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Innovative Fabrication Techniques of Superhydrophobic Coatings for Corrosion Protection of Magnesium Alloy (AZ31): A Review

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Abstract

Magnesium alloys, especially AZ31, are widely used as substitutes for plastic, aluminum, and steel due to their low weight and high mechanical performance. These properties make them a key subject of research and a preferred material in many engineering applications, magnesium earning the title of "Green Engineering Material of the 21st Century". Despite these advantages, their high chemical and electrochemical reactivity cause rapid corrosion when exposed to humid, marine, and chemical environments, leading to structural degradation, increased maintenance costs, and reduced service life in industrial use. To address this limitation, recent research has focused on surface modifications, particularly the development of superhydrophobic (SHP) coatings as a practical, cost-effective solution to enhance corrosion resistance. These coatings reduce contact with corrosive media by combining hierarchical structures with low surface energy materials. This review summarizes the basic principles of wettability and theoretical wetting models, then presents recent advances in SHP coatings for AZ31 alloys. It focuses on standard fabrication methods for developing micro/nano structuring and low surface energy treatments, including hydrothermal synthesis, electrochemical deposition, laser treatment, spraying coating, chemical etching, and micro-arc oxidation. Finally, the paper outlines key challenges and proposes future research directions for improving the corrosion protection of magnesium alloys.

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Keywords: Magnesium Alloy; Corrosion Resistance; Superhydrophobic Coatings.

1. Introduction

Magnesium alloy AZ31 is one of the lightest metallic structural materials. It has attracted significant interest from researchers in applied research and engineering due to their unique properties, such as a high strength-to-weight ratio, low specific gravity, high specific strength and stiffness, sound vibration and shock absorption capacity, high damping capacity, effective electromagnetic shielding, good dimensional stability, biocompatibility, environmentally friendly properties, and recyclability, etc. [1-3]. This makes it an attractive choice in several industrial fields, including the aerospace sector [4], automotive [5], biomedicine [6], transportation [7], communication [8], and other industries. As shown in Fig. 1, Mg alloys offer a unique set of properties and are widely applied in key industrial areas, confirming their significance as high-performance materials in modern engineering systems.

Nevertheless, despite these advantages of magnesium alloys, they are prone to corrosion and performance failure. That is the major obstacle to their use in aqueous and atmospheric environments, especially in solutions containing $Cl^-$and NO_3^-. Magnesium alloys have high chemical activity and a negative electrode potential of -2.37 V, which leads to easy oxidation, corrosion, and surface degradation [9]. However, the spontaneously formed oxide or hydroxide film on Mg alloys is not effective in protecting the surface, as it has a loose and porous structure that cannot prevent the penetration of the corrosive medium to the underlying metal. This results in continued corrosion and weakens the long-term resistance of the alloy [10]. Improving corrosion resistance remains a critical challenge due to its substantial economic and structural impacts. The complete prevention of corrosion is not possible. Still, many methods help to reduce and slow down its progression, such as cathodic and anodic protection and surface coatings. Among these, superhydrophobic (SHP) coatings represent an effective method to reduce corrosion rates because they minimize contact between the surface and corrosive media. The production of protective coatings stands as a widely accepted, practical, and cost-effective solution [11-13].

Nomenclature & Symbols			
Mg AZ31	Magnesium Alloy Containing Al (3%), Zn (1%)	SA	Sliding Angle (°)
SHP	Superhydrophobic	i_{corr}	Corrosion Current Density (A/Cm^2)
Cl^-	Chloride Ion	R_{ct}	Charge Transfer Resistance ($\Omega.Cm^2$)
NO_3^-	Nitrate Ion	θ	Contact Angle (°)
NaCl	Sodium Chloride	γ	Surface Tension
DTMS	Dodecyltrimethoxysilane	f_s	Solid Surface Fraction (Cassie–Baxter Model)
PFOTMS	Perfluorooctyltrimethoxysilane	θ_Y	Young's Contact Angle (Ideal Surface)
WCA	Water Contact Angle (°)	θ_W	Wenzel's Contact Angle
CA	Contact Angle (°)	θ_{CB}	Cassie–Baxter Contact Angle

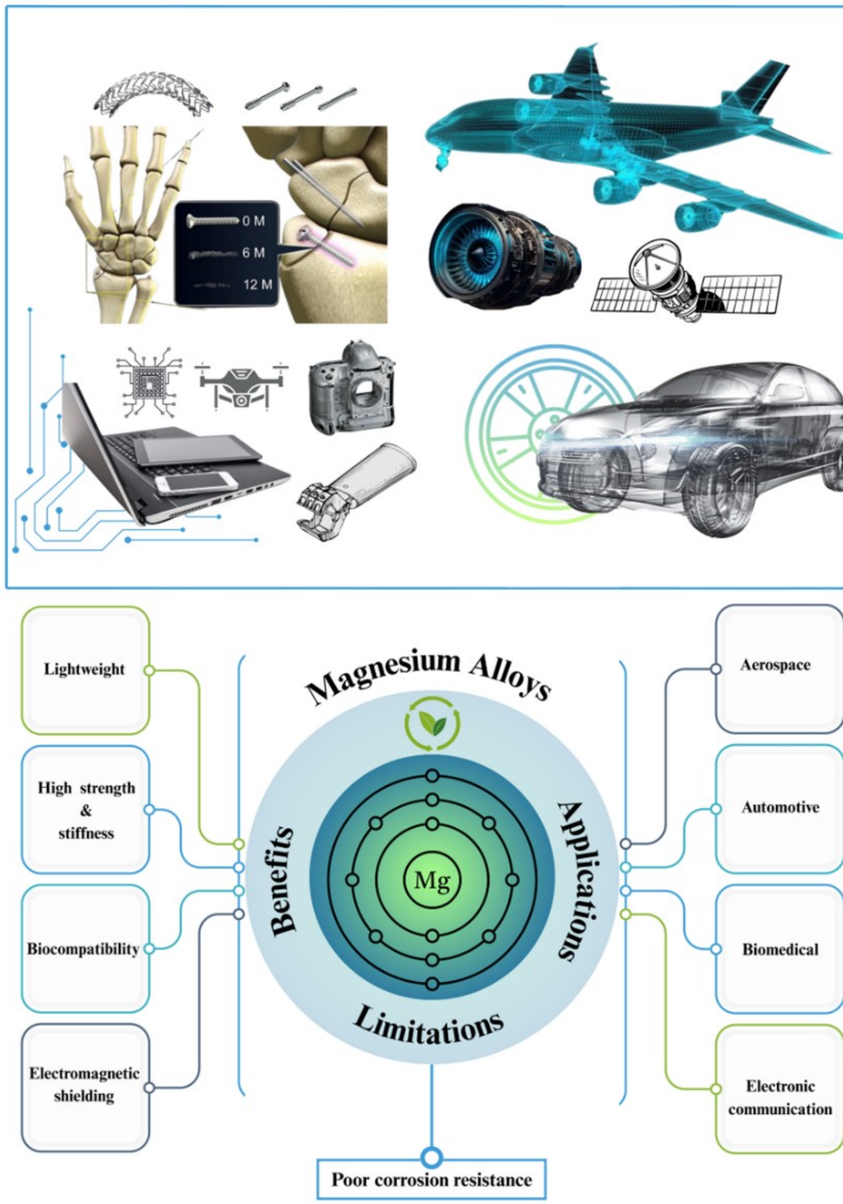

Fig. 1. Magnesium alloys, key properties, and typical applications [14-16]

To improve the effectiveness of protective coatings, researchers have drawn inspiration from nature to create superhydrophobic coatings after discovering similar properties in plants, animals, and insects [17-21]. This observation led them to study these natural mechanisms for developing artificial surfaces with equivalent capabilities. This biological behavior is mainly attributed to surface wettability, which exhibits water repellency due to its special surface textures and low surface energy. In plants, these surfaces act as barriers that prevent water from accumulating. When water contacts the surface, droplets easily roll off, keeping it dry and lightweight, reducing dust, pollen adhesion, and microbial growth. In insects like mosquitoes, the eyes have superhydrophobic coatings that function as an anti-fogging surface. Similarly, in insects, wings and cuticles are also covered with water-repelling layers which maintain their dryness and light-weight structure, thus enhancing their ability to move and survive. As shown in Fig. 2.

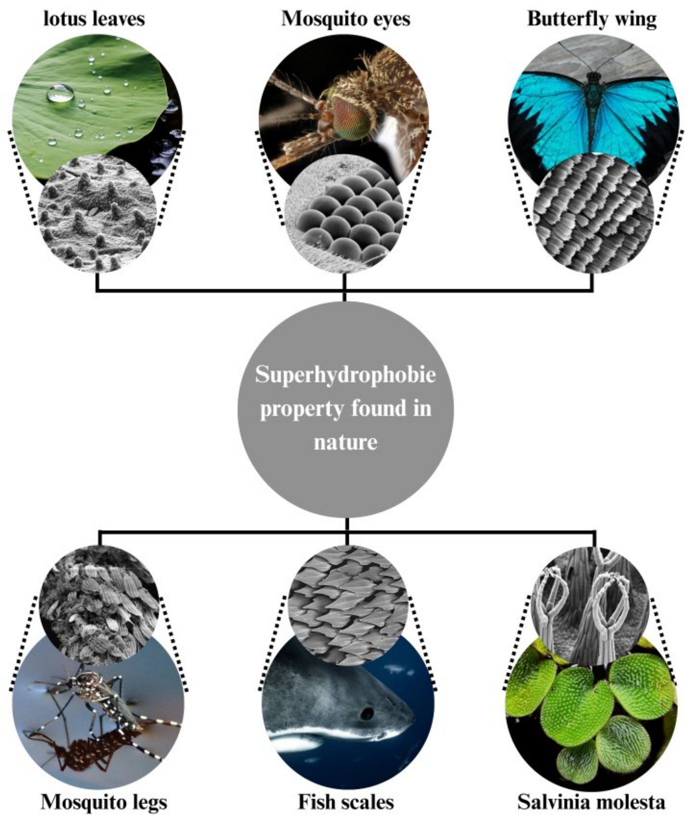

Fig. 2. Superhydrophobic property found in nature [20, 21]

Through the exploration of superhydrophobic surfaces in nature and the analysis of the superhydrophobic theoretical model, researchers have discovered that superhydrophobic surfaces exhibit water contact angles above 150° and sliding angles below 10° [22], relies on two primary techniques: (1) the construction of micro/nano hierarchical structures on the substrates and (2) the chemical modification of a hierarchically structured surface with materials that have low surface energy, such as fatty acids, polymers, hydrocarbons, and fluorocarbon compounds [23-25]. Thus, obtaining similar surface morphological characteristics of the hydrophobic organisms in nature results in a highly rough and porous coating that traps air in the structures to form a physical barrier that prevents direct contact with corrosive water agents. Simultaneously, low surface energy materials can enhance the protection against corrosion by reducing the adherence of water on the coating [26]. This corrosion protection mechanism provided by superhydrophobic coatings is illustrated in Fig. 3.

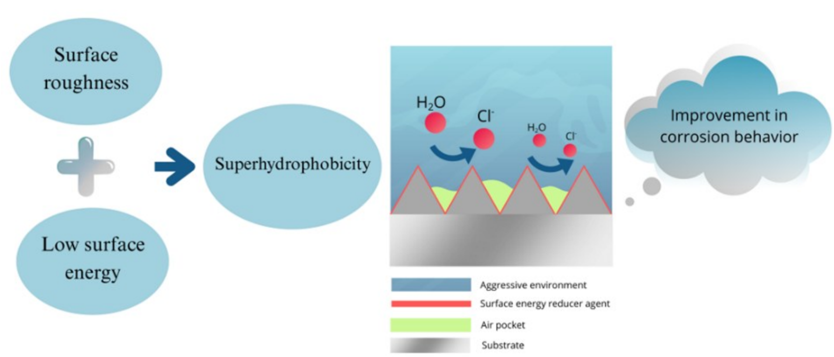

Fig. 3. The schematic of corrosion resistance improvement by superhydrophobicity [27]

In addition to surface roughness, low wettability is a key factor in achieving superhydrophobicity. Surfaces with low wettability promote the formation of nearly spherical water droplets that easily roll off rather than spread. The wetting behavior of solid surfaces is described using three main theoretical models: Young's model, Wenzel's model, and Cassie–Baxter model [28, 29]. These models are used to evaluate the contact angle of the liquid droplet on solid surfaces. Young's model (Fig. 4a) applies to an ideal, smooth, rigid, and homogeneous surface. However, such surfaces rarely exist in practice, as real surfaces inherently possess microscopic roughness. The applicability of Young's model in real-world conditions. The model expresses the contact angle θ_γ as Eq. (1):

$$\cos\theta_\gamma = \frac{\gamma_{sv}-\gamma_{sl}}{\gamma_{lv}} \tag{1}$$

Where γ_{sv}, γ_{sl}, and γ_{lv}, are the surface tensions between the solid-vapor, solid-liquid, and liquid-vapor interfaces, respectively.

In Wenzel's model (Fig. 4b), it is assumed that the liquid fully penetrates the grooves of the rough surface, increases the actual contact area between the liquid and solid, thereby enhancing the inherent wettability of the surface. The Wenzel contact angle θ_w is defined as Eq. (2):

$$\cos\theta_w = r\cos\theta_\gamma \tag{2}$$

Where r is the surface roughness ratio (actual area divided by projected area), in this model, roughness amplifies the surface's intrinsic wettability, making hydrophilic surfaces more wettable and hydrophobic surfaces more water-repellent.

In contrast, Cassie Baxter's model (Fig. 4c) describes a state where the water droplets rest on a composite interface made of solid structures and trapped air pockets. This structure prevents liquid from penetrating the pores and minimizes the solid contact area due to the presence of air. Their model consists of two parts. The first part includes the surface fraction f_1 with contact angle θ_1, and the second part provides f_2 with contact angle θ_2, where in Eq. (3):

$$f_1 + f_2 = 1 \tag{3}$$

The apparent contact angle θ is then given by the general form of Cassie–Baxter as Eq. (4):

$$\cos\theta = f_1\cos\theta_1 + f_2\cos\theta_2 \tag{4}$$

In superhydrophobic applications, the second phase θ_2 is typically air, for which the contact angle is considered 180°. Substituting this simplifies to Eq. (5):

$$\cos\theta_{CB} = f_s\cos\theta_s + (1 - f_s) \tag{5}$$

Where f_s is the solid fraction in contact with the liquid, and θ_s is the intrinsic contact angle of that solid. Most artificial superhydrophobic surfaces conform to the Cassie–Baxter model, where the trapped air plays a critical role in minimizing adhesion and allowing water droplets to roll off easily [20, 25] (Fig. 4).

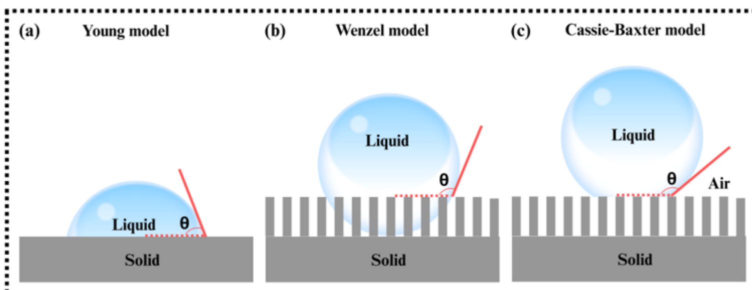

Fig. 4. Schematic of water droplets on smooth and rough surfaces [28]

However, a hydrophilic surface allows corrosive solutions to diffuse and penetrate easily into the magnesium alloy, which leads to corrosion. In contrast, superhydrophobic coatings exhibit multifunctional surface properties, including water repellency, anti-corrosion, anti-fogging, anti-bacterial, anti-icing surfaces, self-cleaning, oil and water separation, drag reduction, water purification, and antifriction. Etc. These properties play a crucial role in reducing the corrosion of Mg alloys in harsh environments such as marine and soil conditions, where they effectively minimize degradation and extend the material's service life [23, 30, 31] (Fig. 5). Hence, various physical and chemical approaches have been devised to prepare superhydrophobic coatings and enhance corrosion resistance of Mg alloys, including electrochemical deposition, wet chemical reaction, spray coating, hydrothermal, micro-arc oxidation (MAO), and electrospinning methods [23, 25, 32]. Each technique aims to create the roughness and low surface energy required to achieve stable and durable superhydrophilicity.

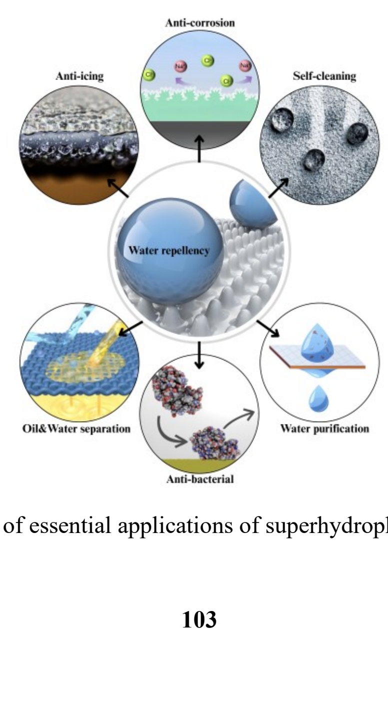

Fig. 5. Schematic of essential applications of superhydrophobic surfaces [33]

2. Techniques for Fabricating Superhydrophobic Coatings

Recent research has focused extensively on developing superhydrophobic coatings for corrosion protection in harsh environments. Various physical and chemical fabrication methods have been investigated, including hydrothermal treatment, chemical etching, spraying, electrodeposition, micro-arc oxidation, and laser-based processing, as shown in Fig. 6. Each method aims to generate the required surface roughness and reduce surface energy to achieve superhydrophobicity. Despite promising outcomes, challenges such as limited durability, weak adhesion, and high process complexity remain. This review summarizes key studies and recent advances in the literature, offering a comparative overview of the most effective techniques applicable to AZ31 magnesium alloy. The advantages and disadvantages of each method regarding cost, scalability, surface adhesion, and corrosion protection efficiency are presented in Table 1.

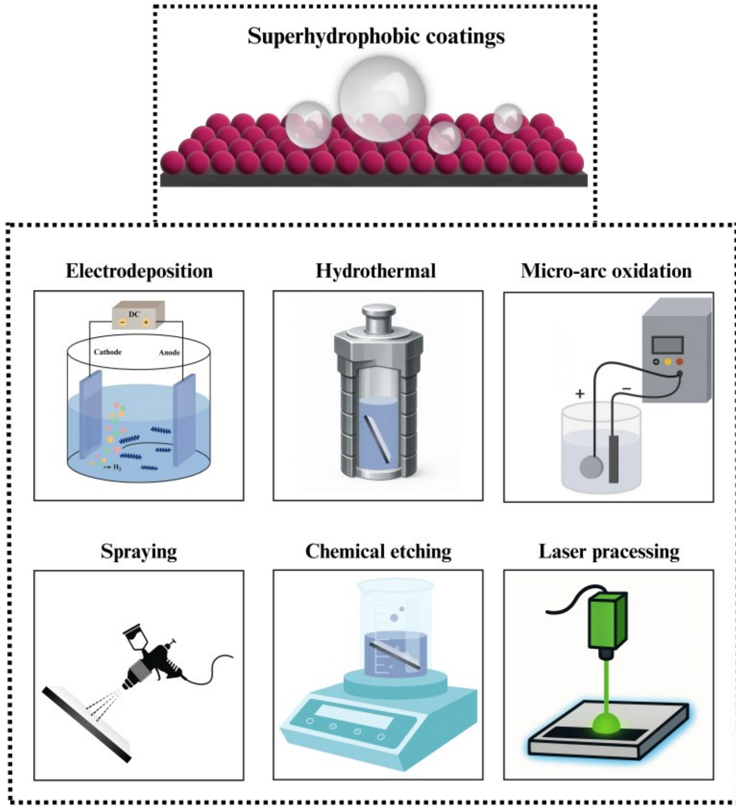

Fig. 6. Fabrication strategies of superhydrophobic coatings [32]

Table 1. Methods used to prepare SHP and their advantages and disadvantages

Method	Cost	Advantages	Disadvantages	Ref.
Electrodeposition	Expensive	Fast, reproducible results, and controllable surface properties.	Requires careful preparation, poor adhesion, and stability.	[34-37]
Hydrothermal	Inexpensive	Simple, produces uniform, dense, and adherent micro/nano-structured layers.	Not suitable for large or complex surfaces.	[25, 32, 38]
Micro-arc oxidation	Expensive	Produces coatings with high hardness and good adhesion, often used as a strong base coat.	Requires high voltage and current density, limited surface structure, and needs post-treatment.	[27, 39-42]
Spraying	Inexpensive	Easy to use, fast, environmentally friendly, and suitable for wide applications.	May result in material loss and uneven coating if the process is not calibrated correctly.	[25, 32, 43]
Chemical etching	Inexpensive	Simple, no complex equipment required, saves time, and produces a rough surface.	Requires precise control to avoid corrosion, producing harmful metal waste.	[44-48]
Laser processing	Expensive	Precise control of surface structure, high speed, and reduced need for chemical treatment.	Costly equipment and operational complexity require expertise to control the process.	[27, 44, 49]

2.1. Electrodeposition technique

Electrodeposition is a practical method for the creation of micro-and nanostructures on magnesium alloys. The process involves the deposition of a thin coating layer onto the surface from a solution containing charged ions or nanoparticles. The process operates at low cost with rapid and reproducible results, which supports efficient large-scale production. Additionally, the main strengths of this method are the ability to control surface properties through adjustments of solution composition, deposition time, and current density. The wide application of electrodeposition exists in electronics, protective coatings, and biotechnology because of its flexibility [34-36]. At present, researchers focus on using electrodeposition technology to form functional layers on magnesium alloys, followed by surface treatment with low-surface-energy materials to enhance water repellency and improve corrosion resistance.

For example, Zheng Yin et al. [50], as shown in Fig. 7, created superhydrophobic organic/inorganic hybrid coatings on AZ31 alloy through a two-step process that started with alkaline surface treatment followed by electrodeposition. The first step involved forming an $Mg(OH)_2$ layer by placing the alloy in a NaOH solution. Then, the electrodeposition method was used in an ethanolic solution containing 0.1 M myristic acid, $CH_3(CH_2)_{12}COOH$, and 0.1 M calcium nitrate, $Ca(NO_3)_2$, at 30 V for 15 minutes at room temperature, followed by drying with hot air. The treatment created a composite coating that contained $Mg(OH)_2$ and $Ca[CH_3(CH_2)_{12}COO]_2$ with a flower-like micro/nano surface structure. The treated surface demonstrated outstanding water-repellent behavior through its 159.2° water contact angle and its 5.2° sliding angle. The corrosion resistance improved substantially because the corrosion current decreased to 1.86×10^{-8} A/cm², the hydrogen evolution rate decreased to 4.2 μL/cm²/h, and the adhesion strength reached 3.21N. The coating demonstrates superior corrosion protection and strong bonding properties, together with superior water-repelling capabilities, which make it suitable for use in humid or corrosive conditions.

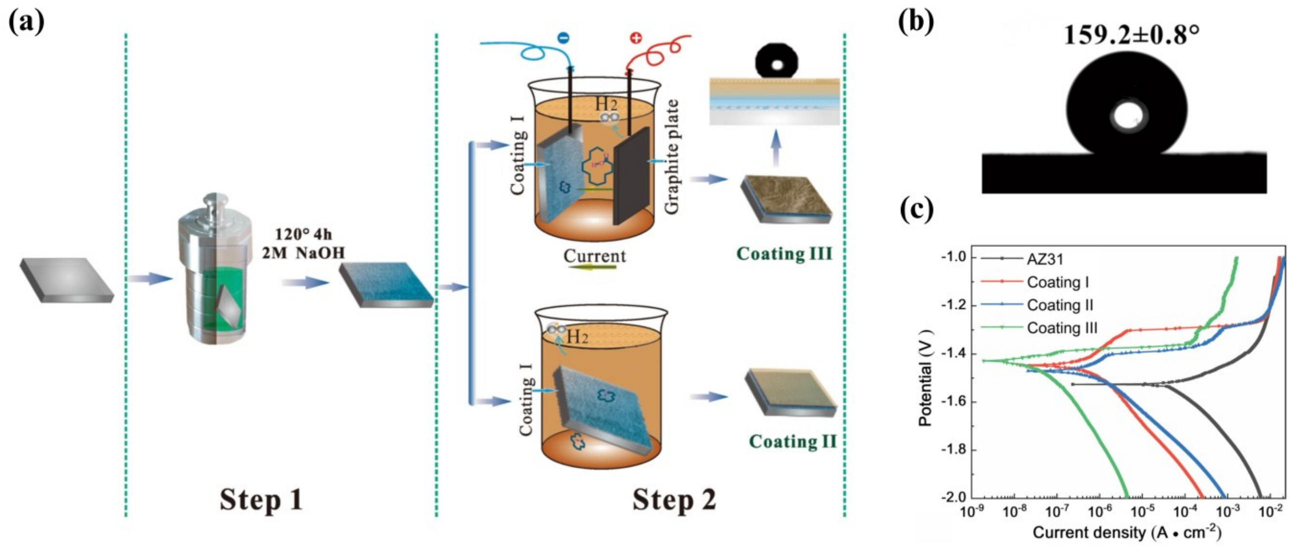

Fig. 7. (a) Fabrication of the composite coatings, (b) CA of coating III, (c) Potentiodynamic polarization curves [50]

Sheng-Jian Zhang et al. [26], as shown in Fig. 8, developed a superhydrophobic composite coating of $Mg(OH)_2$ and dodecyltrimethoxysilane (DTMS) on AZ31 alloy surfaces to improve corrosion protection. The researchers applied hydrothermal treatment followed by electrodeposition as a two-step method. The samples were treated with a sodium hydroxide NaOH solution to create an $Mg(OH)_2$ layer. Then, the electrodeposition method was used in a solution containing 0.2 M KNO_3, 3 ml of DTMS, and 80 ml of ethanol to create the DTMS layer. The final coating exhibited a walnut shell-like nanostructure with excellent hydrophobic properties, with a CA of 165.1 and a SA of 3.5°. The protective coating exhibited excellent corrosion resistance, as the corrosion current density decreased to 1.777×10^{-8} A/cm² compared to 7.342×10^{-5} A/cm² for the untreated surface, indicating a significant improvement in protective performance.

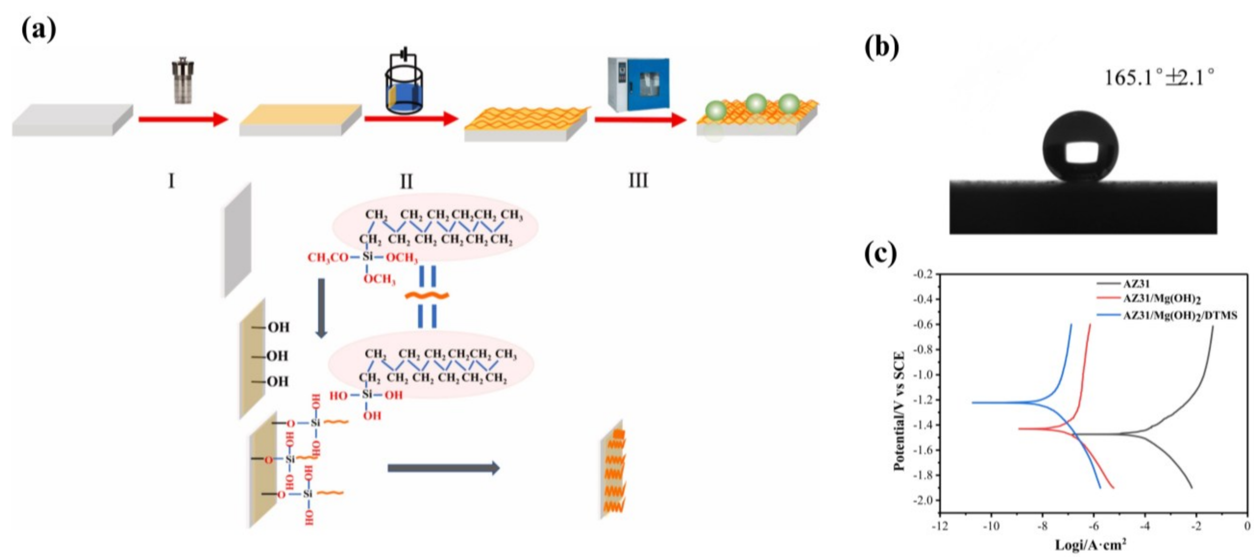

Fig. 8. (a) Schematic diagram of fabricating the $Mg(OH)_2$/DTMS composite coating on the AZ31 substrate, (b) WCA of the $Mg(OH)_2$/DTMS coating, (c) Potentiodynamic polarization curves [26]

Xinghui Sun et al. [51], as shown in Fig. 9, created calcium/cerium myristate (Ca/Ce) coatings on anodized AZ31 alloy through an efficient electrodeposition process. The preparation process started by applying anodic oxidation to the AZ31 alloy at 4 V for 10 minutes in $Na_2C_2O_4$, NaOH, and $C_2H_6O_2$ solution to enhance surface roughness and improve coating adhesion. Then, the electrodeposition of the coating occurred at 50 V for 45 minutes in ($CaCl_2$, $CeCl_3 \cdot 3H_2O$) and myristic acid ethanol solution. The effect of the Ca/Ce ratio on the formation, structure, thickness, hydrophobicity, and corrosion resistance was investigated. The sample with a (40Ca1Ce) ratio showed the best performance by achieving a CA of 165.86°, which indicated excellent water-repellent properties and a nearly crack-free surface. Additionally, the coating showed a decrease in corrosion current by about three orders of magnitude compared to the oxidized alloy and maintained its stability during exposure to 3.5% NaCl solution. The optimal Ca^{2+} to Ce^{3+} ratio produces an effective combination of surface property improvement and corrosion resistance enhancement for magnesium alloy.

However, despite its advantages, the electrodeposition remains expensive and often requires detailed preparation to ensure a uniform coating. In addition, the adhesive strength and long-term stability of electrodeposited superhydrophobic coatings need to be strengthened further.

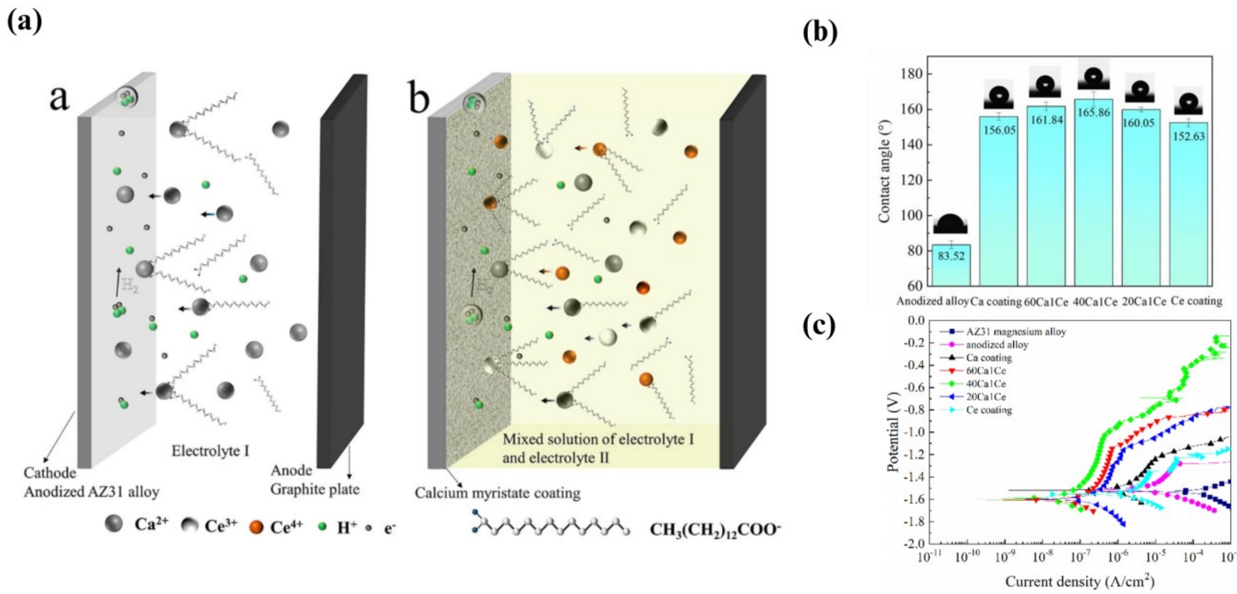

Fig. 9. (a) Process of electrodeposition illustrated schematically: a before and b after electrolyte II is dribbled into electrolyte I, (b) The CA of coatings, (c) The potentiodynamic polarization curves [51]

2.2. Hydrothermal synthesis technique

The hydrothermal method is widely applied to fabricate superhydrophobic coatings on magnesium alloys, due to simplicity, cost-efficiency, and ability to generate a uniform, dense, and tightly adherent micro/nanostructured layer. This process involves placing chemical precursors in a sealed autoclave and exposing them to elevated temperature and pressure. This process is also eco-friendly, typically using water or mild oxidants. Additionally, it allows precise control over surface structure and crystal growth. The resulting final surfaces typically show high water CA, low SA, and improved resistance to corrosion [25, 32]. Many studies have demonstrated the effectiveness of hydrothermal synthesis on magnesium alloy substrates by tuning processing conditions and applying different surface treatments.

For instance, the authors, Zhengwei Song et al. [52], as shown in Fig. 10, fabricated a lanthanum-based superhydrophobic coating on AZ31B magnesium alloy using a one-step hydrothermal method. The reaction solution contained 1.5 g/dm³ of myristic acid $CH_3(CH_2)_{12}COOH$ and 25 g/dm³ of lanthanum nitrate ($La(NO_3)_3 \cdot 6H_2O$), dissolved in ethanol. The mixture was stirred for 10 minutes, then placed with the AZ31B substrate into a 25 mL Teflon-lined stainless-steel autoclave and reacted at 170 °C for 5 hours. After cooling, the coated samples were rinsed with ethanol and dried. The resulting coating of myristic acid and lanthanum at 5 hours immersion (ML-5) displayed a two-layered structure, which included a compact inner oxide layer together with an outer layer of needle-like micro-nano structures. The water contact angle reached 159°, which demonstrated both strong superhydrophobicity and low water adhesion. The ML-5 coating showed a corrosion current density that was five orders of magnitude lower than the bare substrate when tested in 3.5 wt.% NaCl solution. The electrochemical impedance spectroscopy results showed that the coating provided long-term corrosion protection because the low-frequency impedance modulus |Z| at 0.01 Hz reached 9.01×10^6 $\Omega \cdot cm^2$ after 15 days and stayed at 6.85×10^6 $\Omega \cdot cm^2$ after 25 days of immersion.

The high impedance, together with the sustained phase angle above 70° throughout the test period, demonstrated effective barrier performance. The excellent corrosion resistance resulted from the combined effect of the dense inner oxide barrier, the air cushion trapped by the outer layer, and the self-cleaning surface. This method presents an effective method to create durable corrosion-resistant coatings on magnesium alloys.

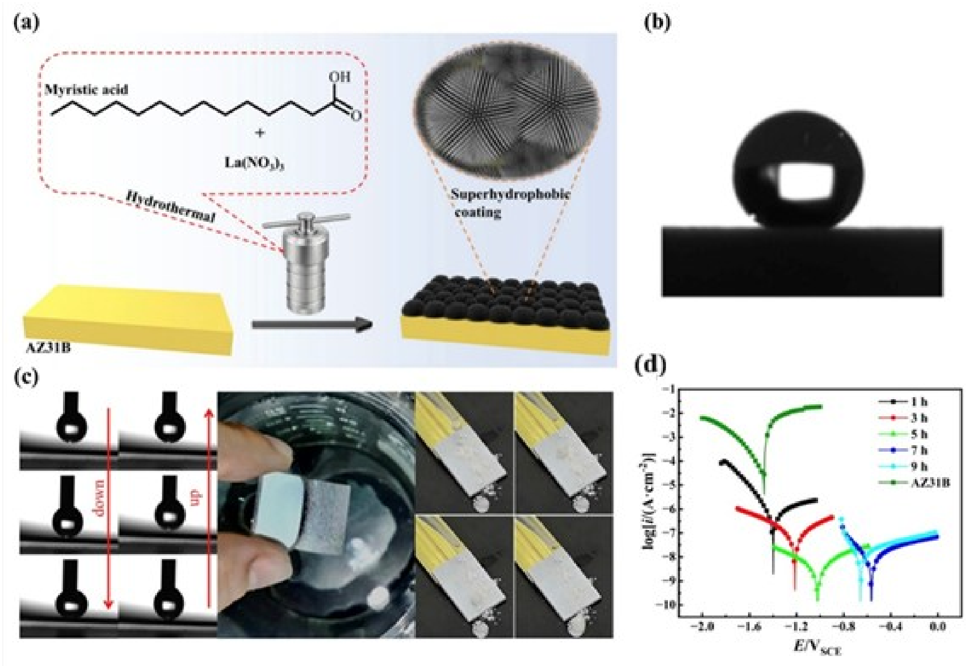

Fig. 10. (a) The schematic diagram for the fabrication process of superhydrophobic coatings on the AZ31 alloy, (b) The CA of coating, (c) Water adhesion test, the air-film in aqueous solution and the self-cleaning behaviour of ML-5, (d) Potentiodynamic polarization curves [52]

In addition to this, Qing Jin et al. [2], as shown in Fig. 11, formed superhydrophobic coatings on AZ31B magnesium alloy through hydrothermal synthesis, then modified the surface with stearic acid $CH_3(CH_2)_{16}COOH$. This treatment produced surface features interlaced with lamellar magnesium hydroxide $Mg(OH)_2$, providing a rough micro-nanoscale surface texture. This coating exhibited a high CA of 159° and a lower SA of 7°. Furthermore, electrochemical testing results showed a corrosion current density 100 times lower than that of the bare alloy. The coating also maintained its protective properties after 10 days of exposure to a sodium chloride solution, demonstrating the coating's durability and stable protective performance.

Zhao-Qi Zhang et al. [53], as shown in Fig. 12, developed a superhydrophobic polypropylene (PP) coating on AZ31 magnesium alloy using a one-step hydrothermal process, after pretreating the surface with $Mg(OH)_2$. The resulting surface had a micro-scale spherical texture that gave it a high-water contact angle of 165.5° and a low sliding angle of 4°, confirming strong water repellency. Additionally, the electrochemical test showed a significant boost in corrosion resistance, with the corrosion current density dropping by four orders of magnitude compared to the uncoated magnesium alloy. This improvement came from the combined impact of the rough structure of the surface and low surface energy. Even after 250 hours of immersion in a 3.5 wt.% sodium chloride solution, the coating kept its hydrophobic performance, demonstrating its ability to withstand long-term exposure and remain mechanically stable in harsh environments.

Moreover, the hydrothermal method enables the creation of superhydrophobic coatings with remarkably enhanced corrosion resistance by decreasing the contact area between the corrosive solution and the sample, due to the water repellency of the coating. Despite these advantages, the method still has some limitations, as hydrothermal processing is not suitable for preparing coatings over large areas and complex shapes.

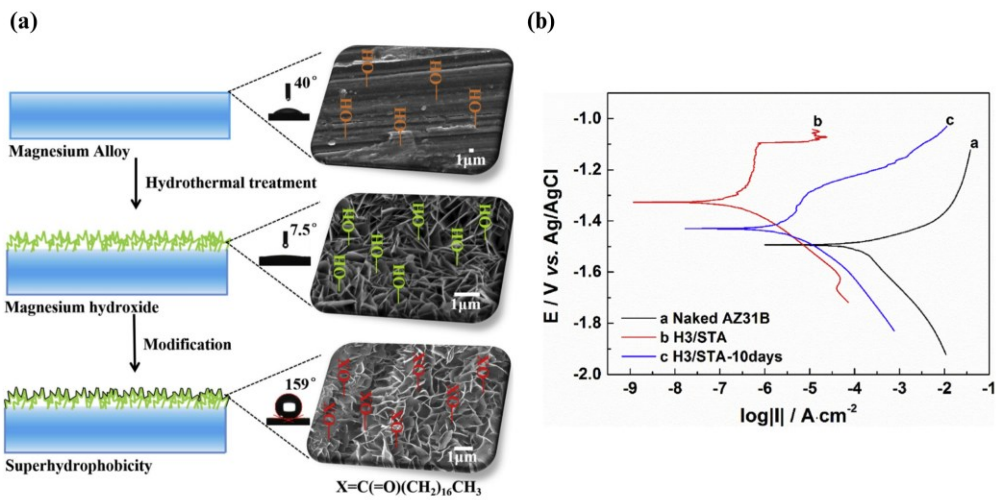

Fig. 11. (a) Schematic illustration of the formation mechanism of superhydrophobic coating, (b) Potentiodynamic polarization curves [2]

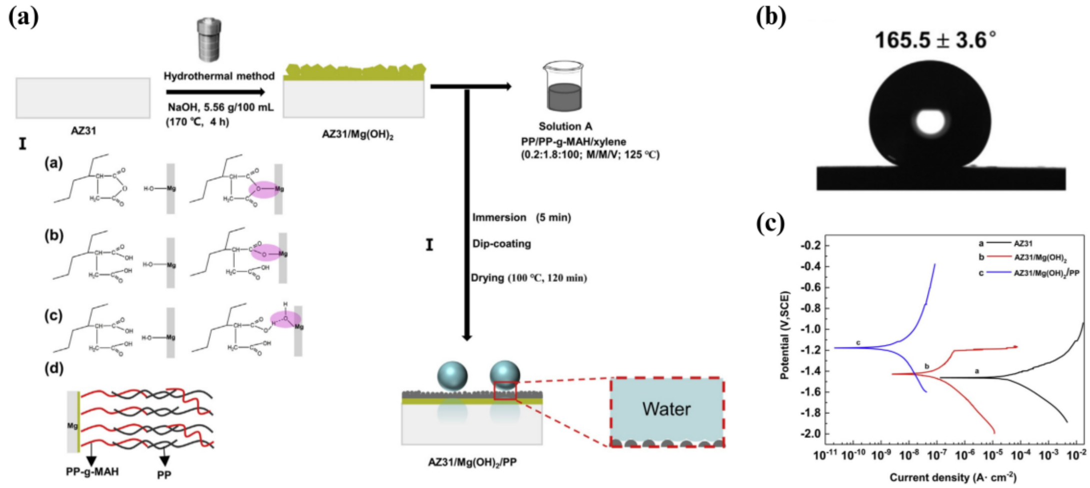

Fig. 12. (a) Fabrication of the $Mg(OH)_2$/polypropylene coating, (b) The CA of the $Mg(OH)_2$/polypropylene coatings, (c) Potentiodynamic polarization curves [53]

2.3. Micro-arc oxidation methods

Micro-arc oxidation (MAO) is a modern method, derived from traditional oxidation techniques, for treating magnesium alloy surfaces. It involves applying a high voltage in a suitable electrolyte solution to form a ceramic oxide film on the alloy surfaces. This resulting coating has a rough microstructure surface that exhibits strong adhesion properties. The porous surface resulting from this process provides a base for chemical modification by using suitable chemical compounds that have low surface energy, for the fabrication of the superhydrophobic coating. It improves the corrosion resistance and abrasion resistance of the Mg alloy. Current research focuses on the development of hybrid coatings by the MAO method, which provide corrosion protection and biocompatibility while enabling superhydrophobic functionality for advanced medical and industrial applications. [27, 32, 39-41].

Dongjie Liu et al. [54], as shown in Fig. 13, prepared a superhydrophobic surface on AZ31 Mg alloy using three main steps, including the formation of a micro/nanostructured LDH/MAO film by using MAO and a hydrothermal process. Then, the surface was treated by coating the layered double hydroxide/micro-arc oxidation (LDH/MAO) film with octyltriethoxysilane (OTES) solution using the spin-coating method to form a superhydrophobic surface. A solution containing Na_2SiO_3, NaOH, and KF was applied at different voltages (250, 300, and 350 V) for 30 minutes, using the MAO process. The MAO-treated surface then underwent a hydrothermal reaction to form an Mg Al-LDH layer. An aqueous solution containing $Mg(NO_3)_2 \cdot 6H_2O$ at a concentration of 0.05 mol/L and different concentrations of $Al(NO_3)_3 \cdot 9H_2O$ (0.01, 0.03, and 0.05 mol/L) was prepared, and the pH was adjusted to 10 using NaOH. The samples were placed in an autoclave at 100°C for 18 hours, resulting in the formation of a micro-/nano-structured LDH/MAO composite layer. In the third step, using the spin-coating method, the surface was modified with an OTES solution through a two-step spin-coating process. The first stage lasted 9 seconds and 600 rpm, while the second stage lasted 20 seconds and 2,100 rpm. In the first stage, the OTES solution was added to the sample surface. After spin-coating, the sample was placed in an electric drying oven for drying. After repeating the process several times, the OTES-LDH/MAO composite coatings were obtained. The results showed that the composite coatings were able to seal the cracks and pores in the MAO film well, achieving a superior hydrophobic effect and high corrosion resistance. Compared with the MgO film, the water CA increased to 155°, with the corrosion current density (4.12×10^{-10} $A \cdot cm^{-2}$) and largest impedance modulus (5.4×10^{-6} $\Omega \cdot cm^2$). The corrosion resistance becomes much higher than that of the MAO film.

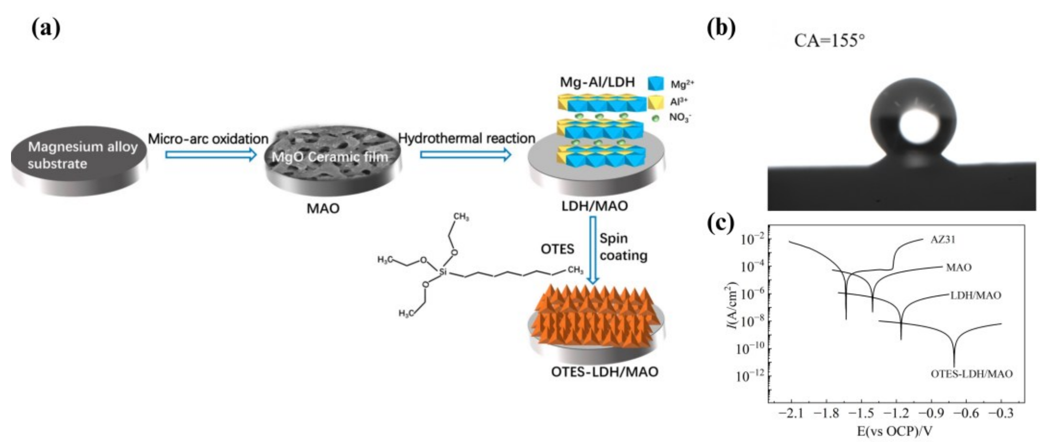

Fig. 13. (a) Schematic illustration for forming the OTES-LDH/MAO composite coatings, (b) The CA of coating, (C) Polarization curves of various samples [54]

Zhao-Qi Zhang et al. [55], as shown in Fig. 14, integrated a superhydrophobic polypropylene (PP) coating on AZ31 magnesium alloy by using the micro-arc oxidation followed by a simple dipping process. The MAO-PP hybrid coating was prepared by applying the first layer of coating, using the MAO process at a constant voltage of 300 V at 350 Hz for 3 min, in an electrolyte solution consisting of NaOH (8 g/L), NaSiO3 (10 g/L), and KF (5 g/L). The as-prepared coating was then washed with distilled water to remove the residual electrolyte. The second layer was prepared from a mixed solution by adding PP (1.5 g) and PP-g-MAH (0.3 g) to xylene (100 mL) at 130 °C. The MAO sample was immersed in the mixed solution for 3 min by the dip-coating method, thus obtaining a PP layer on the MAO surface. The results showed micro-scaled granular structure on the surface, resulting in a high CA of 167.2° and a low SA of 2.7°. In addition, it exhibited superior adhesion, durability, and high corrosion resistance, with a low corrosion current density of 8.76×10^{-9} A/cm² compared with bare Mg alloy. Furthermore, the coating maintained its integrity after 248 hours of immersion in a 3.5 wt.% aqueous sodium chloride solution.

Ai-hui LIU et al. [56] prepared a superhydrophobic coating on AZ31 Mg alloy by using a two-step process involving micro-arc oxidation (MAO) and stearic acid ethanol solution treatment. The first layer was prepared in an aqueous solution of $Na_2SiO_3{\cdot}9H_2O$ and NaOH at concentrations of 15 and 5 g/L, respectively, through the MAO process, using a homemade 10 kW pulse bipolar power supply. The electrolyte solution was stirred and cooled to below 35 °C. The coated samples were then cleaned with distilled water after treatment and air-dried at room temperature. The coating consisted of MgO and Mg_2SiO_4 phases. It contained circular pores with an average diameter of approximately 900 nm and a thickness of 6.86 µm, providing a fine structure at the microscopic and nanoscales. This was achieved at a voltage of 350 V, a frequency of 1000 Hz, and a curing time of 5 minutes. The microarc-oxidized samples were then treated in a 10 g/L stearic acid ethanol solution at room temperature for 3 h and then dried in an oven at 120 °C for 1 h. To form a superhydrophobic coating. This composition significantly improved the CA of 156.96°, and the corrosion resistance of the AZ31 Mg alloy, with the current density decreased by three orders of magnitude, and the amount of hydrogen evolution by 94.77% compared with that of the AZ31 substrate sample.

Moreover, the MAO coatings demonstrate excellent corrosion resistance, yet several challenges restrict their ability to create superhydrophobic surfaces. The process demands high voltage and current density, while surface structure control remains limited, and there is an additional need for post-treatment steps. Due to these factors, the increased complexity and cost of the process hinder its widespread application in practical engineering fields.

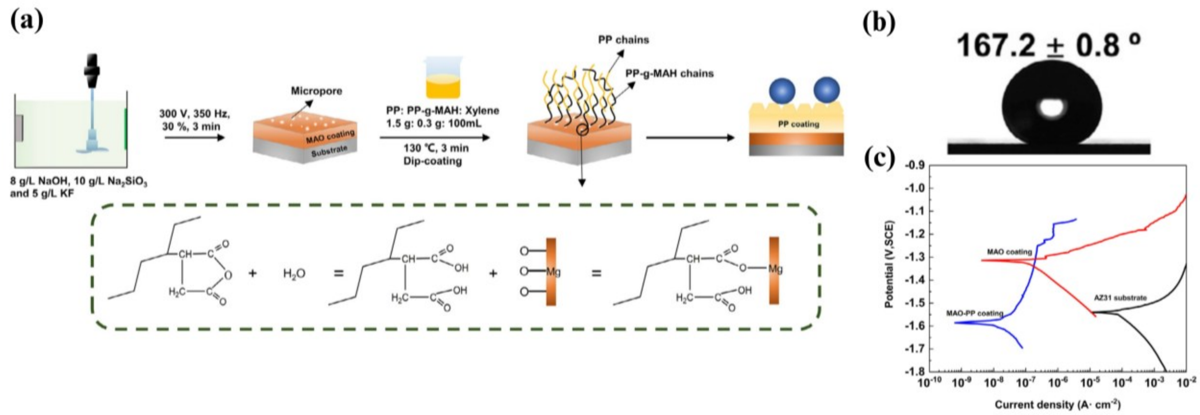

Fig. 14. (a) Flow diagram of the preparation process for superhydrophobic MAO-PP coating on AZ31 Mg alloy, (b) The CA for the MAO-PP coating, (c) Potentiodynamic polarization curves [55]

2.4. Spray method

Spray coating is a widely used technique for surface modification. Many studies highlight its potential for fabricating superhydrophobic surfaces due to easy handling, fast, low cost, environmental friendliness, and simplicity to repair by respraying. Making it an attractive option for large-scale applications. The process involves preparing a spraying solution first, then applying it to the surface, by using a spray gun, often involving heat or chemical methods to solidify the coating, and to obtain the rough micro/nanostructure that imparts superhydrophobicity. One of the key advantages of this technique is that damaged surfaces can be repaired easily by respraying, improving durability and reducing maintenance costs. [25, 32, 57-59].

Lingjie Li et al. [60], as shown in Fig. 15, developed a superhydrophobic coating on AZ31 magnesium alloy using a one-step spraying method, by preparing a solution containing 2.0g of polymethyl methacrylate (PMMA) in 20 ml of acetone, followed by the addition of 2.0g of SA-modified ZnO nanoparticles. It is synthesized by dispersing 0.5g of SA with 0.9 g of ZnO nanoparticles in 12 mL of ethanol. 1.0 ml of the prepared suspension was then sprayed onto the sample surface and dried at 60°C for 2 hours. The results showed that the coated surface had a water contact angle of 157° and a sliding angle of 6°, which shows its excellent hydrophobic properties. It also had good corrosion protection, soiling resistance, and thermal insulation. The coating also showed mechanical and chemical stability under harsh conditions, which makes it suitable for industrial applications requiring durable and efficient surfaces under harsh operating conditions.

Shidong Wang et al. [61], as shown in Fig. 16, prepared a superhydrophobic coating on AZ31 magnesium alloy using a one-step spray technique, using silica-coated polydopamine (PDA@SiO_2) particles and silane. The modified SiO_2 particles were prepared by mixing ($NH_3{\cdot}H_2O$) with ethanol, followed by dropwise addition of tetraethylorthosilicate (TEOS) and continuous stirring for several hours. DA-HMDS particles were then added sequentially, and the mixture was left to hydrolyze for 48 hours. The particles were then filtered and dried. A silane solution was then prepared by mixing diethoxydimethylsilane (DEDMS) and triethoxymethylsilane (MTES) particles with ethanol and water, using acetic acid to adjust the pH. The SiO_2 particles were then added and ultrasonicated. The mixture was then reacted at 60°C for 6 hours under magnetic stirring. The mixture was distributed evenly across the alloy surface through a spray gun, which operated at 0.2MPa pressure from a distance of 15cm. The hot plate was maintained at 120°C during the spraying operation to speed up ethanol evaporation. The sample underwent oven drying at 100 °C for one hour. The coating exhibited a contact angle (CA) above 154°, a sliding angle (SA) below 3, and excellent corrosion resistance. The coating continued to protect the sample surface from corrosion after 15 days of immersion in a sodium chloride solution, demonstrating its high stability in corrosive environments. This is due to the coating's coarse microstructures, which enhance superhydrophobicity. This coating can be relied upon as an effective method for improving the stability and durability of magnesium surfaces in corrosive environments.

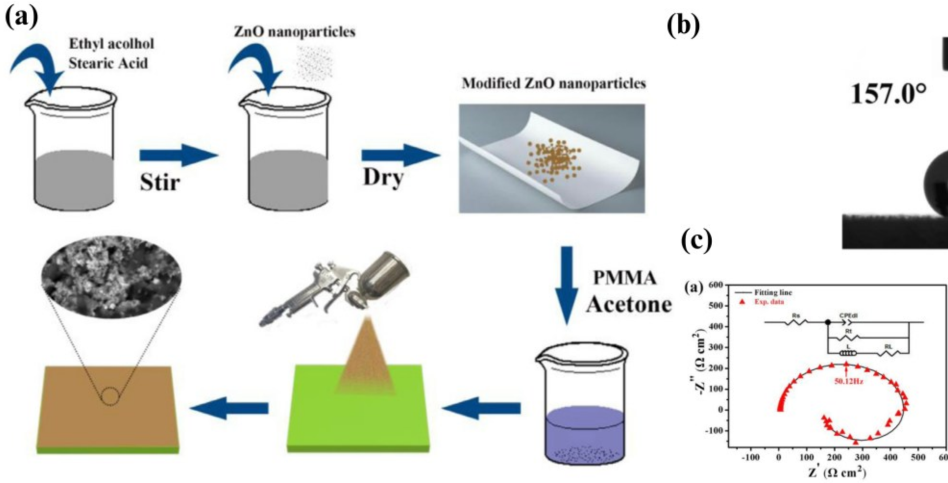

Fig. 15. (a) Schematic illustration of the fabricating process, (b) CA of sprayed AZ31 Mg, (c) Nyquist plots of bare and as-sprayed AZ31 Mg sample [60]

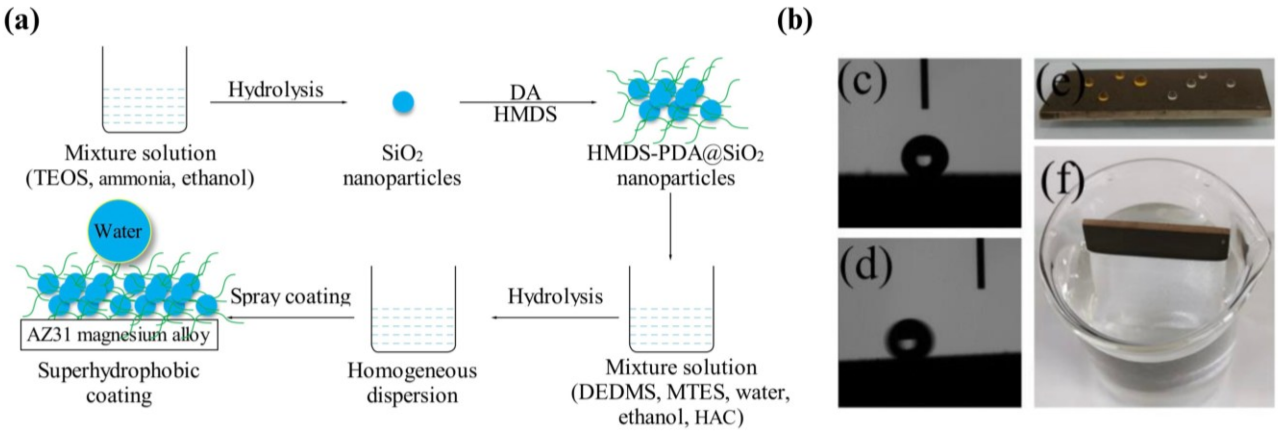

Fig. 16. (a) Schematic diagram of the fabrication process of superhydrophobic coating, (b, c, d, e, f) The CA and SA, droplets of methyl orange solution and water on coated magnesium alloy, and coated magnesium alloy immersed in deionized water [61]

Huimin Zhou et al. [62], as shown in Fig. 17, mimicked the planting grass process to create a ZnO/ epoxy resin coating with micro-nano structure by using a spray method, then modified the surface with stearic acid to form a superhydrophobic coating on the surface of AZ31 magnesium alloy. The ZnO seeds/epoxy coating was prepared by mixing ZnO nanoparticles with different weights with 0.3 g of epoxy resin, respectively. These different proportions of the mixture were added to 20 mL of ethanol and stirred for 30 min. The mixed solution was sprayed with a spray gun at a distance of about 10 cm from the magnesium alloy, and the plate was then placed in an oven at 80 °C to solidify. The superhydrophobic coating was prepared by using a solution containing ($Zn(NO_3)_2\ 6H_2O$) with an appropriate amount of hexamethylenetetramine in 40 ml of deionized water, which was then stirred for 30 minutes. The ZnO seeds/epoxy-coated alloy was placed in the growth solution inside a Teflon-lined vessel for 5 hours at 100°C; then the sample was washed and dried. Finally, the coating was chemically modified with a stearic acid solution in ethanol to achieve superhydrophobic properties. The ZnO/epoxy resin coating exhibited a contact angle up to 163°. This is due to the presence of a fine nanostructure, where the interlocking ZnO rods enable the coating to form multiple air layers, thus inducing the droplets to transform into the Cassie-Baxter state. The coating is also robust and has a high bonding force with the substrate, due to the presence of the epoxy resin, which has strong adhesion. In addition, this coating exhibits high corrosion resistance, chemical stability, and self-cleaning properties, which make this system suitable for demanding industrial applications that require high resistance to wear and friction.

Nevertheless, this technique may require specific equipment, and uneven coating distribution can occur if not properly calibrated, leading to uneven coating thickness or surface defects.

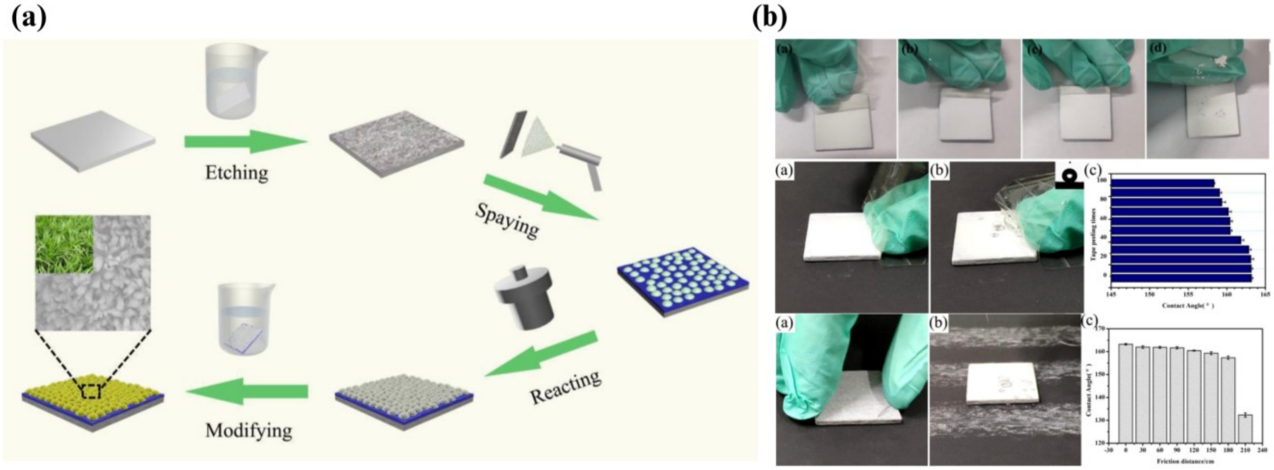

Fig. 17. (a) Schematic illustration of the preparation process of the cluster-like ZnO, (b) The different mass ratios of ZnO to epoxy resin under the growth, Tape peeling test, and Sandpaper abrasion test [62]

2.5. Chemical etching coating

The fabrication of micro/nanostructured surfaces through chemical etching offers an economical and straightforward method that operates without requiring sophisticated equipment, controlled environments, and complex procedures. The process occurs through the removal of the metal surface's thin layer by acidic or alkaline solutions, which trigger redox reactions to produce a rough surface texture. This surface can then be treated by using low-surface-energy compounds to achieve superhydrophobic properties and improved corrosion resistance. The technique shows exceptional effectiveness for modifying alloys such as AZ31 magnesium to produce a super-hydrophobic surface with easy-to-operate, control, low-cost, time-saving, and the ability to form a rough surface structure [44-47].

The researchers Leoš Doskoˇcil et al. [63], as shown in Fig. 18, presented a super aversional coating of water on the surface of the magnesium alloy AZ31 and AZ91, using a simple two-step method, including chemical etching, and then stearic acid modification. The first layer was prepared by using two different chemical etching solutions to create a micro/nan-structured surface. This was accomplished by placing the alloy samples in a 0.25 M $ZnCl_2$ solution or a 0.25 M $SnCl_2$ solution for 2 and 10 minutes, respectively. Then, the samples were rinsed with deionized water and oven-dried at 50°C for 20 minutes. A superhydrophobic surface was then prepared by adding 0.05 M stearic acid to ethanol at 50 or 60°C for different times. The optimal chemical etching time of 10 minutes was chosen for both materials. The alloys etched with ZnCl2 and SnCl2 exhibited rough surfaces with microscopic and nanoscale hierarchical structures consisting of two distinct chemical regions ($Zn/Zn(OH)_2$ or Sn/SnO_2 and $Mg(OH)_2$). The optimum superhydrophobic surfaces were prepared at 50°C for 4 hours, resulting in a superhydrophobic surface with the highest contact angle of about 151.3 ° for AZ31 alloy etched with ZnCl2 and 152.0 ° for AZ91 alloy etched with $SnCl_2$ solution. This leads to improved corrosion resistance compared to bare alloys. This indicates that the chemical etching process affects the microstructure of magnesium alloys to form superhydrophobic surfaces.

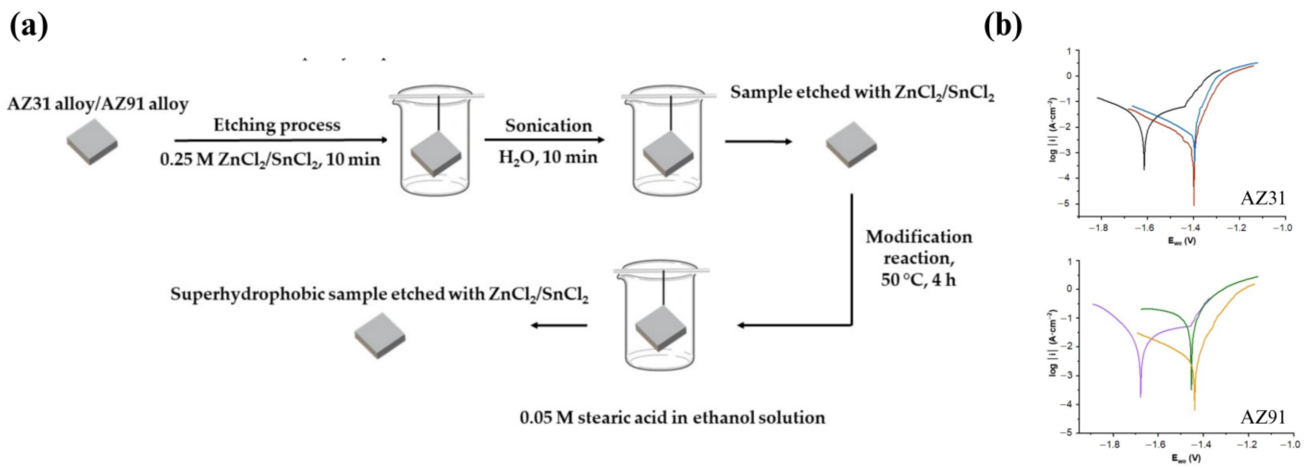

Fig. 18. (a) Schematic illustration of the superhydrophobic surface preparation process on AZ31 and AZ91 alloys etched with ZnCl2 and $SnCl_2$ under optimal conditions, (b) Potentiodynamic polarization curves [63]

Xing Han et al. [64] easily prepared a superhydrophobic anticorrosion film on the etched AZ31 magnesium alloy via in situ growth of layered double hydroxides (LDHs), followed by modification with 1H, 1H, 2H, 2H-perfluorooctyltrimethoxysilane (PFOTMS). The coating was prepared by first forming a rough micro-nano structure on the surface of AZ31 magnesium alloy, through removing the oxide layer by etching the dried substrates in 6.5 wt% nitric acid for 20 s at room temperature. After this step, MgAl-LDH films were prepared on a chemically etched substrate by hydrothermal treatment using a solution containing $Al(NO_3)_3·9H_2O$ (0.01 mol) and $NaNO_3$ (0.005 mol) dissolved in deionized water at pH 10.7 by adding NaOH solution. Hydrothermal treatment was then carried out at 125 °C for different periods (6, 12, 18 h) or at various temperatures (100 °C, 125 °C, 150 °C) for 12 h. Finally, the pre-prepared samples were cleaned with deionized water and ethanol and dried with a warm air stream. The superhydrophobic surface was then fabricated on the MgAl-LDH membrane by immersing it in a solution containing 2 mL of PFOTMS and 100 mL of ethanol at 60°C for 1 h in an oven. After immersion, the samples were dried at 60°C for 1 h in an oven. The results showed the best CA value of 163°, with high corrosion resistance, as the samples showed a current density about four orders of magnitude lower than that of the AZ31 substrate, due to the dual protection derived from the LDH films and their superhydrophobic properties. Furthermore, the coating was able to maintain a contact angle above 140° after immersion in 3.5% NaCl. Solution for 6 days. This indicates the good stability of the super-hydrophobic LDH film. This is due to the micro/nano hierarchical surface morphology of the movie, which consists of island structures obtained after chemical etching and in situ grown MgAl-LDH nanowalls.

M. Yeganeh et al. [65], as shown in Fig. 19, studied the formation of a superhydrophobic (SHP) structure on the surface of an AZ31 magnesium alloy by two steps: chemical etching in $CuCl_2$ and $NiSO_4$ solutions, followed by modification with stearic acid (SA) solution. The samples were first immersed in a 3.4 wt.% $CuCl_2$ solution in water at 90°C for 5 minutes, followed by immersing them in a $NiSO_4$ solution (0.8 g/100 ml water) at 80°C for 10 minutes. To create a rough surface, the samples obtained from the previous step were then immersed in a $CH_3(CH_2)_{16}COOH$ solution for 4 hours at room temperature. Finally, the samples were dried at 120°C for 15 minutes to obtain a superhydrophobic surface. The results showed a contact angle of about 151.5°, with the values of corrosion resistance (R_{ct}) regarding SHP Mg being at least three orders of magnitude higher than that of the bare Mg alloy, due to the presence of a resistant phase of NiO and a superhydrophobic tail of stearic species on the surface. However, the chemical etching method requires precise control of reaction conditions to prevent corrosion and unwanted surface modifications. The technique also results in waste metal ions, which waste resources and pose environmental hazards.

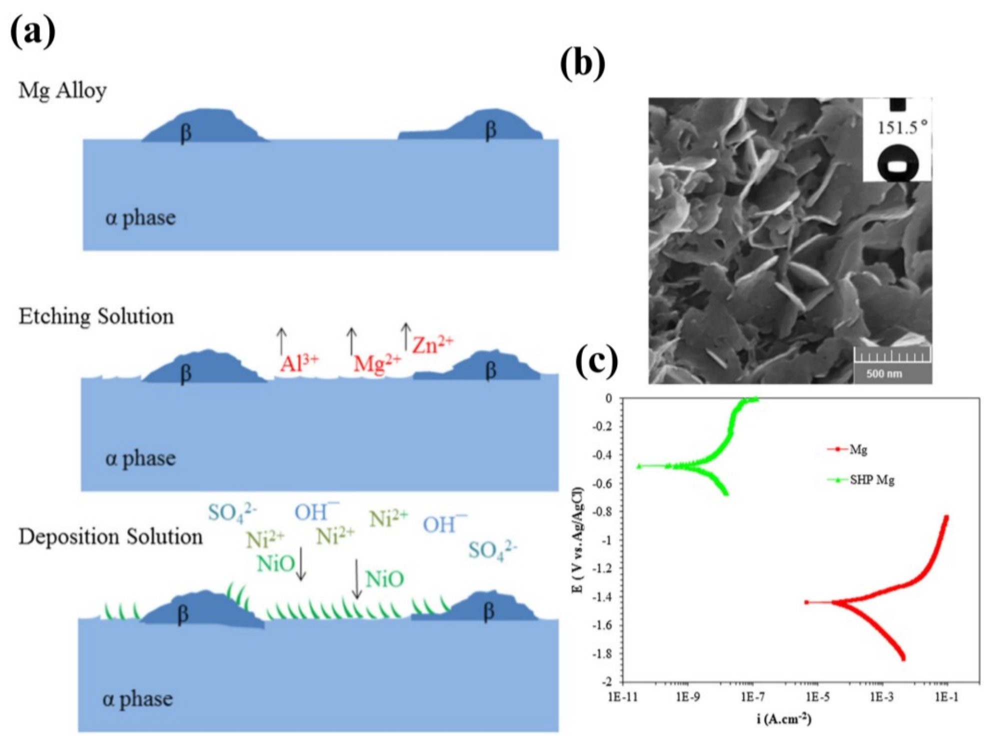

Fig. 19. (a) Schematic process to make a superhydrophobic surface, (b) Surface morphology of SHP Mg with high magnification, with contact angle, (c) Potentiodynamic polarization curves [65]

2.6. Laser processing

In recent years, laser technology has garnered significant attention from researchers to create superhydrophobic surfaces as a contemporary, effective method because it provides precise control over the structure with rapid execution and reduces reliance on chemical treatment. The most prominent types of lasers used include femtosecond lasers (10-15s) and nanosecond lasers, as well as laser texturing systems that use chemical treatment [27, 44, 66-68].

Wei Xiong et al. [69], as shown in Fig. 20, the surface of an AZ31 magnesium alloy was endowed with superhydrophobic properties by a simple, effective laser-chemical surface treatment method combining laser surface structuring and stearic immersion treatment, to modify and control wettability, improving its anti-corrosion and anti-bacterial properties. The laser–chemical surface treatment involved two steps. The first step is using a laser with a 355 nm UV from an MQ5T, Mac Laser marking machine to create a cross-hatch pattern with a 50 µm focal spot. The second step involved immersing the laser-structured specimen in a stearic acid/ethanol solution (0.05 mol/L, 1:350 molar ratio) at 60°C for 10 minutes. The specimen underwent ethanol cleaning followed by air drying after immersion. The research examined three Mg alloy samples, which included untreated samples and samples treated with laser structuring and laser–chemical treatment. The results showed that the laser-induced dual-scale micro/nanostructures, which were modified by stearic acid, exhibited a superhydrophobic surface, with a water CA of about 160°. In addition, significantly enhanced corrosion resistance, stability in corrosion medium, and antibacterial properties up to 82.05% at a rate.

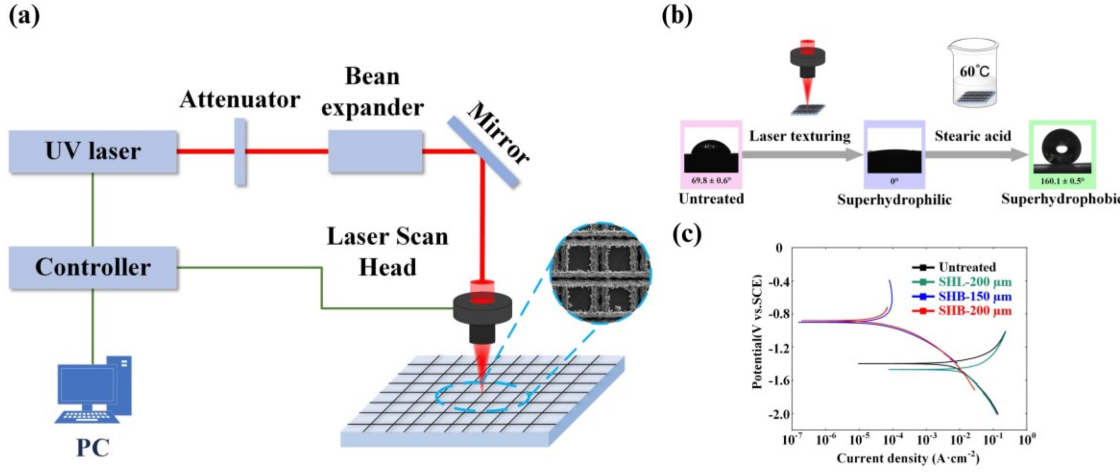

Fig. 20. (a) Process schematic of laser–chemical treatment, (b) Evolution of surface wettability of a specimen after each treatment, (c) PDP curves [69]

Zhe Wu et al. [66], as shown in Fig. 21, proposed a new method to prepare a superhydrophobic surface to effectively enhance corrosion resistance on AZ31B magnesium alloy, based on laser processing and chemical treatment by stearic acid using pulse laser chemistry. This process includes etching 1.5 mm, which was performed using a Nanosecond pulse laser (JW-F30W) to create micro-nanostructures on the surface. After etching, the samples were immersed in stearic acid for 4–6 hours to reduce the surface energy. During this process, the effect of changing laser power, etching speed, and line spacing on the final properties was studied, in addition to the impact of the laser path shape on the surface structure. The results showed that laser etching alone made the surface superhydrophobic. Still, when followed by chemical treatment with stearic acid, the surface transformed into superhydrophobic, with a contact angle of 154.2° and a sliding angle of 8.9°. The study demonstrated that the optimal parameters to achieve this behavior were: a laser power of 10 W, a frequency of 150 kHz, and a spacing of 60 µm between etching lines. The study demonstrated that laser surface structure modification combined with stearic acid treatment provides a practical, low-cost, and environmentally friendly method for producing superhydrophobic surfaces with high corrosion resistance on magnesium alloys.

Dawei Li et al. [70], as shown in Fig. 22, have made the fabrication of superhydrophobic AZ31 magnesium alloy surfaces possible through laser ablation followed by chemical etching and surface modification techniques. The fiber laser system operated at 20 W with 1064 nm wavelength and 20 µm beam spot diameter to create papillary-like micro-pits with adjustable center distances between 80–250 µm and diameters ranging from 40–100 µm. The laser processing parameters included 50% power, 500 mm/s scan speed, 20 kHz repetition frequency, and 100 ns pulse width. The laser-ablated samples underwent 0.1 mol/L $AgNO_3$ etching for 3 minutes, followed by 0.15 mol/L stearic acid ethanol solution modification at room temperature for 1 hour. The treated surfaces developed superhydrophobic characteristics with a maximum water contact angle reaching 158.2°. The electrochemical tests revealed substantial corrosion protection because the corrosion potential shifted positively while the corrosion current density (I_{corr}) decreased by nearly one order of magnitude compared to untreated AZ31. The fabricated surfaces demonstrated both superhydrophobicity throughout a pH range of 4–14 while enduring different temperatures and one month of air exposure. The method presents a practical solution for improving magnesium alloy corrosion resistance while allowing scalability for industrial applications that need adjustable surface wettability and durability.

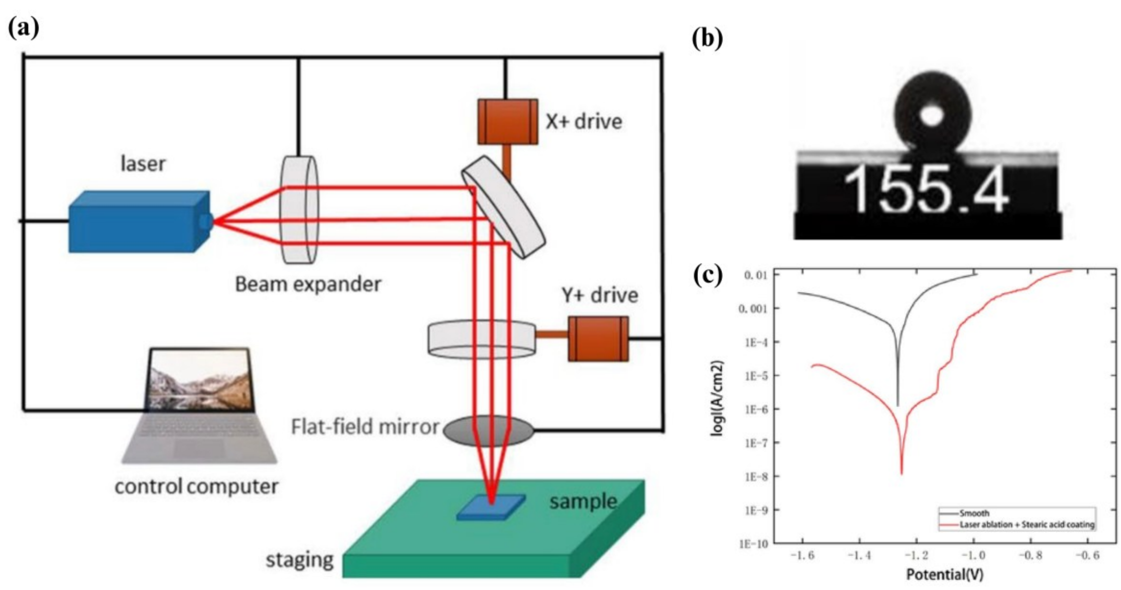

Fig. 21. (a) Laser schematic diagram, (b) The CA of coating, (c) Polarization curve fitting [66]

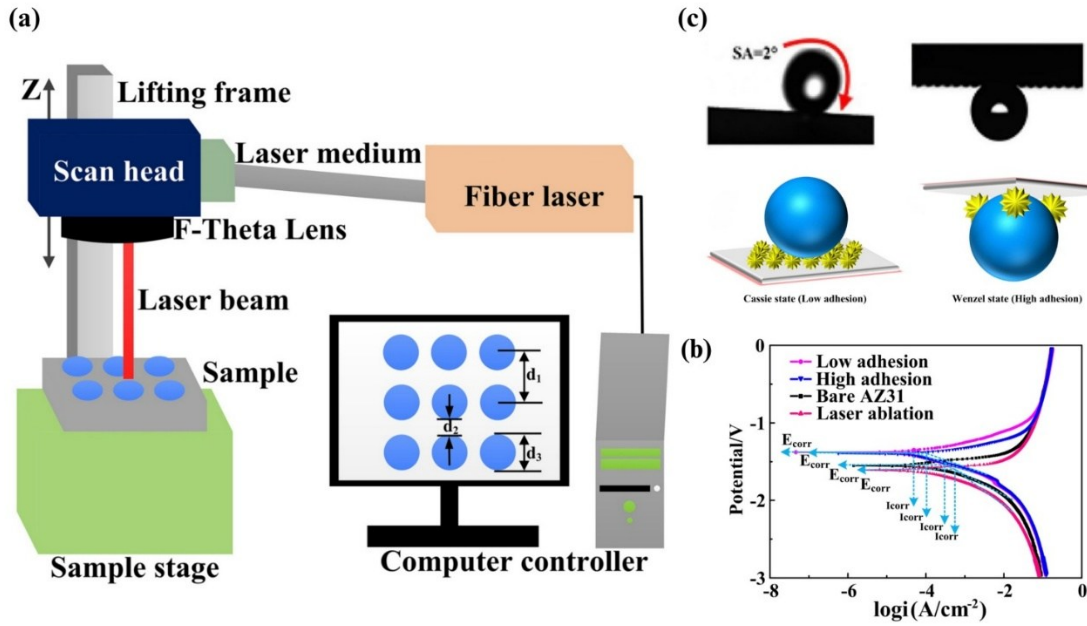

Fig. 22. (a) Schematics of the fabrication process for the superhydrophobic surface on AZ31 alloys, (b) The CA of coating, (c) Polarization curves [70]

In the previous part, summarized the preparation strategy and protection mechanism of superhydrophobic coatings to abate the corrosion behavior of AZ31 alloys in Table 2 [2, 26, 50-56, 60-66, 69, 70]. These methods include electrodeposition, spraying, hydrothermal treatment, chemical etching, and laser-based techniques. Each method offers advantages such as simple equipment, high processing efficiency, and cost-effectiveness. However, several of these techniques face limitations, especially concerning the weak interfacial bonding between the coating and the magnesium substrate, which affects long-term stability.

Table 2. Summary of research about the corrosion resistance of superhydrophobic coating on AZ31 alloys by various preparation strategies

Method	Surface energy reducer agent	CA°	i_{corr} (A.cm^{-2})		Ref.
			Substrates	Coatings	
Dipping + Electrodeposition	Myristic acid	159°	3.36×10^{-5}	1.86×10^{-8}	[50]
Hydrothermal + Electrodeposition	(DTMS)	165°	7.34×10^{-5}	1.77×10^{-8}	[26]
Electrodeposition	Myristic acid	165°	2.73×10^{-4}	8.60×10^{-8}	[51]
Hydrothermal	Myristic acid	159°	6.92×10^{2}	1.63×10^{-3}	[52]
Hydrothermal	Stearic acid	159°	1.62×10^{-5}	1.72×10^{-7}	[2]
Hydrothermal	PP+PP-g-MAH	165°	6.15×10^{-5}	3.12×10^{-9}	[53]
MAO + Hydrothermal	(OTES)	155°	2.78×10^{-4}	4.1×10^{-10}	[54]
MAO + Dipping	PP-g-MAH	167°	7.79×10^{-5}	8.76×10^{-9}	[55]
MAO	Stearic acid	156°	4.21×10^{-4}	2.35×10^{-7}	[56]
Spraying	Stearic acid	157°	-	-	[60]
Spraying	Stearic acid	154°	-	-	[61]
Spraying	(DEDMS) + (MTES)	163°	-	-	[62]
Chemical etching	Stearic acid	151°	26.51×10^{-5}	3.41×10^{-5}	[63]
Chemical etching	(PFOTMS)	163°	6.19×10^{-5}	7.85×10^{-7}	[64]
Chemical etching	Stearic acid	151°	82.70×10^{-5}	1.47×10^{-9}	[65]
Laser-chemical treatment	Stearic acid	160°	1.50×10^{-2}	8.30×10^{-5}	[69]
Laser-chemical treatment	Stearic acid	155°	3.20×10^{-4}	8.71×10^{-7}	[66]
Laser-chemical etching	Stearic acid	158°	5.63×10^{-4}	6.55×10^{-5}	[70]

3. Conclusion

This review has summarized the main fabrication techniques used to produce superhydrophobic coatings on magnesium alloys (AZ31), including electrodeposition, spraying, hydrothermal methods, chemical etching, laser treatment, and micro-arc oxidation. These approaches generally offer advantages such as operational simplicity, cost-effectiveness, and the potential for scalability. In particular, laser-based methods and combined laser–chemical treatments have demonstrated the ability to construct well-defined surface structures that enhance water repellency and corrosion resistance. Despite these advances, the development of new techniques has not eliminated the obstacles that prevent long-term durability and strong interfacial adhesion between coatings and magnesium substrates. The improved bonding achieved through micro-arc oxidation methods requires complex procedures and high energy usage. The majority of laboratory-developed coatings remain restricted from industrial application because of their unstable performance characteristics. Future research should focus on improving coating durability while optimizing process parameters and integrating multifunctional properties to ensure reliable performance in practical applications. Given the simplicity and effectiveness of superhydrophobic surface design, these strategies are expected to play an increasingly important role in magnesium alloy protection.

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