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DESIGN AND DEVELOPMENT OF AN ELECTROLESS COATING SETUP AND INVESTIGATION OF THE MECHANICAL CORROSION AND HARDNESS CHARACTERISTICS OF Ni-P COMPOSITE COATINGS

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A B S T R A C T

$ZnFe_2O_4$, also known as zinc ferrite, is added to electroless nickel-phosphorus (Ni-P) coatings to improve the surface properties of engineering materials. Electroless Ni-P coating is a simple chemical coating method that does not need an external electric current and can form a uniform layer on the material surface. Adding $ZnFe_2O_4$ particles can improve the hardness, wear resistance, corrosion resistance, and overall durability of the coating. The quality and performance of the coating depend on factors such as the amount of $ZnFe_2O_4$, particle size, bath temperature, pH, plating time, and heat treatment. By properly controlling these parameters, a strong and uniform coating can be obtained. Heat treatment can further improve the hardness and structure of the coating. These coatings can be useful for protecting components used in automotive, aerospace, marine, oil and gas, and manufacturing industries. Overall, $ZnFe_2O_4$-reinforced Ni-P composite coating can be an effective method for improving the surface performance and service life of engineering components.

Keywords: $ZnFe_2O_4$, Zinc Ferrite, Electroless Ni-P, Composite Coating, Hardness, Corrosion Resistance, Wear Resistance, Surface Engineering.

1. INTRODUCTION

Surface coating is a process in which a thin layer of another material is applied to a component to protect its surface and improve its properties [3, 4]. It helps reduce corrosion, wear, friction, and damage caused by environmental conditions. Surface coating is widely used in automotive, aerospace, manufacturing, and construction industries to increase the life of components [6, 4]. Surface coating has been used since ancient times through paints, oils, and other protective materials. Modern metal-plating methods became popular during the 1800s. Later, in 1946, Abner Brenner and Grace E. Riddell developed electroless nickel plating as a practical coating process [15, 13]. The main purpose of surface coating is to improve the performance of machine components. Materials such as nickel, chromium, zinc, copper, ceramics, and polymers are commonly used for coating [5, 3]. Each material provides different benefits. For example, nickel improves hardness and corrosion resistance, zinc protects steel from rust, and ceramic coatings are useful in high-temperature applications. The coating thickness depends on the material and application. It may range from a few nanometres to several millimetres. Thin coatings are commonly used for surface protection, while thicker coatings are suitable for heavy wear protection and repairing damaged parts [7, 9]. Surface coating methods are mainly classified into three types based on the physical state of the coating material: gaseous state, molten or semi-molten state, and solution state coating [1, 10]. Gaseous State Coating: Gaseous state coating is a process in which coating materials reach the component surface as gases or vapours and form a protective layer [19]. Common methods include Chemical Vapour Deposition (CVD), Physical Vapour Deposition (PVD), and Ion Beam Assisted Deposition (IBAD). These methods are mainly used to produce thin coatings with good hardness, adhesion, and wear resistance [15, 11]. Chemical Vapour Deposition (CVD): CVD is a process in which chemical gases react on a heated component surface and form a solid coating [14]. Materials such as TiC, TiN, and SiC are commonly used. It provides good adhesion, hardness, and wear resistance. However, it requires high temperatures and special equipment [14, 12]. Physical Vapour Deposition (PVD): PVD is a coating process in which a solid material is converted into vapour inside a vacuum chamber. The vapour settles on the component surface and forms a thin coating [16, 19]. Materials such as TiN, TiC, chromium, and aluminium are commonly used. It provides good hardness and wear resistance, but the equipment is expensive [24, 29]. Solution State Coating: Solution state coating is a process in which a protective layer is formed using a chemical solution. One important method in this category is electroless deposition [8, 9]. Electroless Deposition: Electroless deposition is a chemical coating process in which a metal layer is deposited without using an external electric current. The component is immersed in a chemical bath containing metal salts, a reducing agent, and other required chemicals. A chemical reaction takes place and forms a coating on the surface [8, 9]. Common examples include Ni-P, Ni-B, and copper coatings [1, 7]. Electroless nickel plating was developed as a practical process by Abner Brenner and Grace E. Riddell in 1946. Electroless Ni-P coating generally operates at temperatures of around 80–90°C, depending on the bath conditions [10, 2]. This method produces uniform coatings, even on components with complex shapes. It is widely used in automotive parts, machine components, and electronic equipment because it improves

hardness, wear resistance, and corrosion protection [23]. However, temperature, pH, chemical concentration, and deposition time must be properly controlled to obtain good coating quality.

2. EXPERIMENTAL PROCEDURE:

Mild steel specimens of 20 × 20 mm² were used as the base material for depositing the Ni-P-$ZnFe_2O_4$ composite coating [16]. The specimens were polished using 100, 220, 320, and 420 grit SiC papers to remove scratches and surface irregularities [13, 18]. They were cleaned with deionized water and acetone to remove dust, oil, and grease, followed by treatment with 10% HCl for 2 minutes to remove surface oxides [4, 9]. The electroless Ni-P coating solution was prepared using nickel chloride, sodium hypophosphite, ammonium chloride, and trisodium citrate [29]. Nickel chloride provides nickel ions, while sodium hypophosphite acts as the reducing agent and phosphorus source. The bath temperature was maintained at 88 ± 2°C, and the pH was kept between 4.5 and 5.5 [14, 13]. $ZnFe_2O_4$ nanoparticles were added as reinforcement to improve the hardness and corrosion resistance of the coating [4, 7]. A high-power ultrasonicator was used to break particle clusters and distribute the nanoparticles evenly in the solution [11, 10]. SDS, CTAB, and PVP were used separately at 0.6 g/L to improve nanoparticle dispersion [25, 30]. Before composite coating, the specimens were immersed in the Ni-P bath without nanoparticles for 60 minutes to form an initial layer and improve bonding with the mild steel surface [11, 20]. After that, the specimens were transferred to the bath containing $ZnFe_2O_4$ nanoparticles and the selected surfactant for 120 minutes. During deposition, nickel and phosphorus formed the coating matrix, while $ZnFe_2O_4$ particles became incorporated into the growing layer [8, 11, 12]. The solution was heated using a hot plate and oil bath, while a PID controller and temperature sensor maintained the required temperature [7, 8, 11]. The bath pH was checked regularly, and approximately 150 mL of coating solution was used to maintain consistent experimental conditions [1, 3, 4, 10, 30]. After coating, the specimens were washed with deionized water and dried to remove remaining chemicals and loose particles [2, 3]. The coated specimens were then heat-treated at 400°C to study changes in their structure and properties. This treatment may promote nickel-phosphide formation and improve coating hardness [20, 11, 10]. Finally, the hardness was measured using a Vickers microhardness tester with a 100 g load for 10 seconds, taking five readings and calculating their average [11]. Corrosion resistance was evaluated using a potentiodynamic polarization test in 3.5% NaCl solution to study the protective performance of the composite coating [22, 20].

Table 2.3.1: Chemical Composition of Electroless Solution [14]

Coating Bath Elements	Concentration(g/L)
Nickel Chloride	40
Sodium Hypophosphite	20
Ammonia Chloride	50
Tri Sodium Citrate	25
SDS / CTAB / PVP	0.6
$ZnFe_2O_4$	1–3
Temperature	88 ± 2 °C
pH	4.5–5.5

3. RESULTS AND DISCUSSION

3.1 Surface Roughness: Surface roughness values of the electroless Ni-P-$ZnFe_2O_4$ composite coatings prepared with different zinc ferrite ($ZnFe_2O_4$) nanoparticle concentrations. Particle concentration in the coating bath significantly influences the surface characteristics and finish of the deposited coating [25, 10]. The average surface roughness (Ra) of the mild steel substrate is 2.53 µm. After electroless deposition of the pure Ni-P coating, the surface roughness decreases to 2.53 µm for the substrate and 2.53 µm for the 0 g/L reference only if separately measured; therefore, the measured reference value should be used to establish the actual improvement due to coating deposition [10]. For the $ZnFe_2O_4$-reinforced coatings, the average surface roughness decreases with increasing particle concentration from 1 to 2 g/L [11]. The coating prepared at 1 g/L concentration exhibits an average surface roughness of 1.67 µm, whereas the minimum roughness value of 1.08 µm is obtained at 2 g/L concentration. This indicates that the incorporation of $ZnFe_2O_4$ nanoparticles improves the surface finish of the coating up to an optimum particle concentration of 2 g/L [22, 23]. The reduction in surface roughness at 2 g/L concentration may be attributed to the uniform incorporation and distribution of $ZnFe_2O_4$ nanoparticles within the Ni-P coating matrix. The co-deposition of nanoparticles can influence the growth of the nickel matrix and restrict excessive nodular growth, resulting in a relatively smooth and uniform coating surface [25]. The presence of uniformly distributed reinforcement particles may also help to fill surface irregularities and improve the overall surface finish. However, a further increase in $ZnFe_2O_4$ particle concentration to 3 g/L increases the average surface roughness to 1.97 µm [10]. This increase may be attributed to particle agglomeration and non-uniform distribution of nanoparticles in the coating bath [14, 13]. At higher particle concentrations, agglomerated particles may become incorporated into the coating surface, resulting in local protrusions and surface irregularities. Consequently, the coating fabricated at 3 g/L exhibits a comparatively higher surface roughness than the coating prepared at 2 g/L concentration [16, 17]. Based on the surface roughness measurements, it can be concluded that the concentration of $ZnFe_2O_4$ nanoparticles significantly influences the surface finish of electroless Ni-P composite coatings [19, 11]. Among the investigated concentrations, 2 g/L provides the optimum particle concentration, resulting in the minimum average surface roughness of 1.08 µm [15]. The improvement in surface finish at this concentration can be associated with the favourable incorporation and distribution of $ZnFe_2O_4$ nanoparticles within the Ni-P matrix [13, 12]. Therefore, the Ni-P-$ZnFe_2O_4$ composite coating prepared at 2 g/L concentration exhibits the best surface finish among the investigated coatings [5].

3.2 Micro hardness: Microhardness of the electroless Ni-P-$ZnFe_2O_4$ composite coatings was evaluated by varying the concentration of zinc ferrite ($ZnFe_2O_4$) nanoparticles in the coating bath and compared with that of the mild steel substrate [5, 7]. The amount of secondary reinforcement particles incorporated into the Ni-P matrix significantly influences the microhardness of the composite coating [24, 15]. In the present investigation, Ni-P-$ZnFe_2O_4$ composite coatings were fabricated using three different particle concentrations of 1, 2 and 3 g/L. Figure X illustrates the variation in microhardness of the mild steel substrate, pure Ni-P coating and Ni-P-$ZnFe_2O_4$ composite coatings before and after heat treatment [14, 18]. The microhardness of the mild steel substrate is approximately 280 HV, whereas the pure Ni-P coating exhibits a hardness of 480 HV before heat treatment [10, 29]. All the Ni-P-$ZnFe_2O_4$ composite coatings demonstrate higher microhardness compared with the mild steel substrate [28, 15]. This indicates that the incorporation of $ZnFe_2O_4$ nanoparticles into the electroless Ni-P matrix improves the surface hardness of the mild steel substrate [14, 23]. The improvement in hardness can be attributed to the presence of the relatively hard nickel-based coating matrix and the reinforcing effect of the incorporated $ZnFe_2O_4$ nanoparticles. These particles can restrict the plastic deformation of the Ni-P matrix during indentation and thereby enhance the overall hardness of the composite coating [10, 8]. The microhardness of the Ni-P-$ZnFe_2O_4$ composite coatings increases with an increase in particle concentration from 1 to 2 g/L [25, 14]. Before heat treatment, the coating prepared at 1 g/L concentration exhibits a microhardness of 596 HV, whereas the coating fabricated at 2 g/L concentration achieves the maximum hardness of 635 HV. This improvement can be attributed to the increased incorporation of $ZnFe_2O_4$ nanoparticles into the Ni-P matrix [9, 8]. At an optimum concentration of 2 g/L, a more favourable distribution of reinforcement particles may provide effective resistance to indentation and plastic deformation of the coating matrix. However, a further increase in particle concentration to 3 g/L results in a reduction in microhardness to 537 HV [16, 20]. This reduction may be associated with particle agglomeration and non-uniform distribution of nanoparticles in the coating deposit [14, 19]. Such non-uniformity can create local defects and reduce the effective reinforcement of the Ni-P matrix. Therefore, the coating fabricated at 2 g/L $ZnFe_2O_4$ concentration exhibits the highest microhardness before heat treatment [11, 19]. Heat treatment significantly improves the microhardness of both pure Ni-P and Ni-P-$ZnFe_2O_4$ composite coatings [12, 19]. In the present investigation, the microhardness of the pure Ni-P coating increases from 480 to 608 HV after heat treatment [13, 19]. Similarly, the microhardness of the coatings prepared with 1, 2 and 3 g/L $ZnFe_2O_4$ concentrations increases from 596 to 732 HV, 635 to 892 HV and 537 to 683 HV, respectively [14, 17]. The improvement in microhardness after heat treatment can be attributed to the structural transformation of the initially amorphous Ni-P matrix into a crystalline structure [10, 9]. During heat treatment, precipitation of hard nickel phosphide (Ni_3P) phases takes place along with the formation of crystalline nickel. Microhardness of the coating before and after heat treatment process is shown in the Figure 3.2.1.

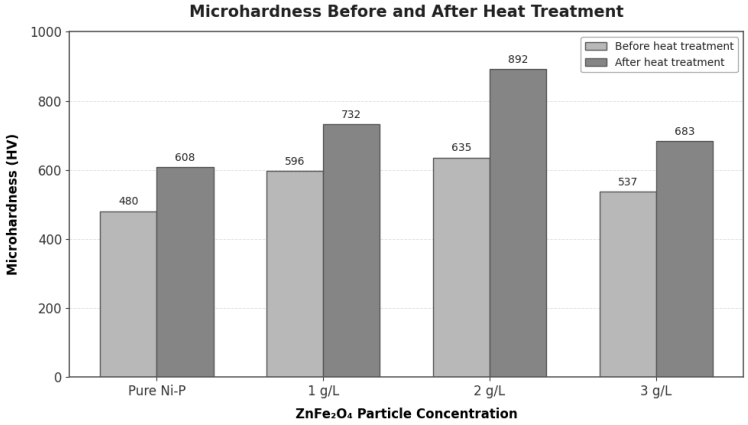


Fig 3.2.1: Micro hardness Before and After Heat Treatment [15]

Among all the investigated coatings, the Ni-P-$ZnFe_2O_4$ composite coating fabricated at 2 g/L concentration exhibits the maximum microhardness of 892 HV after heat treatment [12, 11]. This represents an improvement of approximately 40.5% compared with its corresponding hardness before heat treatment [12, 15]. The maximum hardness at this concentration may be attributed to the combined strengthening effect of the $ZnFe_2O_4$ reinforcement particles and the formation of hard nickel phosphide phases during heat treatment [22, 19]. A favourable distribution of reinforcement particles within the Ni-P matrix may also restrict deformation during indentation and contribute to the enhanced hardness [12, 13]. At 3 g/L concentration, the microhardness decreases to 683 HV after heat treatment, which may be due to particle agglomeration and non-uniform particle distribution in the coating matrix [22, 15]. Compared with the pure Ni-P coating, the heat-treated composite coatings exhibit considerably higher microhardness [2, 3]. The maximum improvement is observed in the coating containing 2 g/L $ZnFe_2O_4$ nanoparticles [22, 13]. The microhardness increases from 280 HV for the mild steel substrate to 892 HV for the heat-treated Ni-P-$ZnFe_2O_4$ composite coating prepared at 2 g/L concentration [30, 1]. This clearly demonstrates the effectiveness of $ZnFe_2O_4$ nanoparticle reinforcement and heat treatment in improving the surface hardness of mild steel [15, 10]. Based on the microhardness results, it can be concluded that the incorporation of $ZnFe_2O_4$ nanoparticles significantly enhances the hardness of electroless Ni-P coatings [15, 14]. Among the investigated particle concentrations, 2 g/L provides the optimum reinforcement level, exhibiting the highest microhardness both before and after heat treatment [22, 19]. Furthermore, heat treatment considerably improves the hardness of all the coatings, with the maximum value of 892 HV obtained for the Ni-P-$ZnFe_2O_4$ composite coating containing 2 g/L nanoparticles [20, 15]. Therefore, the combination of optimum $ZnFe_2O_4$ particle concentration and heat treatment provides an effective approach for enhancing the surface hardness of electroless Ni-P composite coatings.

3.3 Corrosion Studies: An electroless deposition technique was employed to fabricate Ni-P-$ZnFe_2O_4$ composite coatings on a mild steel substrate using different zinc ferrite ($ZnFe_2O_4$) particle concentrations (1, 2 and 3 g/L) [22, 19]. Potentiodynamic polarization studies were conducted to evaluate the

electrochemical corrosion behaviour of the coatings by determining the corrosion potential (E_{corr}) and corrosion current density (i_{corr}). The corrosion behaviour of the coatings was investigated before and after the heat treatment process. [22]

The polarization results indicate that the corrosion resistance of Ni-P-$ZnFe_2O_4$ composite coatings improves with increasing particle concentration from 1 to 2 g/L [16, 18]. Before heat treatment, the corrosion potential shifts in the positive direction from -583 mV at 1 g/L to -521 mV at 2 g/L. Similarly, the corrosion current density decreases from 7.13×10^{-2} to 3.04×10^{-2} µA/cm² [18]. This reduction in corrosion current density indicates a lower corrosion rate and improved corrosion protection at 2 g/L particle concentration [19, 18]. The improvement in corrosion resistance can be attributed to the incorporation of $ZnFe_2O_4$ nanoparticles into the Ni-P matrix, which may enhance the compactness of the coating and reduce the exposure of the underlying mild steel substrate to the corrosive environment [28, 23]. However, at 3 g/L particle concentration, the corrosion potential shifts in the negative direction to -607 mV, accompanied by an increase in corrosion current density to 8.34×10^{-2} µA/cm² [19] . This behaviour indicates a reduction in corrosion resistance compared with the coating deposited at 2 g/L [28, 27]. Excessive particle concentration may result in particle agglomeration and non-uniform particle distribution within the coating matrix, potentially leading to pores and defects [20, 25]. These defects can provide pathways for the corrosive electrolyte to penetrate the coating and reach the underlying substrate [22, 21]. Therefore, the coating prepared with 2 g/L $ZnFe_2O_4$ reinforcement exhibits the best corrosion resistance before heat Treatment.

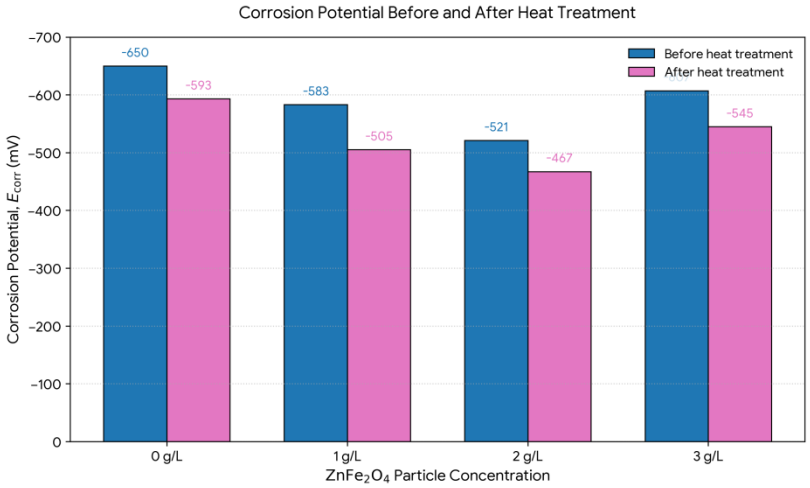


Fig 3.3.1: Corrosion Potential Before and After Heat Treatment [21,19]

The corrosion behaviour of Ni-P-$ZnFe_2O_4$ composite coatings was further investigated after heat treatment, and the corresponding electrochemical parameters are presented in Table 3.3.1 [22, 23]. The results demonstrate a significant improvement in corrosion resistance after heat treatment at all three particle concentrations [22]. A positive shift in corrosion potential is observed from -583 to -505 mV, -521 to -467 mV and -607 to -545 mV for 1, 2 and 3 g/L particle concentrations, respectively [22,23]. In addition, the corrosion current density decreases from 7.13×10^{-2} to 2.64×10^{-2} µA/cm² at 1 g/L, from 3.04×10^{-2} to 2.20×10^{-3} µA/cm² at 2 g/L and from 8.34×10^{-2} to 6.06×10^{-3} µA/cm² at 3 g/L [24]. The improvement in corrosion resistance after heat treatment can be attributed to structural changes in the electroless Ni-P coating [25]. Heat treatment promotes the crystallization of the initially amorphous Ni-P matrix and the formation of crystalline nickel and nickel phosphide phases [25, 26]. These structural transformations can improve coating compactness and reduce the number of electrochemically active sites, thereby limiting the interaction between the coating and the corrosive medium [26]. Consequently, the corrosion current density decreases and the corrosion potential shifts towards more positive values after heat treatment.

Among all the investigated particle concentrations, the coating prepared with 2 g/L $ZnFe_2O_4$ exhibits the highest corrosion potential (-467 mV) and the lowest corrosion current density (2.20×10^{-3} µA/cm²) after heat treatment [26]. This confirms that 2 g/L is the optimum particle concentration for achieving enhanced corrosion protection. [27] The improved performance may be associated with a more favourable distribution of $ZnFe_2O_4$ nanoparticles within the Ni-P matrix, resulting in an effective barrier against the penetration of corrosive species. Although the coating prepared with 3 g/L also exhibits improved corrosion resistance after heat treatment, its performance remains inferior to that of the 2 g/L coating [28].

Based on the potentiodynamic polarization results, it can be concluded that Ni-P-$ZnFe_2O_4$ composite coatings provide improved corrosion protection to the mild steel substrate both before and after heat treatment [29, 30]. Among the investigated concentrations, 2 g/L $ZnFe_2O_4$ reinforcement provides the best corrosion resistance, as evidenced by the most positive corrosion potential and the lowest corrosion current density [30]. Furthermore, heat treatment significantly enhances the corrosion resistance of all the composite coatings [29]. The minimum corrosion current density of 2.20×10^{-3} µA/cm² obtained for the heat-treated coating containing 2 g/L $ZnFe_2O_4$ confirms its superior electrochemical performance.

4. CONCLUSIONS:

This study focuses on preparing an electroless Ni-P-$ZnFe_2O_4$ composite coating on mild steel and studying its structure, surface properties, hardness, and corrosion resistance.[1,2,3] Mild steel specimens of 20 × 20 mm² were used as the substrate. Before coating, the specimens were polished using different grades of SiC papers to obtain a clean and smooth surface. [26, 25] They were then washed with deionized water and acetone to remove dust, oil, and other impurities. After cleaning, the specimens were activated using 10% HCl for 2 minutes to improve the adhesion of the coating. [27] The electroless coating solution was prepared using nickel chloride, sodium hypophosphite, ammonium chloride, and trisodium citrate.[28] Nickel chloride

provided the nickel ions, while sodium hypophosphite acted as the reducing agent and phosphorus source. Ammonium chloride was used as a bath constituent, and trisodium citrate helped to stabilize the solution.[29,28] $ZnFe_2O_4$ nanoparticles were added to the coating solution as the reinforcement. SDS, CTAB, and PVP were used separately as surfactants to improve the dispersion of the nanoparticles and reduce their agglomeration.[29] Sonication was also used to obtain better dispersion of $ZnFe_2O_4$ particles in the coating solution. During the coating process, the bath temperature was maintained at 88 ± 2 °C and the pH was kept between 4.5 and 5.5.[25] Initially, a Ni-P pre-coating was deposited on the mild steel specimen for 60 minutes. This was followed by the deposition of the Ni-P-$ZnFe_2O_4$ composite coating for 120 minutes.[27,26] Since the coating process does not require an external electric current, it is known as electroless coating. After deposition, the specimens were washed properly and dried before further testing.[28,29] The coated specimens were analyzed using Surface Roughness, hardness testing, and corrosion testing. Vickers microhardness testing was carried out using a 100 g load with a holding time of 10 seconds.[30,27] Five indentations were taken for each specimen, and their average value was considered as the hardness value. Corrosion behaviour was studied using the potentiodynamic polarization method in a 3.5% NaCl solution [29]. The coating acts as a protective layer over the mild steel surface and helps reduce direct contact between the steel and the corrosive environment[29, 30]. The $ZnFe_2O_4$ nanoparticles are expected to influence the hardness and corrosion behaviour of the Ni-P coating, while the surfactants and sonication help in achieving better particle distribution[30].

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