Feasibility Study of Nickel Based Coating on LM-25 Aluminum Alloy

Pooja Shah
M tech (Industrial Metallurgy)
Indus University, Rancharda
Ahmedabad – 382115, India

Vaibhav Bhavsar
Engineer, FCIPT Division
Institute for Plasma Research
Gandhinagar - 382016, India

Mrunalkumar Chaudhary
Professor, Metallurgy Department
Indus University, Rancharda
Ahmedabad – 382115, India

Nirav Jamnapara
Engineer-SF, FCIPT Division
Institute for Plasma Research
Gandhinagar - 382016, India

***Abstract* - High speed winding drums are used for winding cotton or cotton/synthetic blend yarn. Commercially, different types of winding drums are available such as Aluminum hard anodized drums, Bakelite drums and Steel drums due to their superior wear resistance. Steel drums are widely used in textile industries. However, it consequently results in more power consumption owing to its high inertia. In this article, a feasibility study has been conducted to evaluate the potential application of LM-25 alloy. The LM-25 alloy has relatively high strength to weight ratio and low coefficient of thermal expansion vis-à-vis pure aluminum. However, LM-25 alloy does not have the adequate wear resistance, which is a prerequisite for winding drums. Therefore, Nickel coating was applied on LM-25 via Electroless coating method to improve its wear resistance. Subsequent to the Ni coating on LM-25 alloy, the sample was heat treated to various conditions. The purpose of heat treatment of Ni coating on LM-25 alloy is to precipitate hard intermetallic phase (Ni_3Al), which is known to improve the hardness of the coating. The as-prepared coated samples and heat treated samples were characterized by X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy-Dispersive X-ray Spectroscopy (EDS) and micro-hardness measurement.**

Keywords- LM-25 Alloy, Electroless Coating, Heat Treatment, Coefficient of Thermal Expansion, Adhesive Strength

1. INTRODUCTION

Aluminum and its alloys are attractive for many application in Automobile, Marine, Rail, Textile and Aerospace industries because of their excellent properties such as high Specific Strength (i.e. strength to weight ratio), high electrical and thermal conductivities, and good formability along with good corrosion resistance. Although, their wear resistance is poor and coefficient of friction is relatively high as compared to wear resistant steel. In the last couple of decades, continuous efforts are being made in making use of lightweight aluminum in applications that were traditionally reserved for harder and more wear-resistant materials[1]. Quest for improved tribological performances led to many new developments and investigations in the area of surface coating processes such as electrolytic[1], electroless[2], physical vapor deposition (PVD)[3], chemical vapor deposition (CVD)[4] and laser beam coating[3]. In PVD, it is reported that high temperature magnetron deposition of nickel could only be achieved either by sputtering Ni-V target[5], which is non-magnetic or by heating Ni target above the curie temperature[6]. Recently, a method based on Ni-B electroless deposition, followed by diffusion heat treatment was proposed for improving wear resistance of Al-7075alloy[1]. The diffusion of nickel and boron present in the coating allowed a remarkable hardening of coating on the substrate[7]. Similar experiments were performed on magnesium[8] and copper[9] alloys as substrate. However, not much is reported on Ni-P electroless coating on LM-25 (Al-7.5% Si-0.6% Mg) alloy. This article is mainly focused on Ni-P electroless coating on LM-25 alloy. The as-deposited Ni-P coating[10] was heat treated to increase the hardness and wear resistance of coating. The LM-25 alloy was chosen as substrate material because it is lightweight (density of LM-25 alloy is about 1/3 that of steel or copper alloys)[11]. In addition, LM-25 alloy has excellent castability, good combination of high mechanical strength and high corrosion resistance[11], which is required for winding drum application. Superior wear and corrosion resistance is provided by electroless Ni coating on LM-25 alloy.

Electroless Ni-P (EN)[11] coating is a well-established surface engineering process and is widely used in automotive and aerospace industries. It provides high hardness[12] and excellent resistance to wear, abrasion and corrosion[13]. The automobile industry takes particular advantage of the uniformity of the electroless nickel deposit on irregular surfaces, direct deposition on surface activated non-conductors, and the formation of less porous, more corrosion resistant deposits[2].

The electroless Ni-P coating was deposited on samples made from LM-25 Al alloy and subsequently the as-coated samples were heat treated to explore its feasibility as an alternate material for winding drum. The resultant coating was analyzed using XRD, SEM, EDS and micro-hardness measurement.

2. EXPERIMENTAL WORK

2.1 Material

The aluminum-silicon-magnesium (LM-25) alloy is chosen as the substrate material. The composition of the alloy is given in Table 1. The chemical composition was determined by optical emission spectrometer.

Table 1 Chemical composition of LM-25 alloy.

Alloy	Si	Mg	Cu	Fe	Zn	Al
LM-25	7.5	0.6	0.2	0.5	0.1	Balance

2.2 Sample preparation

Sample of 26 mm diameter and 2 mm thickness of LM-25 material was used for the experimental work. Foregoing to deposition of electroless Ni-P coating, each sample was ground using emery paper of various grit sizes (240 to 1200 grit size papers), followed by polishing using a Disc Polisher with 0.05 micron diamond paste. Each sample was then etched by Keller's reagent (190 ml Distill water, 5 ml HNO_3, 3 ml HCl and 2 ml HF). Electroless coating of Ni-P was deposited on sample at APEX Industries, Gandhinagar, Gujarat, India.

2.3 Preparation of Ni-P coating on LM-25 samples

The Ni-P coating was produced by electroless deposition process. It is well-known that the property of Ni-P coating varies with the amount of P content. In general, Ni-P coating with low P (less than 5wt%) is used for wear resistance applications and those with high P content (i.e. greater than 15wt%) is used for high corrosion resistance. The winding drum used in textile industries requires high wear resistance. Hence, the electroless process was designed to produce Ni-P coating with low P content. To produce Ni-P coating with low P content, the composition of the bath for electroless deposition process was optimized. The bath comprises of nickel chloride as the source of nickel, sodium hypophosphite as the reducing agent, and sodium citrate as the complexing agent. Temperature of the bath was maintained at 85 ± 1 °C during the deposition of Ni-P coating. The pH of the bath was maintained between 9 and 10 by addition of sufficient quantity of ammonia solution[14].

2.4 Heat Treatment

Prior to heat treatment, samples were ultrasonically cleaned with acetone for 10 minutes. Heat treatment was performed at 500 °C for 24 h of soaking time, followed by furnace cooling. System parameters were set at – 500V with duty cycle 33% min. The working pressure of Argon was set at 3 mbar. The actual temperature in the furnace would be around 550°C due to the argon bombardment. The as-coated samples were heat treated to precipitate NiP and Ni_2Al_3 phases in the coating (shown in Figure 1), which consequently increased its hardness.

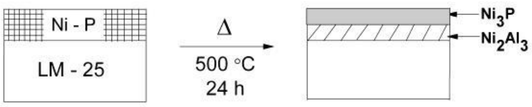


Fig. 1: Mechanism of phase formation after heat treatment

3. RESULTS AND DISCUSSION

Ni-P coating was deposited on LM-25 samples by electroless deposition process. The process for Ni-P coating is discussed in brief in section 2.3. As-coated and heat treated samples were characterized by XRD, SEM and Hardness tester. Results are discussed below.

3.1 Surface morphology

Microscopic examination on various samples were performed. Figure 2(A) shows the surface morphology of the electroless nickel deposited surface having smooth surface. The round particle like features seen on the image is the area with additional deposition of Ni-P coating. This feature is common in electroless Ni-P deposition on metal substrate. Further, it was observed that the thickness of the coating on both sides of the substrate was uniform. The appearance of the deposits was homogeneous.

The heat treatment of as-coated sample resulted in flaking of Ni-P coating. This may be due to the formation of intermetallics between aluminum and Ni-P coating. These intermetallics are formed mainly due to inward diffusion of nickel into Al-rich phase (Al_3Ni_2). For higher diffusion of nickel in LM-25 substrate, higher temperature is required but it is in contradiction to the temperature required for Ni-P coating formation. The volume fraction of intermetallic phase(s) increases with increasing temperature and time and consequently results in generation of non-uniform residual stresses during the heat treatment. The Ni-P coating flakes off when the magnitude of residual stress exceeds interfacial bond strength. The resultant image of coating is shown in the Figure 2(B). After peel off, the coating thickness was measured and noted to be of 20µm by SEM analysis.

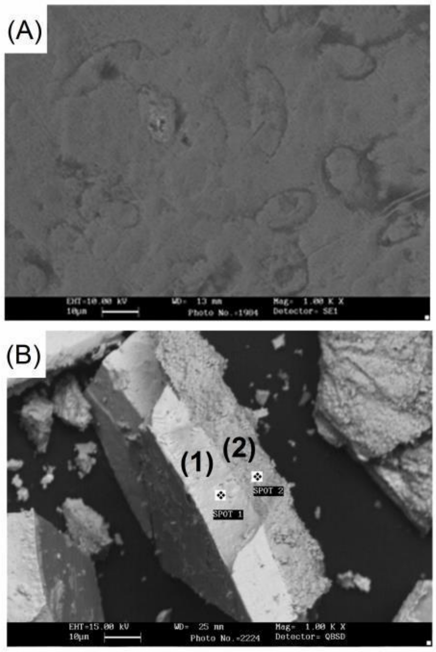
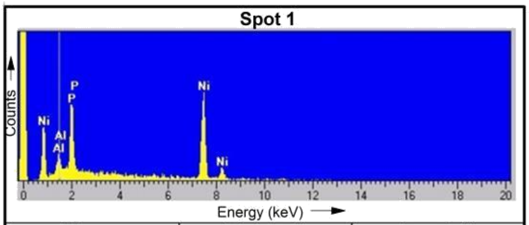


Fig. 2: SEM image of (A) Surface of Electroless Ni-P coated on LM-25 surface, (B) Flaked off Ni-P coating after heat treatment.

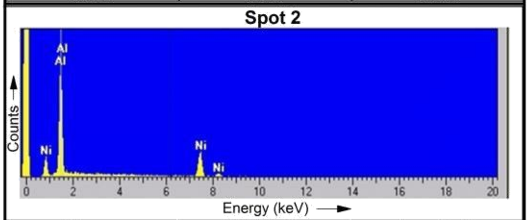
3.2 EDS analysis

Two different spots on the coating flake were analyzed as shown in Figure 3. EDS analysis confirmed the presence of P at spot 1 of 2.02 ± 0.1 wt.% with nickel and aluminum being balance amount. Since no significant oxygen or other element is found, therefore it is reasonable to believe that the coating consists of Ni and P with no contribution from other elements.

The EDS analysis at spot 2 confirmed that underneath the Ni-P coating and above LM-25 Alloy substrate, the region is enriched with Ni. This confirms that during heat treatment, Ni diffused into the Al matrix and resulted in the formation of Al-Ni intermetallic phase.



Element	Weight %	Atomic %
Al K	0.54	6.06
P K	2.02	19.68
Ni K	14.47	74.27



Element	Weight %	Atomic %
Al K	41.23	60.42
Ni K	58.77	39.58

Fig. 3: EDS spectra of Spot-1 and Spot-2 as shown in Figure 2(B).

3.3 XRD analysis

The XRD measurements were performed with Cu-Kα radiation (λ = 1.5406 Å), 40 kV, 40 mA with a step size of 0.05° in Bragg-Brentano mode. The 2θ scanning range was from 20° to 100°. The resultant scan data was analyzed using ICDD database.

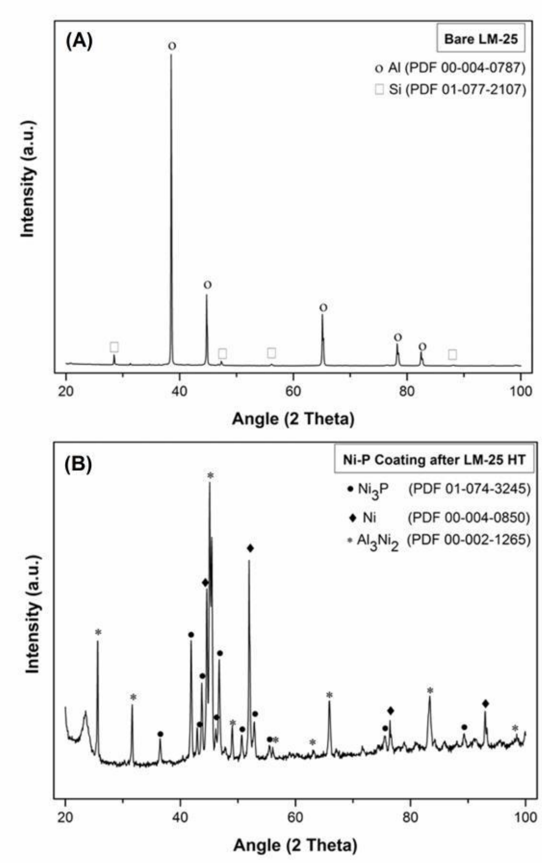


Fig. 4: X-ray diffractograms of (A) LM – 25 alloy and (B) Ni – P Coating after heat treatment.

Figure 4(A) shows the XRD graph for uncoated LM-25 sample. The strong diffraction peaks at 2θ = 38.5°, 44.7°, 65.1°, 78.3° and 82.4° corresponding to (111), (200), (220), (311) and (222) planes respectively of Al. This is in good agreement with the values of standard datacard (PDF 00-004-0787). Some weak peaks at 2θ = 28.5, 47.2 and 56.1 corresponding to (111), (220) and (311) planes respectively of eutectic Si phase were observed as per the standard datacard (PDF 01-077-2107). The XRD graph of Ni-P coating after heat treatment is shown in Figure 4(B). It clearly reveals the formation of Ni_3P and Al_3Ni_2 phases as per the standard datacard (PDF 01-074-3245) and (PDF 00-002-1265), respectively. These two phases are consistent to the observation made from the SEM data (discussed in previous section).

3.4 Hardness measurement

Micro-hardness tests were carried out at various locations (from center to edge of the sample) using a Vickers indenter at 10 grams load. An average of 5 measurements is reported in this paper. Hardness of uncoated LM-25 alloy is 71.76 HV and the hardness of electroless Ni-P coating is 629.40 HV in the as-coated condition. No hardness measurement was performed in heat treated Ni-P coated samples since Ni-P coating flaked off after heat treatment.

4. CONCLUSION

- The Ni-P coating was successfully deposited on LM-25 sample by electro deposition technique. The thickness of Ni-P coating was 20µm and it was uniform throughout the sample.
- XRD analysis confirmed the formation of Al-Ni based intermetallic phase (Ni_2Al_3) in the coated layer during heat treatment of Ni-P coated sample. In addition, the coated layer also consisted of NiP precipitate in heat treated sample.
- Hypothesis to explain the source for flaking of coating is proposed below:

(1) Generation of residual stresses caused by the mismatch of coefficient of thermal expansion between the coating and substrate material due to heat treatment. The coefficient of thermal expansion of LM-25 Alloy is 22µm/m/˚K and that of Ni-P Coating is 11µm/m/˚K.

Therefore, the heat treatment process needs to be optimized by reducing the cooling rate after heat treatment and obtaining the volume fraction of intermetallic phases to an optimum level so that flaking of Ni-P coating is prevented after heat treatment. This peeling off can be prevented by forming strong metallurgical bond between Ni-Al coating and LM-25 substrate.

(2) Improper adhesion of Ni-P on the LM-25 could be due to the presence of aluminum oxide (Al_2O_3) layer on substrate surface. It is well-known that diffusion of Ni through Al_2O_3 is relatively slow and consequently, Ni-rich intermetallic phase (i.e. Ni_3Al) did not form. The formation of Ni_3Al is very much essential to ensure strong interfacial bonding between the coating and substrate. Therefore, it is recommended to ensure that aluminum oxide layer is

properly removed from the surface of the substrate prior to the deposition of Ni-P coating. Further work is required to prove the hypothesis and improve the process.

ACKNOWLEDGMENT

We hereby express our sincere thanks to Mr. Sidhharth Kinariwala (Director, NPK Winding Drums & Cylindrical Cams) for the procurement of LM-25 substrates, Mr. Dinesh Patel (APEX Industries) for carrying out electroless treatment of the substrates, Dr. Sujoy Krishna Chaudhary (HoD, Metallurgy Dept., Indus University) and Hiren Patel (Scientific Assistant, FCIPT Division, Institute for plasma research) for their advice and useful discussion during the project. We also acknowledged HT-XRD lab, FCIPT-IPR Gandhinagar for conducting XRD.

REFERENCES

[1] K. Brunelli, M. Dabalà, and C. Martini, ―Surface hardening of Al 7075 alloy by diffusion treatments of electrolytic Ni coatings,‖ *la Metall. Ital.*, no. 7, 2013.

[2] R. Rajendran, W. Sha, and R. Elansezhian, ―Abrasive wear resistance of electroless Ni--P coated aluminium after post treatment,‖ *Surf. Coatings Technol.*, vol. 205, no. 3, pp. 766–772, 2010.

[3] P. J. Kelly and R. D. Arnell, ―Magnetron sputtering: a review of recent developments and applications,‖ *Vacuum*, vol. 56, no. 3, pp. 159–172, 2000.

[4] W.-P. Sun, H. J. Lin, and M.-H. Hon, ―CVD aluminide nickel,‖ *Metall. Trans. A*, vol. 17, no. 2, pp. 215–220, 1986.

[5] S. A. Chang, M. B. Skolnik, and C. Altman, ―High rate sputtering deposition of nickel using dc magnetron mode,‖ *J. Vac. Sci. Technol. A Vacuum, Surfaces, Film.*, vol. 4, no. 1986, p. 413, 1986.

[6] A. Caillard, M. El Mokh, N. Semmar, R. Dussart, T. Lecas, and A.-L. Thomann, ―Energy Transferred From a Hot Nickel Target During Magnetron Sputtering,‖ *Plasma Sci. IEEE Trans.*, vol. 42, no. 10, pp. 2802– 2803, 2014.

[7] G. O. Mallory and J. B. Hajdu, *Electroless plating: fundamentals and applications*. William Andrew, 1990.

[8] J. E. Gray and B. Luan, ―Protective coatings on magnesium and its alloys—a critical review,‖ *J. Alloys Compd.*, vol. 336, no. 1, pp. 88–113, 2002.

[9] W. Sha, X. Wu, and K. G. Keong, *Electroless copper and nickel-phosphorus plating: processing, characterisation and modelling*. Elsevier, 2011.

[10] ―Properties of Electroless Nickel at Elevated Temperature-a Review,‖ *Procedia Eng.*, vol. 97, pp. 1698–1706, 2014.

[11] A. Jayakumar and M. Rangaraj, ―Property Analysis of Aluminium (LM-25) Metal Matrix Composite,‖ *Int. J. Emerg. Technol. Adv. Eng.*, vol. 4, no. 2, pp. 495– 501, 2014.

[12] K. G. Keong, W. Sha, and S. Malinov, ―Hardness evolution of electroless nickel--phosphorus deposits with thermal processing,‖ *Surf. Coatings Technol.*, vol. 168, no. 2, pp. 263–274, 2003.

[13] P. Sahoo and S. K. Das, ―Tribology of electroless nickel coatings--a review,‖ *Mater. Des.*, vol. 32, no. 4, pp. 1760–1775, 2011.