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Experimental investigation on surface roughness of electroless Ni–B–TiO_2 nanocomposite coatings

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Abstract. Magnesium and its alloys like AZ91 are used for various automotive, military and aviation applications. Nanocomposite coatings on AZ91 show significant improvement in the surface properties like wear resistance, hardness and corrosion properties. Electroless coatings have unique physicochemical and mechanical properties, for which they are being used increasingly. Most applications of the electroless coatings are based on their wear and corrosion resistance. However, the characteristic like luminescence has a great potential in defence and aerospace applications. In this research, surface roughness of AZ91 magnesium alloy due to nanocomposite coating processes of ENi–B–TiO_2 is experimentally investigated. It has been observed that as concentration of second-phase (TiO_2) particles increases, the surface roughness of coatings also increases.

Keywords. Coatings; Nanocomposite; Hardness; AZ91 Mg alloy; surface roughness.

1. Introduction

Electroless plating can produce more elastic binding on the entire surface of the shape complex. An electroless process can produce a coating that fits into the adhesive layer. Electroless process can produce coating that is homogeneous across the coating thickness. Electroless-plated coatings are much better than electroplated coating, because the coatings are less porous and provide excellent corrosion protection to steel-based substrates. Non-electronic covers can be fitted with airtight, non-commercial, complimentary and non-conductive components. Complicated jigs or racks are not needed. There is a flexibility of metallicity and size. Chemical recycling can be monitored automatically and a sophisticated filtering method is not required. Electroless coating deposition technique is used for coating alloy or a solid work-piece, such as metal or plastic, also referred to as autocatalytic chemical deposition techniques. Because of the number of advantages, electroless coatings are mostly used in every type of industries [1]. Figure 1 shows the usage of electroless nickel coatings. While having a number of advantages, a few important limitations of electroless coatings are the smaller life span of chemicals and greater cost of waste treatment due to fast chemical regeneration [2, 3].

Electroless nickel coatings have been used either as decorative or protective coatings in industries such as electronics, computer, aerospace, printing, automotive, textile, plastics, optics, paper and food [4, 5]. Some of the exceptional characteristics of electroless nickel coatings are superior corrosion resistance, excellent uniformity, large domain of thickness, good solderability, and improved physical and mechanical properties [6].

Most of the magnesium alloys contain 8–9% aluminium with small amounts of zinc [7]. The addition of several alloying elements such as aluminium, zinc and rare earths has been reported to improve the corrosion resistance, technologically that does not satisfy the requirement for several applications [8, 9].

Hence, the application of a surface engineering technique is the most appropriate method to further enhance the surface properties and corrosion resistance. Among the various surface engineering techniques that are available for this purpose, coating by electroless nickel is of special interest, especially in the electronic industry, due to its conductivity and several other engineering properties Electroless nickel is well known for its corrosion resistance and hardness [10–12].

2. Bath composition and operating conditions of ENi–B–TiO_2 composite coatings

Selection of an appropriate bath along with suitable operating conditions is a key to ensuring desired deposition onto the base substrate. Numerous experiments were carried out prior to selection of the most optimum bath constituents along with their quantities for ensuring successful

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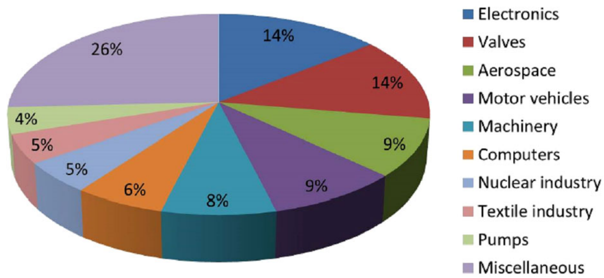

Figure 1. Use of electroless nickel coatings.

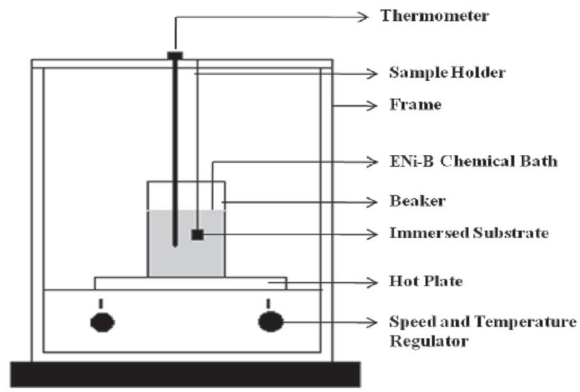

Figure 2. Schematic drawing of experimental set-up.

Table 1. Chemical composition of ENi–B–TiO_2 nanocomposite coating.

Operation conditions	Bath composition
Nickel sulphate hexahydrate (g/L)	30
Sodium borohydride (g/L)	2–4
Ethylenediamine (98%) (ml/L)	50
Hydrofluoric acid (40% V/V) (ml/L)	12
Nano-titanium-oxide (g/L)	0–15
Bath stirrer (rpm)	0–200
Temp.	80–85°C
Immersion time (h)	1.5

deposition onto the substrate. The final chemical composition and operating conditions for successful deposition of ENi–B–TiO_2 nanocomposite deposition on AZ91 magnesium alloy used is summarized in table 1.

In acid bath, for electroless nickel–boron coatings, boron contents vary from 2 to 4%. The components of electroless nickel–boron coatings are very often used in industrial wear applications for their as-plated hardness, which is higher than that of nickel–phosphorus. They have good soldering, brazing and ultrasonic bonding characteristics, when boron is more than 1% [13]. In composite coatings, second-phase particles act as a barrier for plastic deformation of coated layer on the application of load. It results in increase in hardness of composite coatings. In ENi–B–TiO_2 nanocomposite coating, titanium particles will act as the second-phase material.

3. Experimental set-up

The experimental set-up encompassed a carefully prepared electroless bath (chemical bath) with suitable chemicals in adequate quantities, in addition to a hotplate to provide appropriate amount of energy in the form of heat to the bath constituents, to obtain the desired composite deposit. A square plate of AZ91 magnesium alloy having dimensions 20 mm × 20 mm × 1 mm was taken as the base substrate for composite deposition. The square plate was provided with a pin hole drilled close to one of the corners to enable undisturbed suspension in the chemical bath. A schematic of experimental set-up is shown in Figure 2. The surface morphology of the coated substrate was observed under scanning electron microscopy (SEM) to study morphological changes due to the introduction of second-phase particles and at different bath agitations. The effects of different bath agitations (0, 100 and 200 rpm) are also recognized by the different combinations of the composite shown in Figure 3. The composite samples treated at 100 rpm bath agitation show better or uniform deposition as compared with other samples.

X-ray scattering analysis is used to determine the composition of the composite coat by weight percentages of nickel, boron, oxygen and titanium particles. Figure 4(a)–(d) shows the EDX plot of the ENi–B–TiO_2 composite composites for different concentrations of secondary titanium particles (0, 5, 10 and 15 g/L) in the bath.

From the EDX plots it is observed that the percentages of nickel and boron weight decrease and percentage weight of oxygen increases with the increase in the second-phase (TiO_2) particles. Also, it can be observed that the amount of particles getting implanted in the coating increases.

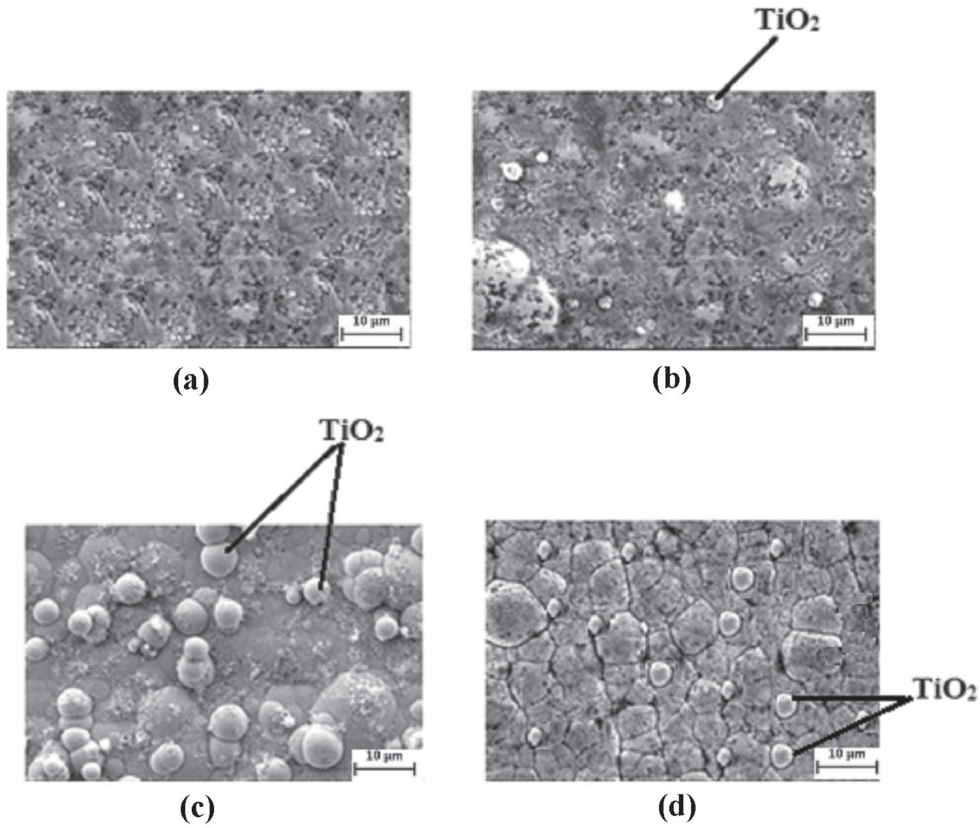

Figure 3. SEM micrographs of ENi–B–TiO_2 composite coatings: (**a**) Ni–B without TiO_2, (**b**) ENi–B–TiO_2 with no agitation, (**c**) ENi–B–TiO_2 with agitation at 100 rpm and (**d**) ENi–B–TiO_2 with agitation at 200 rpm.

4. Measurement of surface roughness of coatings

One of the purposes of using a electroless coating for a surface of the AZ91 for any application has to do with providing smoothness and extending surface roughness properties. The surface roughness of coatings depends on coating parameters and the amount of second-phase particles incorporated in the coated layer. For this purpose, effect of coating parameters on surface roughness ENi–B–TiO_2 composite coatings needs to be investigated.

In order to study the surface roughness of substrate after ENi–B–$\mathbf{TiO_2}$ composite coatings the coated specimens were suitably placed in a Profilometer equipment, which is used to measure the surface roughness of the composite coating obtained. Figure 4 shows a graphical representation of the experimental data of the surface roughness of ENi–B–TiO_2 at 0, 5, 10 and 15 g/L concentration of TiO_2. TiO_2 nanoparticles of concentration 0–15 g are incorporated into the Ni–B coatings.

At lower concentration of TiO_2 nanoparticles, the chemical reaction occurs over the entire bulk of the solutions rather than a controlled autocatalytic reaction across the specimen surface and it is noticed that the bath becomes unstable after an hour. In such conditions only a small fraction of nanoparticles is deposited onto the substrate. This explains the enhancement in the surface roughness of the electroless nickel coating. The agglomeration of TiO_2 particles at higher concentration is the reason for increase in surface roughness [14, 15].

However, there is no effect of agitation speed on the surface roughness of the coatings because in all the bath composition parameters of agitation are the same. The average surface roughness obtained for ENi–B–TiO_2 at different concentrations of TiO_2 is shown in table 2.

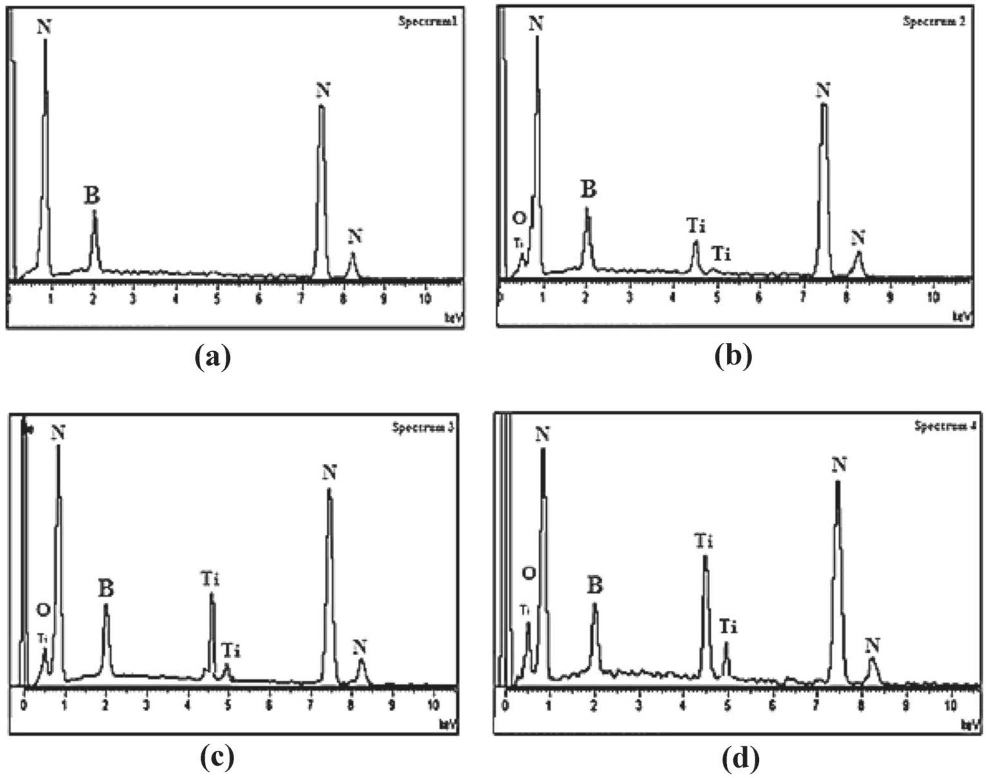

Figure 4. EDX plots for ENi–B–TiO_2 composite coatings at 100 rpm: (**a**) 0 g/L, (**b**) 5 g/L, (**c**) 10 g/L and (**d**) 15 g/L TiO_2 in the bath.

Table 2. Average roughness value of AZ91 without and with ENi–B–TiO_2 composite coatings at 100 rpm agitation.

Parameter	AZ91 without coating	AZ91 with ENi–B–TiO_2 coating at different concentrations of TiO_2			
		0 g/L	5 g/L	10 g/L	15 g/L
Roughness R_a (μm)	0.42	0.48	0.54	0.79	1.07

5. Conclusion

In the present work, the effect of coating process parameters on the surface roughness of ENi–B–TiO_2 composite coatings has been investigated. The investigation shows that the incorporation of $\mathbf{TiO_2}$ particles has greater impact on the surface roughness of the composite coatings. Increase in amount of these particles increases the roughness of the composite coatings. The optimum surface roughness of ENi–B–TiO_2 is obtained at 10 g/L titanium particles. There is no effect of agitation speed on the surface roughness of the coatings.

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