durability, corrosion resistance, tribological properties and electrical conductivity. The tribological and corrosion phenomenon impose a huge damage to economy and industry. Therefore, the surface modification technology can up to some extent control the threat of wear and corrosion. The use of appropriate surface technologies can prevent the damages or at least delay them. Further, the selection of suitable process usually requires the economic and environmental concerns. One of such suitable processes is Electrodeposition. It is an electrochemical process which has a long history and is a cost effective process to produce a coating. In this process, a monolayer or multilayer coating can be formed from the electrochemical reactions occurring at electrode/electrolyte interface and reduction of ions from the plating bath. Literature relevant to composite Electrodeposition suggests reports on a variety of Ni-P-X composite coatings (X=W, Fe$_2$O$_3$ etc.). However, studies on Ni-B-X alloy coatings are rare with the exception of Ni-B-Mo [1] Ni-B-Ti [2], Ni-B-P [3] and Ni-Co-B [4]. As far as Ni-B is concerned, it can be deposited either by electroless [5] or electrodeposition [6] resulting in promising properties with excellent hardness, wear and corrosion resistance properties as Ni-B is one of the hardest alloys which can be electrochemically prepared. Ni-B coating especially electroless Ni-B and its ternary alloys are widely used in aerospace, nuclear, petrochemical, electronics, plastic molding, and food processing industries due to its excellent wear resistance, low friction coefficient, better solderability, and coating uniformity with minimal porosity [7]. In general, the B content of Ni-B coatings in application is 0.1-10 wt.%. These coatings are more corrosion resistant than electroless Ni-P and higher hardness than commercial hard chrome coatings when heat treated. However, Ni-B coatings deposited through electroless deposition suffer from...

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sluggish deposition rates, low bath life, less control over deposition composition. Ni-B coatings have higher melting point (1350-1360°C) compared to their Ni-P (880-1200°C) counterparts depending upon B and P content. This leads to their (Ni-B) excellent thermal stability. In addition, Ni-B coatings have low electrical resistivity (89 × 10⁶ Ω·cm) which results in their application in electronics industries.

Although Ni-B coatings have better properties over Ni-P, the former are more expensive than latter. Therefore, in order to reduce the cost of deposition we introduced Fe₂O₃ as an element with Ni-B to get Ni-B-Fe₂O₃ ternary alloy. The use of ternary alloying element in Ni-B has been reported to give excellent magnetic properties (e.g. Ni-B-Mo, Ni-B-Ti, Ni-Co-B). These coatings depict superior chemical resistance, thermal stability, excellent electrical, non-magnetic properties [8]. Our earlier work on Ni-B-Zn [6] coatings demonstrated improved mechanical properties and corrosion resistance compared to Ni-B coatings.

The main aim of the present work is to study the influence of Fe₂O₃ incorporation in Ni-B matrix on the structure, hardness and corrosion resistance of Ni-B-Fe₂O₃ composite coatings. For that purpose, we have introduced DMAB as source of B in ternary Ni-B-Fe₂O₃ alloy.

**Experimental Details**

Ni-B and Ni-B-Fe₂O₃ composite coatings were deposited on mild steel substrates having size of 22 × 2 mm. All the mild steel substrates were mechanically ground using different SiC papers 320-4000 to remove any contamination present and to achieve a flat mirror-like surface finish. The substrates were then cleaned in an alkaline solution and thoroughly washed with distilled water. At last, before immersing into the solution bath, the substrates were cleaned in an alkaline solution and thoroughly washed with distilled water. The composition of binary Ni-B and ternary Ni-B-Fe₂O₃ composite coatings were deposited on mild steel substrates having size of 22 × 2 mm.

The bath chemical composition, quantity, and working conditions are presented in Table 1. The chemical composition and working conditions adopted for electrodeposition of Ni-B and Ni-B-Fe₂O₃ coatings are tabulated in Table 1. The coating bath consists of nickel sulphate (source of nickel), dimethylamine borane (DMAB, source of boron), boric acid (complexing agent) and iron oxide (as a source of iron). The choice of DMAB is due to its stability over a wide range of pH as compared to borohydrides [9].

Pure nickel plate was employed as anode and low carbon steel substrate was used as cathode. The coating deposition was conducted at 55 ± 2°C, for a time period of 30 minutes, during which the bath was vigorously agitated with a magnetic stirrer. The Ni-B-Fe₂O₃ coatings were synthesized by adding iron oxide into the Ni-B solution. The chemical composition of the synthesized coatings was determined with ICP-AES (thermo iCAP, 6500, USA) and EDX analysis. X-ray diffraction analysis was carried out to check the phase purity and structure of synthesized coatings. X-ray Diffractometer (Rigaku, miniflex 2 desktop, Tokyo, Japan) equipped with Cu-Kα radiations. The analysis of the surface morphology of the coatings was determined with the help of FE-SEM (FE-SEM-Nora Nano 450; Netherlands). The AFM device MFP-3D atomic force microscopy (USA) equipped with a silicon probe (Al reflex coated veeeco model-OLTESPA, Olympus, spring constant: 2 N/m, resonant frequency: 70 kHz) was used in the experiments. The phase transformation of the coatings was studied with differential scanning calorimetry (DSC, JADE, Perkin Elmer). The scans were conducted from room temperature to 450°C using argon as protective medium. The nano-mechanical properties (hardness and elastic modulus) of Ni-B and Ni-B-Fe₂O₃ coatings were determined using MFP-3D NanoIndenter with Standard tip indenter (with typical spring constant equals 4000 N/m). The Berkovich diamond indenter tip composed of an industrial diamond brazed to a screw-threaded hex toolied metal chuck was used. The indentation measurement was carried out using 1 mN of the indentation force. The contact depth was estimated from the unloading curve using Oliver and Pharr method. Results about nano-mechanical properties were evaluated from five different indentations zones. The corrosion resistance of the synthesized coatings in their as deposited state was investigated by Potentiodynamic linear polarization technique employing Gamary 3000 (30K BOOSTER potentiostat/Galvan state/ZRA, USA). The linear polarization scans were conducted at room temperature in 3.5 wt.% NaCl aqueous solution using conventional triple electrode system.

**Results and Discussions**

**Phase composition and surface morphology**

The composition of binary Ni-B and ternary Ni-B-Fe₂O₃ composite coatings data is presented in Table 2. It is evident that both B and Fe₂O₃ can be successfully co-deposited with Ni using present experimental conditions. The chemical composition of Ni-B and Ni-B-Fe₂O₃ composite coatings is also obtained using EDX as shown in Figure 1. The presence of Ni and B can be clearly observed in Figure 1a. In Figure 1b, in addition to nickel and boron, Fe₂O₃ peaks also appeared in the EDX pattern which confirms the co-deposition of Fe₂O₃ into the nickel matrix. These results suggest that Fe₂O₃ can be successfully co-deposited in Ni-B coatings using dimethylamine borane under present bath conditions.

The incorporation of B in binary and ternary alloy is attributed to the presence of DMAB in electrolyte which decomposes to elemental B upon adsorption at the growing Ni lattice during electrodeposition. The results are in agreement with our earlier work [10]. Similar observation was made by Krishnaveni et al. [9]. The presence of Fe₂O₃ in solution however led to decrease in Ni as well as B content of ternary Ni-B-Fe₂O₃ alloy when compared with binary Ni-B. Fe₂O₃ is

| Bath chemical composition | Quantity | Working conditions |
|---------------------------|----------|--------------------|
| NiSO₄.6H₂O (Nickel sulphate) | 240 (g, L) | pH 4.0-4.5 |
| NaCl.6H₂O (Nickel chloride) | 45 (g, L) | Bath temperature 55 ± 2°C, Deposition time 30 min |
| H₃BO₃ (Boric acid) | 30 (g, L) | Current 50 mA/cm², Stirrer speed 600 rpm |
| DMAB (Dimethylamine borane complex) | 3 (g, L) | Current 50 mA/cm², Stirrer speed 600 rpm |
| Iron oxide particles | 0-15 (g, L) | Stirrer speed 600 rpm |

**Table 1:** Chemical composition and working conditions for electrodeposition.

| Coatings | Ni (Wt.%) | B (Wt.%) | Fe₂O₃ (Wt.%) |
|----------|-----------|----------|--------------|
| Ni-B     | 84.12     | 15.88    | -            |
| Ni-B-Fe₂O₃ | 57.46     | 2.65     | 39.89        |

**Table 2:** Compositional analysis of Ni-B and Ni-B-Fe₂O₃ composite coatings.
The surface morphology of binary Ni-B and ternary Ni-B-Fe\textsubscript{2}O\textsubscript{3} was analyzed under SEM. The results are shown in Figure 3. As also observed by other investigators [17] and our earlier work [10], the Ni-B coatings depict nodular or typical “cauliflower” type morphology. Similar morphology is also evident for Ni-B-Fe\textsubscript{2}O\textsubscript{3} alloy. It has been observed that, the microscopic nodular size is controlled by segregation of metalloid in Ni during electrodeposition. Wasekar et al. [18] presented the mechanism of morphology formation in Ni coatings in presence of metalloid (e.g. S, B etc.) which has negligible solid solubility in Ni. They observed that, when metalloid segregates at grain boundary, larger is metalloid content of Ni coating (due to large GB area), smaller is grain size of Ni and the larger is size of nodule. This happened when lateral growth is prevailed than vertical growth. The surface roughness was lower for large nodular coatings. This phenomenon is attributed to segregation of metalloid at grain boundary or colony boundary due to its low solid solubility. Upon analogy with Wasekar et al. [18], when B segregates at grain boundary, it results in extremely fine grain size as evident in Ni-B alloy. Due to large grain boundary area as compared to nodule boundary, the higher B content results in fine grain size and larger, smooth nodule size. When B segregates at nodule boundary whose surface area is less compared to grain boundaries, the nodule size is smaller and grain size larger. The results are consistent with XRD results where Ni-B coatings with higher B content are amorphous than Ni-B-Fe\textsubscript{2}O\textsubscript{3} alloy which are nanocrystalline in nature. The above phenomenon is also confirmed using 3D AFM at submicron level. The results are shown in Figure 4a and 4b. The Ni-B coatings illustrate smooth growth morphology at submicroscopic level due to large nodule size. This in turn is known to inhibit the deposition of alloying elements as per anomalous deposition mechanism [11,12]. In this mechanism, Fe\textsubscript{2}O\textsubscript{3} being less noble (standard electrode potential: -0.44 V) inhibits the deposition of more noble Ni (-0.25 V). The results are consistent with work of Wang [13] where increase in Fe\textsubscript{2}O\textsubscript{3} content at the expense of P in Ni-P-Fe\textsubscript{2}O\textsubscript{3} alloy was observed.

The X-Ray diffraction (XRD) pattern corresponding to Ni-B and Ni-B-Fe\textsubscript{2}O\textsubscript{3} coatings are presented in Figure 2. For comparison purpose, the XRD pattern of substrate is also depicted. The pattern suggests the presence of single phase binary and ternary FCC Ni alloy. Ni-B alloy displays only single broad peak at 2θ ~ 43.78° suggesting amorphous nature of these coatings. Two small peaks at 2θ ~ 65° and 82° correspond to Fe substrate. Since solid solubility of metalloid B (atomic radius: 1.4 Å) in Ni (atomic radius: 1.62 Å) is negligible at room temperature, the crystalline Ni-B coatings may not be possible. Therefore, Ni-B alloys are generally supersaturated solid solution of B in Ni. It has been observed by several investigators [14-16] that for B content above 5 wt.%, Ni-B alloys are amorphous in nature. Ni-B alloy with 3-5 wt.% B are mixture of crystalline and amorphous phases whereas those with less than 3 wt.% B are crystalline in nature.

In contrast to Ni-B, the Ni-B-Fe\textsubscript{2}O\textsubscript{3} coatings are represented by comparatively sharp peaks at 2θ ~ 43.78 and two small peaks at 2θ ~ 51.85° and 75.08° corresponding to (111), (200) and (220) crystallographic planes of FCC Ni. The addition of Fe\textsubscript{2}O\textsubscript{3} in Ni-B led to crystalline nature of ternary Ni-B-Fe\textsubscript{2}O\textsubscript{3} alloy which can be attributed to lower B content than binary Ni-B as evident in Table 2.

The surface morphology of binary Ni-B and ternary Ni-B-Fe\textsubscript{2}O\textsubscript{3} was analyzed under SEM. The results are shown in Figure 3. As also observed by other investigators [17] and our earlier work [10], the Ni-B coatings depicts nodular or typical “cauliflower” type morphology. Similar morphology is also evident for Ni-B-Fe\textsubscript{2}O\textsubscript{3} alloy. It has been observed that, the microscopic nodular size is controlled by segregation of metalloid in Ni during electrodeposition. Wasekar et al. [18] presented the mechanism of morphology formation in Ni coatings in presence of metalloid (e.g. S, B etc.) which has negligible solid solubility in Ni. They observed that, when metalloid segregates at grain boundary, larger is metalloid content of Ni coating (due to large GB area), smaller is grain size of Ni and the larger is size of nodule. This happened when lateral growth is prevailed than vertical growth. The surface roughness was lower for large nodular coatings. This phenomenon is attributed to segregation of metalloid at grain boundary or colony boundary due to its low solid solubility. Upon analogy with Wasekar et al. [18], when B segregates at grain boundary, it results in extremely fine grain size as evident in Ni-B alloy. Due to large grain boundary area as compared to nodule boundary, the higher B content results in fine grain size and larger, smooth nodule size. When B segregates at nodule boundary whose surface area is less compared to grain boundaries, the nodule size is smaller and grain size larger. The results are consistent with XRD results where Ni-B coatings with higher B content are amorphous than Ni-B-Fe\textsubscript{2}O\textsubscript{3} alloy with low B content which are nanocrystalline in nature. The above phenomenon is also confirmed using 3D AFM at submicron level. The results are shown in Figure 4a and 4b. The Ni-B coatings illustrate smooth growth morphology at submicroscopic level due to large nodule size. This in turn is known to inhibit the deposition of alloying elements as per anomalous deposition mechanism [11,12]. In this mechanism, Fe\textsubscript{2}O\textsubscript{3} being less noble (standard electrode potential: -0.44 V) inhibits the deposition of more noble Ni (-0.25 V). The results are consistent with work of Wang [13] where increase in Fe\textsubscript{2}O\textsubscript{3} content at the expense of P in Ni-P-Fe\textsubscript{2}O\textsubscript{3} alloy was observed.
contrast to Ni-B-Fe₂O₃ alloy having smaller nodular size suggesting vertical growth as evident in Figure 4b. The more insight at nanoscale can be obtained using AFM surface profile as illustrated in Figure 5. As evident in Figure 5a and 5b, Ni-B alloy coatings appear to be smooth as compared to their ternary Ni-B-Fe₂O₃ counterparts. The corresponding surface profile of these coatings is presented in Figure 5c and 5d. The average surface deviation of Ni-B and Ni-B-Fe₂O₃ coatings is 3.125 and 9.793 nm respectively. The above observations are consistent with the work of Lee et al. [17] who reported increase in nodule size and reduction in surface roughness with increase in B content.

Thermal stability

The thermal stability of Ni-B and Ni-B-Fe₂O₃ coatings were studied using DSC technique. As depicted in Figure 6, the DSC curves are represented by exothermic peaks for both Ni-B and Ni-B-Fe₂O₃ alloy. However, substrate do not show any peaks within the temperature range studied. For binary Ni-B alloy, the exothermic peak was observed at ~376°C which is attributed to the formation/crystallization of Ni₂B phase inside Ni-B matrix [9]. For binary Ni-B alloy, the crystallization temperature varies with B content. In fact, increase in B content results in lower crystallization temperature. For B content of 14-16 wt.%, the observed peak at 376°C is in agreement with literature [17]. As shown in DSC curves (Figure 6) the incorporation of Fe₂O₃ in Ni-B, results in decrease in crystallization temperature to 296°C for ternary Ni-B-Fe₂O₃ alloy. This temperature is ascribed to the crystallization of Ni₃Fe intermetallic phase in ternary alloy matrix. The Ni₃Fe phase is low temperature phase when compared to Ni₂B/Ni₃B or Fe₂B phases [19,20] which crystallizes at relatively higher temperatures. Therefore, present results suggest that addition of Fe₂O₃ decreases the thermal stability of Ni-B-Fe₂O₃ alloy.

Mechanical properties

The mechanical properties (hardness and elastic modulus) of Ni-B and Ni-B-Fe₂O₃ coatings were obtained using nano-indentation tests.
using Oliver-Pharr method [21]. The comparison of hardness and elastic modulus of the substrate, Ni-B and Ni-B-Fe₂O₃ deposition in their as deposited condition is as shown in Figure 7. From Figure 7a, it can be noticed that the average hardness of the Ni-B is 12.5 GPa. With the addition of the Fe₂O₃, the hardness has been improved and has increased from 12.5 GPa to 19 GPa. The enhancement in the mechanical properties of composite coatings can be explained by solid solution strengthening mechanism. Similarly, a significant increase improvement in elastic modulus has also been with the addition of Fe₂O₃ into the Ni-B matrix. The elastic modulus has increased from 130 GPa (for Ni-B) to 190 GPa (for Ni-B-Fe₂O₃). The hardness and elastic modulus increased by 52% and 46% respectively. This unusual increase in mechanical properties is attributed to semi-amorphous nature of Ni-B coatings and nano-crystalline nature of Ni-B-Fe₂O₃ coatings where in the dislocation motion is impeded by sub nanocrystalline domains [15,22-24]. The results are in agreement with our previous work [10].

Electrochemical corrosion

The electrochemical corrosion performance of Ni-B and Ni-B-Fe₂O₃ coatings were analyzed in comparison with substrate in 3.5 wt.% NaCl using polarization tests. Potentiodynamic polarization curves for
The calculated corrosion results of the substrate, Ni-B and Ni-B-Fe₂O₃ coatings are shown in Figure 8. The results indicate that addition of Fe₂O₃ into the Ni-B improves the corrosion resistance of the coatings as the Ecorr value is less negative (-479.0 mV) and the corrosion rate (Icorr) is lower for the Ni-B-Fe₂O₃ (7.15 µA) when compared to the substrate and Ni-B deposition. Therefore, Ni-B-Fe₂O₃ coatings are more resistant to corrosion than Ni-B deposition and substrate. The improved corrosion resistance for ternary Ni-B-Fe₂O₃ in comparison with Ni-B is attributed to dense and porosity free coatings as revealed from mechanical property evaluation. The results suggest that Ni-B-Fe₂O₃ coatings are more protective in nature than Ni-B coatings. The protection efficiency (η) of coatings was evaluated using following equation [25],

\[ \eta = 100 \times \frac{\text{i}_{\text{corr (coated)}} - \text{i}_{\text{corr (uncoated)}}}{\text{i}_{\text{corr (coated)}}} \]

The results are presented in Table 3. It is clear that ternary Ni-B-Fe₂O₃ alloy coatings protect the substrate better than Ni-B alloy due to higher protection efficiency. The improvement in corrosion resistance is attributed to dense nature of Ni-B-Fe₂O₃ coatings which is reflected in its mechanical properties (elastic modulus). Given its simplicity, these coatings can lead to further improvements in properties by manufacturing of multilayer electrodeposition process [26].

**Conclusions**

The main aim of the present work was to study the influence of Fe₂O₃ incorporation in Ni-B matrix on the structure, hardness and corrosion resistance of Ni-B-Fe₂O₃ composite coatings. For that purpose, we introduced DMAB as source of B in binary Ni-B and both DMAB and iron oxide as source of B and Fe₂O₃ in ternary Ni-B-Fe₂O₃ alloy respectively. The main conclusion drawn from presented study is as follows:

1. Both binary and ternary Ni-B and Ni-B-Fe₂O₃ alloy coatings were successfully deposited using electrodeposition using DMAB.
2. The addition of iron source reduced the B content of Ni-B-Fe₂O₃ alloy.
3. The surface roughness of ternary Ni-B-Fe₂O₃ was higher than binary Ni-B.
4. The ternary Ni-B-Fe₂O₃ alloy depicted improved mechanical and electrochemical corrosion resistance than their binary Ni-B counterpart.
5. Ni-B-Fe₂O₃ alloy can be best economical option towards conventional electro-less Ni-B alloy.

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