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Influence of Heat Treatment on Crystal Structure, Microhardness and Corrosion Resistance of Hybrid Electroless Ni-P-Al_2O_3-Mo_2C Coating on 90wt% Al-10wt% Mg alloy

Shetha Daniel[a,*], Nilotpal Banerjee[a], Manik Majumder[a], and Sudhir C. V.[b]

[a] Department of Mechanical Engineering, National Institute of Technology, Durgapur 713209, India

[b] Department of Mechanical & Industrial Engineering, College of Engineering, National University of Science & Technology, P.O.Box : 2322, CPO Seeb 111, Al Hail, Sultanate of Oman

Abstract

The aim of this study was to investigate the properties of an electroless Ni-P-Al_2O_3-Mo_2C coating on a 90wt% Al-10wt% Mg alloy. A thermal process was conducted at 400 °C for 1 hour to assess the impact of temperature on the coating properties. The structure of the bilayer coatings was analyzed using X-ray diffraction (XRD), and the surface topography of the hybrid coating was observed using a scanning electron microscope. It was found that at a concentration of 2 g/L, the Al_2O_3 and Mo_2C distributions in the Ni-P coating bath exhibited a more uniform nodular or columnar structure. The hardness of the deposit was measured using a Vickers hardness tester, and the Ni-P-Al_2O_3-Mo2C coating sintered with a nanoparticle concentration of 2 g/L showed the highest hardness value of 692 VHN. This value was 19.32% higher than the unsintered Ni-P-Al_2O_3-Mo_2C external layered coating. Furthermore, potentiodynamic polarization studies were performed in a 3.5% NaCl solution to evaluate the corrosion behavior of the hybrid coating under ambient conditions. The results indicated that the Ni-P-Al_2O_3-Mo_2C coating formed at 2 g/L provided the highest corrosion protection, as confirmed by the highest positive E_{corr} value of -0.2.4 V, attributed to the maximum particle deposition rate. Overall, the combination of NiP_3, Al_2O_3, and Mo_2C phases in the coating exhibited synergistic effects, leading to improved anti-corrosion and mechanical properties.

Keywords: Electroless Ni-P coating; Heat treatment; Microhardness; Corrosion resistance; Hybrid coatings

1. Introduction

The development of surface engineering techniques has resulted in significant advancements in improving the mechanical, tribological, and corrosion resistance properties of materials [1-3]. One technique that has gained considerable attention is the application of electroless coatings [4, 5]. This method stands out due to its simplicity, cost-effectiveness, and ability to enhance the surface properties of a wide range of substrates. Electroless coating has revolutionized the world of surface finishing, finding applications in various industries such as automotive and electronics. It has become the preferred method for enhancing the durability and aesthetics of different materials. Among the different types of electroless coatings, Ni-P coatings stand out for their ability to provide higher hardness and resistance to corrosion and wear [5-7]. These coatings are fabricated through the reduction of nickel ions from a metal source in the presence of a hypophosphite reducing agent. Extensive studies have confirmed that Ni-P coatings significantly enhance the hardness and resistance of alloys to corrosion.

To enhance the properties of electroless Ni-P coatings, the addition of suitable additives in the plating bath can be employed. These additives are used to improve specific characteristics of the Ni-P coating. For instance, the incorporation of stabilizers, wetting agents, or brighteners can enhance corrosion resistance, adhesion, or appearance of the coating [6, 8-10]. Recent studies have also demonstrated that the

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*Corresponding author e-mail: shethamanu80@gmail.com.; (Shetha Daniel).

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inclusion of nano particles in the plating bath can further enhance the performance of electroless Ni-P coatings [6, 11, 12]. The electroless Ni-P coating offers numerous advantages; however, it does have a few drawbacks. One notable disadvantage is the formation of hydrogen bubbles during the deposition process, which can result in the development of pores in the coating structure. This compromises its protective capabilities. To overcome this challenge, innovative strategies such as duplex or hybrid coating (integration of two different coating systems for enhanced performance) and composite coating have been devised to bolster the effectiveness of electroless Ni-P coating [13-16]. For an instance Zhang et al. conducted a study on the microstructure and properties of duplex Ni-P-TiO_2/Ni-P nanocomposite coatings. These coatings were applied onto brass substrates through electrodeposition [17]. The purpose of the inner layer of high phosphorus Ni-P coatings was to enhance corrosion resistance. On top of that, low phosphorus sol-enhanced Ni-P-TiO_2 coatings were deposited to improve mechanical strength. In another study by Sudhakar Uppada et al., the researchers investigated the effect of heat treatment on the crystal structure, microhardness, and corrosion resistance of bilayer electroless Ni-P-SiC/Ni-P-Al_2O_3 coatings [18]. Duplex Ni-P-Al_2O_3/Ni-P-SiC coatings were developed on mild steel using the electroless method. Both duplex coatings exhibited a uniform and dense morphology. The electrochemical analysis revealed that the Ni-P-Al_2O_3 outer layered duplex coatings provided superior corrosion protection compared to the Ni-P-SiC external layered coatings. This was attributed to the higher electrochemical resistivity of the aluminum particles in the Ni-P-Al_2O_3 outer layered bilayer coatings. Furthermore, the annealing process resulted in the formation of a dense surface structure, significantly enhancing the corrosion resistance of both duplex coatings in a 3.5% NaCl solution.

Recently, we explored the use of a high Mg content Al alloy and successfully produced a new 90wt% Al-10wt% Mg alloy through die-casting method [19]. Further, to enhance the Ni-P coating on this alloy, we incorporated nano Al_2O_3 into the Ni-P bath. The results showed that the Ni-P-5wt% nano-Al_2O_3 composite coatings on the 90wt% Al-10wt% Mg alloy exhibited an impressive hardness value of 598 HV. In order to further enhance the properties of this alloy, our current work aims to comprehensively analyze the impact of heat treatment on the crystal structure, microhardness, and corrosion resistance of Hybrid electroless Ni-P-Al_2O_3-Mo_2C coatings. To achieve this objective, a series of controlled experiments were conducted and the samples characterized using advanced techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), microhardness testing, and electrochemical corrosion resistance measurements. The findings from this research will contribute to a better understanding of the effects of heat treatment on the properties of electroless coatings. This knowledge will be valuable for optimizing and developing high-performance coatings in various industrial applications, including automotive, aerospace, and chemical processing industries.

2. Experimental

The substrate material employed in this study was a 90wt% Al-10wt% Mg alloy with dimensions measuring 20 mm × 20 mm × 2.5 mm, as detailed in our earlier investigation [19]. Further, the substrate underwent mechanical polishing using SiC papers of different grades to achieve a smooth surface. Acetone and deionized water were then used to clean the substrate, removing any oil and dirt. The adhesion of the deposit was enhanced by subjecting the substrate to a 10% HCl solution for 60 seconds. The chemical bath method outlined in Table 1 was employed to create the hybrid coating. To form the Ni-P-Al_2O_3-Mo_2C outer layer coating, the treated substrate was immersed in an Al_2O_3 and Mo_2C nanoparticle solution for 90 minutes, resulting in the deposition of the Ni-P-Al_2O_3-Mo_2C layer coating, the initially polished surface was immersed in an Al_2O_3 chemical solution with the assistance of ultrasonic agitation, ensuring even distribution of the nanoparticles. The Al_2O_3 and Mo_2C nanoparticles were prepared through mechanical ball milling upto 30 h to get nano-particles in the range of ~40-60 nm. During the deposition process, the coating solution was maintained at a constant temperature (88 °C (± 2)) using an oil bath and hotplate. The temperature was monitored using a thermocouple-attached PID controller. The entire process was performed using a fixed volume of 150 ml, and the pH of the solution was measured using a pen-type pH meter. To assess the impact of temperature on the coating properties, a thermal process was carried out at 400 °C for 1 hour without the use of an atmospheric controlled furnace. Figure 1 shows coating process used in this study.

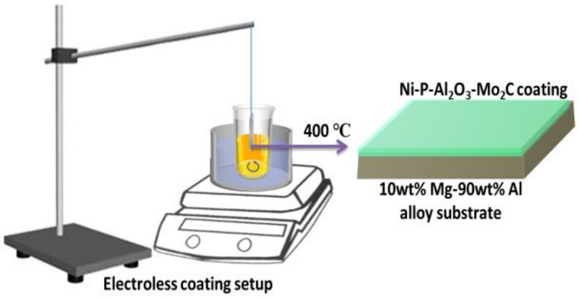

Fig. 1. Experimental setup of Hybrid electroless Ni-P-Al_2O_3-Mo_2C coating on 90wt% Al-10wt% Mg alloy

The structure of the bilayer coatings was analyzed using X-ray diffraction (XRD). Additionally, a scanning electron microscope was used to observe the surface topography of hybrid coating. The hardness of the deposit was measured using a Vickers hardness tester with a load of 100 g and a dwell time of 10 s. The microhardness of the hybrid coating was represented by the average of five readings. To evaluate the corrosion behavior of the coating under ambient conditions, potentiodynamic polarization studies were conducted in a 3.5% NaCl solution. The CH Instruments, CHI608E electrochemical system was employed for these studies.

Table 1
Coating bath composition for hybrid electroless Ni-P-Al_2O_3-Mo_2C coating

Composition	**Concentration (g/L)**
Nickel-chloride	40
Sodium-hypophosphite	20
Tri sodium-citrate	25
Ammonia-chloride	50
CTAB	0.8
Al_2O_3 and Mo_2C nanoparticles (Average size ~40-60 nm)	1, 2, and 3
pH	4.5 to 5.5
Temperature	88 °C (± 2)

3. Results and discussion

The XRD profile of the as-prepared 90wt% Al-10wt% Mg alloy substrate and the substrate coated with electroless Ni-P-Al_2O_3-Mo_2C composite is depicted in Figure 2. From the figure, it is evident that the XRD profile peaks of the bare substrate align closely with Al and Mg, as previously reported in our earlier study [19]. Upon coating the substrate with electroless Ni-P-Al_2O_3-Mo_2C and subjecting it to a heat treatment at 400 °C, additional crystalline peaks emerge in the XRD profile. Following the Ni-P-Al_2O_3-Mo_2C coating, the XRD peaks can be attributed to the NiP_3 (ICSD PDF Ref no 98-000-7968), Al_2O_3 (ICSD PDF Ref no 98-003-2923), and Mo_2C (ICSD PDF Ref no 98-000-0693) phases. The NiP_3, Al_2O_3, and Mo_2C phases presented in the Ni-P-Al_2O_3-Mo_2C coating improve its anti-corrosion and mechanical properties through various mechanisms. The NiP_3 phase acts as a corrosion inhibitor, forming a protective layer on the surface that prevents the penetration of corrosive agents [20]. It also ensures strong adhesion to the substrate, providing long-term corrosion protection. The Al_2O_3 phase acts as a barrier against corrosive species, creating a dense and impermeable layer that effectively blocks the diffusion of corrosive agents [21]. This enhances the coating's resistance to corrosion, especially in harsh environments. The Mo_2C phase contributes to the coating's mechanical properties, offering excellent hardness and wear resistance, which protects against abrasive wear and erosion [22]. It also improves the coating's ability to withstand mechanical stresses and extends its service life. The combination of NiP_3, Al_2O_3, and Mo_2C phases in the coating produces synergistic effects, enhancing both anti-corrosion and mechanical properties [6, 23]. These phases work together, leveraging each other's strengths and minimizing individual weaknesses. This synergistic effect results in a highly effective coating system that demonstrates superior performance in corrosion resistance and mechanical durability.

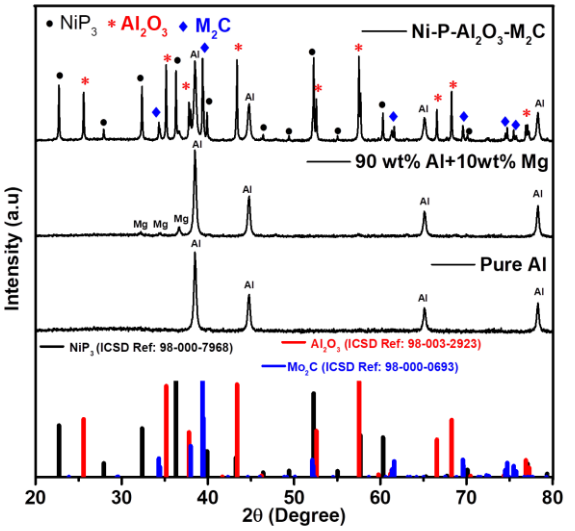

Fig. 2. XRD profile of electroless Ni-P-Al_2O_3-Mo_2C hybrid coating on 90wt% Al-10wt% Mg alloy

Surface morphology changes of an Al-Mg alloy before and after a coating process were analyzed using SEM images (Figure 3). The SEM image in Figure 3(a) displays a 90wt% Al-10wt% Mg alloy produced through die-casting, with irregularly shaped particles and sharp edges [19]. The as-cast surface appeared smooth but rough, with the presence of voids due to air entrapment during preparation and the hindrance of metal diffusion by the nano-ceramic phase. This resulted in the incorporation of Mg atoms into the Al matrix, creating a brittle material. Figure 3(b) shows the surface morphologies of the Ni-P-Al_2O_3-Mo_2C external hybrid coatings annealed at 400 °C. The type of nanoparticle used for reinforcement significantly affected the surface morphology. At a concentration of 2 g/L, the Al_2O_3 and Mo_2C distributions in the coating were more uniform, exhibiting a nodular or columnar structure. This structure enhanced the adhesion of the coating to the substrate and overall coating uniformity. A fine-grained microstructure within the coating was desirable for improved microhardness, corrosion resistance, and mechanical properties such as hardness and resistance to wear and deformation [24, 25]. Furthermore, the fine-grained structure reduced diffusion paths for corrosive species, contributing to better corrosion performance. However, some particles were unevenly dispersed and tended to cluster in specific regions. To determine the thickness, a metallurgical microscope was employed. The Leica microscope measured the coating thickness to be approximately 30 µm.

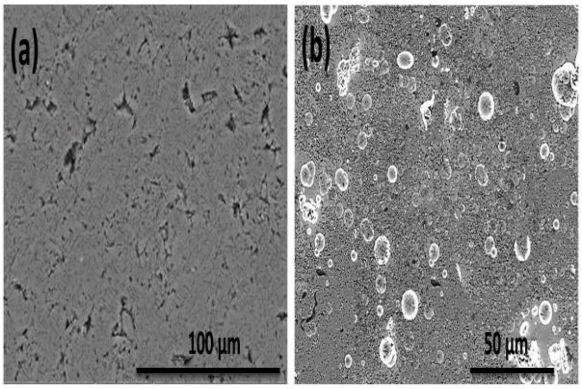

Fig. 3. SEM images of (a) 90wt% Al-10wt% Mg alloy (b) 90wt% Al-10wt% Mg alloy coated with hybrid electroless Ni-P-Al_2O_3-Mo_2C coating

The microhardness of the hybrid coatings made from Ni-P-Al_2O_3-Mo_2C was investigated by altering the concentrations of Al_2O_3 and Mo_2C particles. The findings showed that the microhardness was significantly influenced by the changes in particle concentration. Figure 4 and Table 2 presents the microhardness of the coatings produced with varying amounts of Al_2O_3 and Mo_2C particles. The hardness values of the Ni-P-Al_2O_3-Mo_2C coating were enhanced by increasing the nanoparticle concentration from 1 to 2 g/L, both before and after sintering at 400 °C (Table 2). This enhancement in hardness was attributed to the creation of hard intermetallic nickel phosphide compounds during annealing at temperatures ranging from 350 to 450 °C [26]. A higher particle concentration in the coating solution resulted in a greater number of particles in the coating. At a particle concentration of 2 g/L, the maximum number of particles was uniformly distributed in the Ni-P-Al_2O_3-Mo_2C coating, preventing plastic deformation in the alloy matrix. As a result, the hybrid coating prepared with a particle concentration of 2 g/L exhibited the highest microhardness. Conversely, at a concentration of 3 g/L, particle agglomeration occurred in the coating bath, leading to a lower number of particles with nonhomogeneous distribution in the deposit. This agglomeration negatively impacted the microhardness, causing the coatings prepared with a concentration of 3 g/L nanoparticles to have reduced microhardness values compared to other samples.

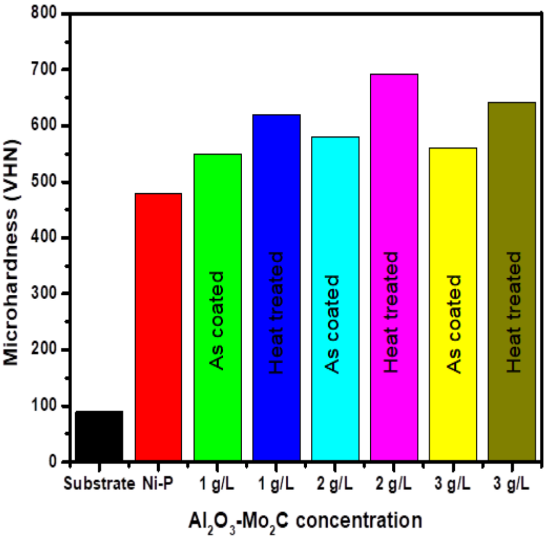

Fig. 4. Microhardness of the Hybrid Ni-P-Al_2O_3-Mo_2C coating before and after heat treatment process at different concentration of Al_2O_3 and Mo_2C on 90wt% Al-10wt% Mg alloy

The Ni-P-Al_2O_3-Mo_2C coating sintered after being fabricated with a nanoparticle concentration of 2 g/L achieved the maximum hardness value of 692 VHN, which was 19.32% higher than the unsintered Ni-P-Al_2O_3-Mo_2C external layered coating. Increasing the particle concentration from 1 to 2 g/L accelerated the deposition rate of Mo_2C and Al_2O_3 particles in the coatings. Additionally, increasing the concentration of Al_2O_3 and SiC particles from 1 to 2 g/L resulted in higher Al and Mo concentrations in the coating. At a particle concentration of 2 g/L, the low phosphorus concentration minimized the amorphous phase and improved the crystalline phase, leading to maximum microhardness. On the other hand, the non-uniform distribution of secondary particles at a particle concentration of 3 g/L decreased the microhardness of the coating, even with the lowest phosphorus content. The formation of phosphide acted as a barrier to dislocation movement in the nickel matrix, resulting in increased hardness due to the improved resistance to plastic deformation of the coating under loading.

Hybrid Ni-P-Al_2O_3-Mo_2C coatings were created via an eco-friendly electroless technique on an Al-Mg alloy. Potentiodynamic polarization studies were conducted to examine the electrochemical properties of the coatings, including corrosion current density (icorr) and corrosion potential (E_{corr}). Table 2 provides the results of the electrochemical analysis for the Ni-P-Al_2O_3-Mo_2C coatings, considering both plated and annealed coatings.

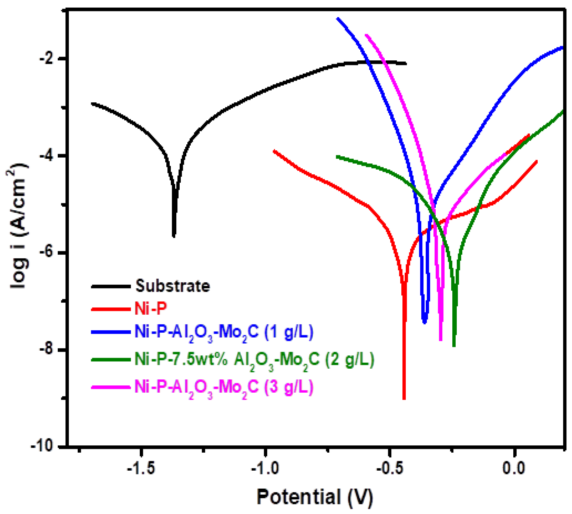

Fig. 5. Polarization curves of the 90wt% Al-10wt% Mg alloy substrate coated with electroless Ni-P and the Ni-P-Al_2O-Mo_2C hybrid composite coatings after heat treating

Figure 5 illustrates the polarization behavior of the Ni-P-Al_2O_3-Mo_2C external layered deposit. The corrosion resistance of the coatings was influenced by the particle concentration within them. Increasing the particle concentration from 1 to 2 g/L in the coating solution improved the corrosion resistance, as indicated by the positive shift of E_{corr} from -0.36 to -0.24 mV. Higher particle concentration also enhanced the rate of particle deposition in the coating, reducing the potential for galvanic corrosion. The highest positive E_{corr} value of -0.2.4 V confirmed that the Ni-P-Al_2O_3-Mo_2C coating formed at 2 g/L provided the highest corrosion protection due to the maximum particle deposition rate. However, at a concentration of 3 g/L, insufficient particle reinforcement in the coating resulted in surface pores and poor corrosion protection.

The improvement in corrosion resistance after the heat-treatment process was primarily attributed to the crystallization of the coating [26, 27]. The shift in E_{corr} value towards the noble direction for the hybrid coating formed at all particle concentrations indicated superior corrosion resistance compared to the as-plated samples. The increased phosphorus content led to a phase transformation from amorphous to crystalline Ni_3P, which played a significant role in enhancing the corrosion resistance of the coatings.

Table 2
Experimental parameters of 90wt% Al-10wt% Mg alloy substrate, the Ni-P coating, and the Hybrid Ni-P-Al_2O_3-Mo_2C coatings at different concentration.

Coating	Micro hardness	i_{corr} (A/cm^2)	E_{corr} (V)
Substrate	90	1.5×10^{-4}	-1.36
Ni-P coating	480	1.8×10^{-6}	-0.44
Ni-P-Al_2O_3-Mo_2C coating @ 1g/L	550 (As coated)	6.2×10^{-7}	-0.36
	620 (Heat treated)		
Ni-P-Al_2O_3-Mo_2C coating @ 2g/L	580 (As coated)	0.6×10^{-7}	-0.24
	692 (Heat treated)		
Ni-P-Al_2O_3-Mo_2C coating @ 3g/L	560 (As coated)	3.2×10^{-7}	-0.29
	642 (Heat treated)		

X-ray diffraction (XRD) results confirmed this phase transformation. Through Ni atom diffusion into the Al_2O_3-Mo_2C lattices, the formation of coherent boundaries between Al_2O_3-Mo_2C particles and the Ni-P lattice resulted in a dense and homogeneous Ni-P-Al_2O_3 external coating. Therefore, annealing proved beneficial in improving the corrosion protection ability of the coating. Moreover, the Ni-P-Al_2O_3-Mo_2C coating contains NiP_3, Al_2O_3, and Mo_2C phases, which enhance its anti-corrosion and mechanical properties. The NiP_3 phase acts as a corrosion inhibitor by forming a protective layer on the surface, preventing the penetration of corrosive agents. It also provides excellent adhesion to the substrate, ensuring long-term protection against corrosion. The Al_2O_3 phase acts as a barrier against corrosive species, forming a dense, impermeable layer on the coating surface [28]. This effectively blocks the diffusion of corrosive agents and enhances the coating's resistance to corrosion, even in harsh environments. The Mo_2C phase contributes to the coating's mechanical properties, offering high hardness and wear resistance. This provides enhanced protection against abrasive wear and erosion, as well as improved ability to withstand mechanical stresses, increasing its service life. The combination of NiP_3, Al_2O_3, and Mo_2C phases in the coating creates synergistic effects, where they work together to complement each other's strengths and minimize weaknesses. This results in a highly effective coating system that exhibits superior performance in both corrosion resistance and mechanical durability. Overall, the presence of NiP_3, Al_2O_3, and Mo_2C phases in Ni-P-Al_2O_3-Mo_2C coating provides a comprehensive approach to enhancing both anti-corrosion and mechanical properties. These phases offer corrosion inhibition, barrier effects, hardness, wear resistance, and synergistic effects, making the coating highly effective in protecting the substrate from corrosion and mechanical stress.

The hybrid electroless Ni-P-Al_2O_3-Mo_2C coating on 90wt% Al-10wt% Mg alloy draws its exceptional properties from the synergistic effects of its constituent phases, each contributing unique mechanical and anti-corrosion characteristics. The Ni-P (Nickel Phosphorus) phase, with phosphorus imbuing nickel with an amorphous structure, significantly enhances the toughness, hardness, and wear resistance of the alloy. The lack of crystalline grain boundaries in this amorphous matrix translates into superior corrosion resistance, as those boundaries are common initiation sites for corrosion. Al_2O_3 (Aluminum Oxide) delivers additional strength to the coating due to its inherent hardness and the ability to resist wear and thermal stress, further shoring up the mechanical robustness of the material. The chemical inertness of Al_2O_3 makes it an outstanding barrier against oxidative and corrosive forces, guarding the integrity of the underlying alloy. The third component, Mo_2C (Molybdenum Carbide), with its high hardness and melting point, integrates into the coating to fortify hardness and bolster the structure against high-temperature degradation. Moreover, Mo_2C enhances the coating's resilience against more localized forms of corrosion, such as pitting and crevice attack, by providing galvanic protection. When combined within a hybrid coating, these phases distribute optimally to create a ductile yet tough Ni-P matrix, which is both supported and reinforced by the hard Al_2O_3 particles and Mo_2C, resulting in a uniform structure with fewer defects and increased overall strength. The coating markedly benefits from the complimentary mechanical properties where the Ni-P phase mitigates stress around the stiffer Al_2O_3 particles, and Mo_2C acts as a bulwark against crack propagation and corrosion. Consequently, such an advanced material solution promisingly withstands both mechanical duress and aggressive corrosive environments, positioning it as an ideal candidate for utilization in cutting-edge sectors like aerospace and automotive engineering—

industries where the balance between high hardness and steadfast corrosion resistance is paramount. This fusion of insightful research and engineering finesse at the microscopic phase level holds the potential to revolutionize materials performance on a much larger scale.

4. Conclusion

This study explored the properties of an electroless Ni-P-Al_2O_3/Mo_2C coating on a 90wt% Al-10wt% Mg alloy. A thermal process at 400 °C for 1 hour was employed to investigate the influence of temperature on the coating properties. The analysis of the bilayer coatings using X-ray diffraction (XRD) revealed that a concentration of 2 g/L resulted in a more uniform distribution of Al_2O_3 and Mo_2C, exhibiting a nodular or columnar structure. The Vickers hardness test showed that the sintered Ni-P-Al_2O_3/Mo_2C coating with a nanoparticle concentration of 2 g/L achieved the highest hardness value of 692 VHN, representing a 19.32% improvement compared to the unsintered coating. The potentiodynamic polarization studies conducted in a 3.5% NaCl solution demonstrated that the Ni-P-Al_2O_3/Mo_2C coating formed at 2 g/L provided the highest corrosion protection, as evidenced by the highest positive E_{corr} value of -0.2.4 V. These results highlight the synergistic effects of the NiP_3, Al_2O_3, and Mo_2C phases in the coating, leading to enhanced anti-corrosion and mechanical properties. Overall, this research provides valuable insights into the potential application of electroless Ni-P-Al_2O_3/Mo_2C on Al-Mg alloys for improved performance and durability.

5. Author contributions

Shetha Daniel: Conceptualization, Methodology, Software, Field study, Data curation, Writing-Original draft preparation, Validation **Nilotpal Banerjee:** Super vision, Reviewing and Editing **Manik Majumder:** Reviewing and Editing **Sudhir C. V.:** Reviewing and Editing.

6. Conflicts of interest

The authors declare no conflicts of interest

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