Research Progress on Corrosion Resistance of Magnesium Alloys with Bio-inspired Water-repellent Properties: A Review

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Abstract

Thanks to its excellent mechanical properties, magnesium alloys have many potential applications in the aerospace and other fields. However, failure to adequately solve corrosion problems of magnesium alloy becomes one of the factors restricting its wide use in many industrial fields. Inspired by nature, researchers designed and fabricated bio-inspired water-repellent (superhydrophobic and slippery liquid-infused porous surface) surfaces with special wetting properties by exploring the surface microstructures of plants and animals such as lotus leaf and nepenthes pitcher, exhibiting excellent corrosion-resistant performance. This article summarizes the research progress on corrosion resistance of magnesium alloys with bio-inspired water-repellent properties in recent years. It mainly introduces the corrosion reasons, types of corrosion of magnesium alloys, and the preparation of magnesium alloys with bio-inspired water-repellent and anti-corrosion coating on the surface of magnesium alloy by surface treatment. It is hoped that the research in this review can broaden the application range of magnesium alloys and provide a powerful reference for the future research on corrosion resistance of magnesium alloys.

Keywords: magnesium alloy, bio-inspired water-repellent, corrosion resistance, superhydrophobic, slippery liquid-infused porous surface, research progress

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1 Introduction

Magnesium alloy has the advantages of light weight, high specific strength and specific rigidity, good shock absorption performance, excellent electrical and thermal conductivity, and is easy to cut and renewable^[1-6]. It is recognized as the most promising lightweight and green engineering material, and has many potential applications and increasingly positive development prospects in the fields of aerospace, transportation, electronic devices, biomedicine, and our daily life^[7-11]. Magnesium and its alloys are expected to become alternative materials for other traditional metals by virtue of abundant resources and many excellent properties^[12-15].

The positive prospect of magnesium alloy as a structural material, however, is in distinct contrast to the situation it has been facing nowadays^[16]. One of the reasons is the corrosion problem of magnesium^[17,18]. As a matter of fact, the chemical properties of magnesium are very active, and its standard electrode potential is

-2.37 V, which is lower than that of Fe, Zn, Al and other metal elements^[19,20]. It is very easy to form galvanic corrosion with impurity elements or the second phase^[21]. Moreover, magnesium alloy products are also easily oxidized during processing and use^[22–24], and thus the loose and porous surface oxide film is difficult to form stable and effective protection for the alloy^[25,26]. It is obvious that poor corrosion resistance has become a bottleneck restricting the potential of magnesium alloys^[27]. Therefore, improving the corrosion resistance of magnesium alloys is not only extremely important in practical applications but can offer significant economic returns^[28,29].

At present, the main methods to improve the corrosion resistance and extend the service life of magnesium alloys include micro-alloying^[30–32], microstructure control^[33,34], surface treatment^[35,36] and preparation of functional coatings^[37]. The widely used and effective method is to construct a coating with anti-corrosion performance on the surface of magnesium alloy through surface treatment^[38,39]. Bio-inspired water-repellent

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surfaces with its unique interface characteristics and advantages have become a new idea to solve the corrosion problems of metal materials^[40,41]. The bio-inspired water-repellent surfaces introduced in this article are divided into superhydrophobic surfaces and Slippery Liquid-Infused Porous Surfaces (SLIPSs)^[42,43]. The fabrication of magnesium-based bio-inspired water-repellent surface helps to construct a function on the substrate of magnesium alloy, improving the corrosion resistance of magnesium alloy in an effective manner^[44–46]. The fabricated bio-inspired water-repellent surfaces can effectively cut off the direct contact of corrosion medium such as humid air and erosion solution to the magnesium alloy substrate and reduce the corrosion^[47–49].

This article summarizes the research progress of corrosion resistance of magnesium alloys in five parts covering factors which affect corrosion and different types of corrosion, basic principle of wettability and practical applications of the bio-inspired water-repellent surface in corrosion resistance of magnesium alloys and highlights the development of corrosion resistance of bio-inspired water-repellent magnesium alloys as well as scientific problems.

2 Corrosion of magnesium alloy

2.1 Influencing factors of magnesium alloy corrosion

Magnesium alloys are prone to electrochemical reactions under the corrosion environment of simulated seawater. The reaction process is as follows^[50,51]:

$$Mg(s) + 2H_2O(aq) \rightarrow Mg(OH)_2(s) + H_2\uparrow, \quad (1)$$

where the electrolyte is a NaCl aqueous solution, Cl^- is a corrosive medium, and the Mg^{2+} produced by the anode reaction will react with Cl^- . The reaction equation is:

$$Mg^{2+} + 2Cl^{-} \rightarrow MgCl_{2}.$$
 (2)

In practical applications, there are many factors that affect the corrosion of magnesium alloys: alloy elements^[52–54], secondary phase size and distribution^[55,56], grain size^[57–59], crystal orientation and texture strength^[60,61], crystal defects^[62,63], and environmental factors^[64–66] (Fig. 1). The corrosion of magnesium alloys is mostly galvanic corrosion, and the α -Mg substrate is mainly corroded. Therefore, the corrosion resistance of the substrate is the key to determining the corrosion behavior of the entire alloy. Researchers analyzed the factors affecting the corrosion of magnesium alloy, and improved the corrosion resistance of magnesium alloy by a number of measures including adding alloy elements^[67–71], reducing the size of the second phase and making the distribution uniform^[72–76], refining the grains^[77–81], changing the crystal orientation and texture strength^[82–86] as well as reducing crystal defects^[87–89] (Fig. 2). Table 1 summarizes some of factors which affect the corrosion resistance of magnesium alloy.

Adding alloy elements may change the chemical components of magnesium alloy directly or indirectly and drive change in its form of organization, and the second phase size and distribution, consequently improving the corrosion resistance behavior of magnesium alloy^[90,91]. On the other hand, grain size of magnesium alloy can be reduced by alloying^[92] and plastic deformation^[93], so that mechanical properties of alloys can be improved effectively. Thus, fine grain strengthening is one of the key ways to improve the mechanical properties of magnesium alloy, and changing the grain size will create an important impact on corrosion resistance of magnesium alloy^[94,95]. The presence of alloying elements will lead to the appearance of a second phase in the magnesium alloy. The second phase is usually a compound of Mg and Al^[96,97], Zn^[98], rare earth and other metal elements^[99,100], and its electrochemical stability is higher than that of the matrix phase. Therefore, the corrosion behavior of magnesium alloys is mainly

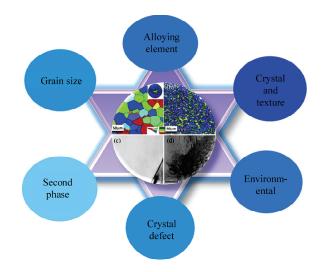


Fig. 1 Main factors influencing corrosion of magnesium alloy^[2].

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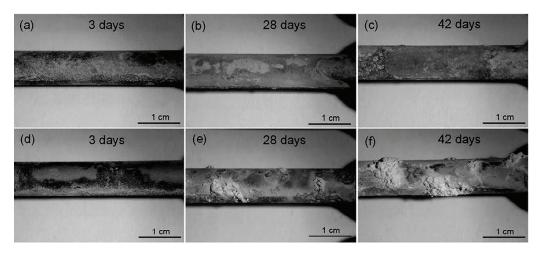


Fig. 2 Morphology of the surface of the salt spray test sample: (a - c) Mg0.5Zn0.2Ca, $(d - f) Mg0.5Zn0.2Ge^{[73]}$.

Influencing factors	Technology	Material	Functions	Corrosion types	Refs.
Alloying element	Hot-rolling	Mg-xSn-1Zn-0.5Ca	Good corrosion resistance and mechanical prop- erties	Galvanic corrosion	[67]
	Direct-chill casting	Mg-0.5Zn alloy	Mg-Zn-Ca possessed the highest corrosion resistance	Pitting corrosion	[68]
	High pressure casting technology	Mg-Ga alloy	Good corrosion resistance.	Galvanic corrosion	[69]
Second phase size and distribution	Screw rolling	Mg alloy	The corrosion rate of the magnesium alloy screw rolled at 300 °C is the lowest	Galvanic corrosion	[72]
	Casting and hot-rolling	AM60 alloy	AM60+1In alloy has the strongest corrosion resistance	Total corrosion	[73]
	Casting and extrusion	Mg-Sm-Zn-Zr alloy	The corrosion resistance is increased by 3 times.	Galvanic corrosion	[75]
Grain size	Hot-rolling	Mg-1Ca alloy	Refinement of the structure significantly reduces the corrosion rate	Local corrosion	[78]
	Rolling and annealing	AZ61 alloy	Reduced grain size and improved corrosion resistance	Galvanic corrosion	[80]
	Hot-rolling	Mg-4Li-1Ca alloy	Improved alloy strength and corrosion resistance	Total corrosion	[81]
Crystal orientation and texture strength	Rolling	Mg-5Li-1Al alloy	The corrosion resistance of the alloy is improved.	Hydrogen induced cracking	[82]
	Cut	AZ31 Mg alloy	Improve the corrosion resistance.	Hydrogen induced cracking	[85]
	Directional solidifica- tion	Mg-4wt% Zn alloy	Improved alloy corrosion resistance.	Pitting corrosion	[86]
Crystal defect	Cast and extrusion	AZ91D magnesium alloy	Improved alloy corrosion resistance.	Galvanic corrosion	[87]
	Compressive defor- mation	Mg-Y alloy	Improved the electrochemical corrosion perfor- mance.	Pitting corrosion	[88]
	Pre-stretch	AZ31 alloy	Improved corrosion resistance	Hydrogen induced cracking	[89]

Table 1	Some of	factors i	influencing	corrosion	resistance o	f magnesium a	ılloy
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micro-galvanic corrosion because of the potential difference between the second phase and the substrate^[101,102]. Although the second phase is generally not corroded, the type, content, morphology and distribution of the second phase can affect the corrosion of the magnesium matrix, so the second phase plays a vital role in the corrosion of magnesium alloys^[103,104]. However, there are also a few opinions that the increase of grain boundary area will reduce the corrosion resistance of magnesium alloy, and a small number of crystal defects inside the magnesium alloy can provide strong driving force for corrosion reaction, and hence make the surface

form a thin and dense $Mg(OH)_2$ corrosion protection film, thus delaying the corrosion process and improving the corrosion resistance^[105].

2.2 Corrosion types of magnesium alloy

The corrosion process of magnesium alloy can be classified from different perspectives. According to different corrosion environments, it can be divided into natural corrosion and industrial environment medium corrosion. By the type of corrosive media, it is divided into atmospheric corrosion, water corrosion and soil corrosion. When it comes to the mechanism of corrosion process, corrosion occurs by chemical corrosion and electrochemical corrosion. According to the type of corrosion and local corrosion. Local corrosion also includes galvanic corrosion^[106,107], pitting corrosion^[108], stress corrosion^[109–111], fatigue corrosion, intergranular corrosion and hydrogen induced cracking (Fig. 3)^[112].

Magnesium alloy is widely used as a kind of light metal structure material while poor corrosion resistance becomes its worst disadvantage. Corrosion often occurs in the hidden parts which are not easy to be detected, which will reduce the strength, plasticity and toughness of magnesium alloy, and then lead to the failure of structural materials. For instance, "catastrophic corrosion", such as bridge fracture, oil and gas pipeline explosion, will bring huge economic loss and even personal harm. It is of great significance to study the factors affecting the corrosion of magnesium alloy for controlling the corrosion behavior of alloy and effectively avoiding or reducing the occurrence of corrosion.

3 Basic principle of wettability

3.1 Theoretical study on wettability of superhydro phobic surfaces

Neinhuis^[113] and Barthlott^[114] carried out a large amount of research on water-repellent plant and found that there were micron-grade raised rough structures and wax layer on the surface of the plant. Feng *et al.*^[115] researched the "Lotus-Effect" and explored that the true secret of the superhydrophobic effect of the lotus leaf surface was the micro-nano composite structure on the lotus leaf surface. The bumps of the two sizes were compounded with each other, so that the surface of the lotus leaf had good hydrophobic property^[116]. Researchers fabricated bio-inspired water-repellent surface with superhydrophobic property by mimicking the micro-nano papilla structure of lotus leave (Fig. 4). In addition, they defined the superhydrophobic surface and identified two key factors to obtain the surface: one is to construct a complex micro/nano rough structure, and the other is to modify the surface with low surface energy materials^[117,118].

For the qualitative analysis of the wettability of liquid drops on solid surfaces, the size of contact angle (CA) of water drops is an important indicator to determine the wettability of the surface. Wettability can be divided into four types according to the CA: superhydrophilic surfaces (CA $\leq 5^{\circ}$), hydrophilic surfaces ($5^{\circ} \leq$ CA $\leq 90^{\circ}$), and hydrophobic surfaces ($90^{\circ} \leq$ CA $\leq 150^{\circ}$), and superhydrophobic surfaces (CA $\geq 150^{\circ}$)^[119–121].

The Wenzel model and Cassie-Baxter model are commonly used to analyze and explain the mechanism of solid superhydrophobic surfaces with different adhesion behaviors^[122–124], as shown in Fig. 5a. Wenzel introduced the γ dimensionless surface roughness factor as a modification of the Young's equation^[125]:

$$\cos\theta' = \frac{\gamma(\sigma_{\rm SV} - \sigma_{\rm SL})}{\sigma_{\rm LV}} = \gamma\cos\theta, \qquad (3)$$

where γ is the ratio of the actual surface area to the apparent area. As the value of γ is greater than 1, the surface roughness structure has a strengthening effect on the wettability.

The Cassie-Baxter model is similar to the wetting state of water droplets on the surface of lotus leaves in nature^[126], and exhibits a surface characteristic of low adhesion, as shown in Fig. 5b. When the surface composite contact reaches equilibrium, the applicable solid surface wetting equation is deduced from the thermo-dynamic angle^[127, 128]:

$$\cos\theta = f_1 \cos\theta_1 + f_2 \cos\theta_2, \qquad (4)$$

where f_1 and f_2 represent the area fraction of solid-liquid and liquid-gas interface contact at the solid interface, θ_1 and θ_2 represent the intrinsic contact angles of the solid-liquid and liquid-gas interface. For liquid-gas-solid three-phase compound interface equilibrium, that is, $\theta_2 = 180^\circ$, $f_1+f_2=1$, substituting into Eq. (4) can deduce:

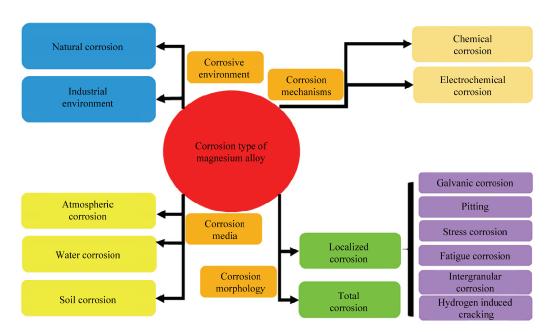


Fig. 3 Corrosion types of magnesium alloy.

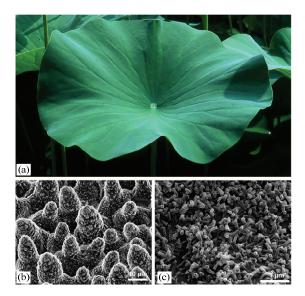


Fig. 4 (a) Superhydrophobic lotus leaf surface, (b) and (c) SEM photographs of the lotus leaf surface at different magnification^[117].

$$\cos\theta = f_1(\cos\theta_1 + 1) - 1. \tag{5}$$

When the solid surface roughness increases, it is beneficial to increase the contact area between the liquid and the air film at the liquid-solid contact interface. Some scholars have found that during the preparation of superhydrophobic surfaces of magnesium alloys, the corrosion resistance of the Cassie state (low adhesion) on the superhydrophobic surface is better than that of the

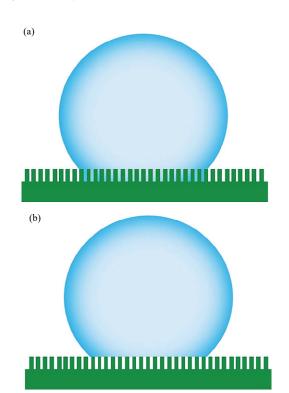


Fig. 5 Schematic diagram of wetting condition. (a) Wenzel model; (b) Cassie model.

Wenzel state (high adhesion) on the superhydrophobic surface. High-adhesion superhydrophobic surfaces have a larger contact area with liquids than low-adhesion superhydrophobic surfaces^[129]. A large amount of air present on the low-adhesion superhydrophobic surfaces

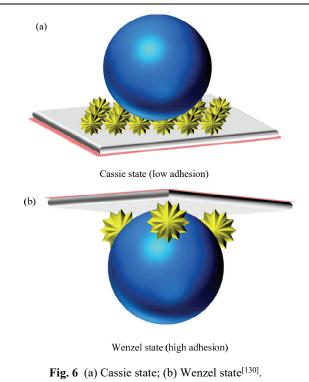
(Cassie state) acts as an air cushion, which can prevent the corrosion solution from directly eroding the magnesium alloy^[130] (Fig. 6).

3.2 Theoretical study on wettability of SLIPSs

Inspired by pitcher plants in nature (Figs. 7a–7e), bio-inspired slippery surface means the surface with certain lubrication effect achieved by lubricating liquid filling^[131]. The new bio-inspired surface has emerged in recent years and exhibited special surface wettability, so it is a derivative exploration of superhydrophobic surfaces. Wong *et al.*^[132] first proposed "SLIPS" in 2011, and gave three criteria for designing SLIPS: first, lubricating oil can penetrate into the rough structure of solid-phase substrate to wet the solid-phase substrate, and realize the solid combination of the two. Second, in order not to be replaced by other liquids, the solid phase substrate should be wetted by lubricating oil preferentially. Third, the lubricating oil and the tested liquid must not be mutually soluble (Fig. 7f).

Aiming to meet the second principle, the lubricant and solid-phase substrate must be matched in physical and chemical properties so as to form a solid working system. Moreover, the lubricating oil is not compatible with the test liquid. The surface energy of the solid-liquid interface is E_a when the test liquid thoroughly wets the solid substrate. When the test liquid floats on the top and the lubricant completely wets the solid phase substrate, the surface energy of the solid-liquid interface is E_{α} . When no test liquid floats on the top and the lubricant thoroughly wets the solid substrate, the surface energy of the solid-liquid interface is E_{β} . In order to ensure that the solid phase substrate is preferentially wetted by the lubricating oil, and the lubricating oil stored in the microstructure is not replaced by the test liquid, $\Delta E_{\alpha} = E_a - E_{\alpha} > 0$ and $\Delta E_{\beta} = E_a - E_{\beta} > 0$ must be met.

Preston *et al.*^[133] and Anand *et al.*^[134] focused on the first and second principles and analyzed the characteristics of porous substrates filled with lubricating oil with different surface energies. In summing up the failed design cases, it was found that there were five types of failures caused by the interaction between the oil layer and the test liquid (Fig. 8): first, the surface energy of the oil layer was too low, the "cloaks" phenomenon of the



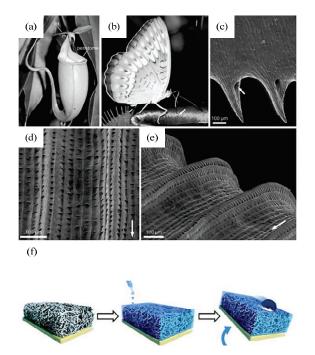


Fig. 7 (a - e) nepenthes pitcher and peristome morphology^[131]; (f) schematic illustration of fabricating the SLIPS^[132].

wrapped test liquid occurred, resulting in the gradual loss of oil layer. Second, the surface energy of the lubricating oil was high, and the test liquid could not condense into droplets and slide down. Third, the oil layer failed to completely wet the rough substrate

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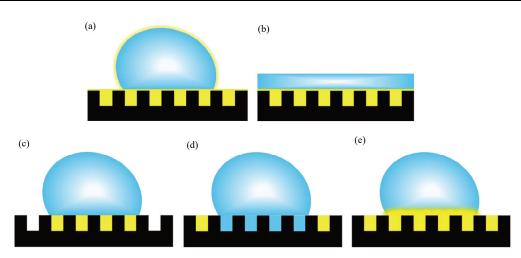


Fig. 8 Five failure types of SLIPS surface: (a) cloak phenomenon; (b) external media spread on SLIPS surface; (c) partial infusion of lubricant into porous substrate; (d) penetration of external liquid into substrate; (e) miscibility of lubricant and external liquid^[133].

surface. Fourth, part of the oil layer was replaced by the test liquid. Fifth, the oil layer was miscible with the test liquid. In short, the SLIPS design must satisfy the following 5 formulas:

The oil layer will not "cloak" the test liquid:

$$S_{\rm ol(v)} = \gamma_{\rm lv} - \gamma_{\rm lo} - \gamma_{\rm ov} < 0. \tag{6}$$

The test liquid cannot be completely spread on the surface of the oil layer:

$$S_{\rm lo(v)} = \gamma_{\rm ov} - \gamma_{\rm lo} - \gamma_{\rm lv} < 0.$$
 (7)

The oil layer can completely wet the substrate:

$$S_{\rm os(v)} = \gamma_{\rm sv} - \gamma_{\rm os} - \gamma_{\rm ov} > -\gamma_{\rm ov} R. \tag{8}$$

The oil layer can still spread on the surface of the substrate in the test liquid environment:

$$S_{\rm os(l)} = \gamma_{\rm ls} - \gamma_{\rm os} - \gamma_{\rm lo} > -\gamma_{\rm lo} R.$$
(9)

The oil layer and the test liquid are not miscible:

$$\gamma_{10} > 0, \tag{10}$$

where S is the spreading coefficient, γ is the surface tension, s, o, v, l are the solid substrate, lubricating oil, gas environment and test liquid respectively, R is the roughness index.

4 Preparation technology of magnesium-based bio-inspired superhydrophobic surface

Bio-inspired superhydrophobic surfaces have

gradually become a new idea to solve the problem of poor corrosion resistance of metal materials due to their unique interface characteristics and advantages^[135-137]. The existence of superhydrophobic coating can effectively cut off the direct contact of corrosive media such as humid air and corrosive solution to the magnesium alloy substrate, thereby improving the corrosion resistance of magnesium alloy and expanding its applications in the industrial field. In the past decades, researchers have proposed various methods to prepare magnesium alloys with superhydrophobic property, such as hydrothermal method^[138,139], laser processing method^[130,140], micro-arc oxidation^[141-143], electrochemical deposition^[144–148], anodic oxidation^[149–151], dipping method^[152-154], chemical etching^[155,156], and solution deposition method^[157-159] (Fig. 9). Table 2 summarizes the innovative technologies applied to the surface processing of magnesium alloys.

4.1 Hydrothermal method

Hydrothermal treatment is achieved by placing the precursor in an autoclave and making it react at high temperature and pressure conditions. The equipment required for this treatment is simple and easy to operate, and the energy-efficient and low-cost process can occur at high temperature and pressure conditions and be used in a wide range of applications. Besides, the nanoscale materials treated by this process exhibit high purity, crystallinity and dispersibility and are controllable in morphology^[192]. Li *et al.*^[193] prepared a

Table 2 Summary on the processing methods of superhydrophobic magnesium alloys for corrosion resistance	;

Technique	Advantage/disadvantage	Material	Morphology	CA	SA	Refs.
		Wrought magnesium alloy MA8	Nanocomposite layer with multimodal roughness	≥160°	-	[141]
		Mg-Li alloy	Porous oxide coating	$163^{\circ} \pm 2^{\circ}$	-	[142]
	Clean and pollution-free, simple process, uniform and hard film layer,	MA8 alloy	Nanocomposite layer with multimodal	$166.0^\circ \pm$	$5.2^{\circ} \pm$	[143]
Micro-arc oxidation	high adhesion/porous surface, elec-	2	roughness	3.0°	3.3°	
	trolyte is easy to penetrate, requiring composite packaging treatment	Mg-Mn-Ce alloy	Multi-scale coarse structure	153.7°±3.2°	7°	[129]
	1 1 8 8	AZ31 alloy	Flake-like structure Pores, island-structure and volcano-like	151.21°	-	[160]
		AZ31 alloy	structure	151.5°	-	[161]
		AZ31B alloy	Intersected lamellar magnesium hy- droxide	159°	-	[162]
method ditions, not suitab	Coating structure is dense/poor con- ditions, not suitable for large-scale production preparation	ZK60 alloy	Mammillaria-herrerae-like micro- spheres, carnation-like microclusters and carnation-petal-like nano-slices with hierarchical biomimetic mi- cro/nanostructures	158.5°	2°	[163]
		AZ31 alloy	Curved hexagonal platelets	163.7°±2.9°	<1°	[164]
		AZ31B Mg alloy	Clinochrysotile-like magnesium silicate nanotubes	155°	5°	[165]
		Magnesium alloy AZ31	Core/shell structure	153°	-	[152]
	Simple operation, high efficiency,	AZ31B alloy	Nano-sheeted structure	167.3°±2.1°	$\begin{array}{c} 2.7^\circ \pm \\ 0.8^\circ \end{array}$	[153]
Dipping method	large-scale preparation/ environmen- tal pollution, high cost of waste liquid treatment	AZ31 alloy	Nano- and microsheets	158°±2°	-	[154]
11 8		AZ31 alloy	White and bulges spots	162°±3.4°	$5^\circ\pm0.6^\circ$	[166]
		AZ31 alloy	Nanosheets	153°±2°	7°	[167]
		AZ31 alloy	Rough petal-like, micro/nanoscale structure	160°	-	[168]
		AZ61 Mg	Network structure	156.8°	-	[169]
Lar	Large-area preparation, suitable for a	AZ31B alloy	Micrometer and nanometer scale binary structures	155°	-	[170]
Spraying method	variety of substrates, simple opera- tion, low cost/uneven coating	AZ31B Mg alloy	Irregular structures with accumulated SiO ₂ nanoparticles	151.8°	5.1°	[171]
		AZ31 magnesium alloy	Micron scale mastoid structure	161°	4°	[172]
		NZ30K alloy	Silica film	151°	-	[173]
	Excellent film performance, easy to control/requires multiple processes, the process is complex and cannot be generated in large quantities	AZ31 alloy	Micron-sized spherical structure	$165.5^\circ \pm 3.6^\circ$	$4^{\circ}\pm0.6^{\circ}$	[174]
Composite		AZ91 alloy	Micro-nano hierarchical structure	159°	-	[175]
		AZ31 alloy	Flower cluster-like	163°	-	[176]
method		AZ31 magnesium alloy	Carbon nanofiber	145°-150°	$7^\circ\pm2^\circ$	[177]
		AZ61 magnesium alloy	Nodular structure	156.5°	2°	[178]
		Mg alloy AZ61	Nanometer scale cavities and mastoids	165.2°	2°	[179]
	Fast, large scale, low cost, easy to control, low energy consump- tion/weak substrate binding, heavy metal pollution, and harsh conditions	AZ91D alloy	Hierarchical flowerlike structures	167.3°±1.3°	1°	[180]
Electrochemical		Mg-Sn-Zn (TZ51) alloy	Uniformly micro/nano binary strip and flower-like structure	160.4°±0.7°	-	[181]
deposition		Mg alloy	Flower-like	154.3°±3.1°	$5.6^{\circ}\pm0.8^{\circ}$	[182]
		AZ31 alloy	Micro-nano hierarchical structures	156.6°	-	[183]
		AZ31 Mg alloy	Some spherical clusters in micrometer scale	155.2°±1.5°	6.0°±0.5°	[184]
Chemical etch-	Simple equipment, easy opera- tion/pollution of the environment, high cost of waste liquid treatment	AZ31B alloy	Micro-flowers	152.65°	5°	[155]
		Mg-Li alloy	Peonylike micronanoscale hierarchical structures	160°	<5°	[156]
ing		AZ31 alloy	Nano-vertical plate pattern	152°	-	[185]
		AZ31 alloy	Nubby cluster with binary mi- cro-nanometer scale	142°	5°	[186]
Laser pro-	High precision, stable structure, good	AZ31 alloy	Micro-scale papillary-like pits	158.2°	-	[130]
Laser pro- cessing	surface quality/high cost, low effi- ciency, not large-scale production	Mg-9Al-1Zn alloy	Wavy surface morphology	158.8°±2°	-	[140]

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	One-time film formation, strong adhesion, excellent abrasion resistance. low	TZ51 magnesium alloy	Pits and crack	163°	-	[149]
Anodic oxidation	cost/environmental pollution, large brit- tleness, porous, difficult to process com-	AZ31 Mg alloy	Micro- and nano-scale binary structures	153°	-	[150]
	plex workpieces	AZ91D alloy	Filamentous structure	>150°	-	[151]
		AZ31 alloy	Plate structure	150.5°	-	[157]
Solution deposi- tion	Easy operation, low cost, large-scale preparation/polluting the environment, high cost of waste liquid treatment	AZ31 alloy	Flake-like morphology with nano-sized thick- ness	151.5°	-	[158]
	ingli cost of maste inquite inclinent	AZ31 alloy	Foamed tremella	153°	4°	[159]
	Simple operation, low cost/environmental	AZ31B alloy	Island-like platforms	157°	-	[187]
Conversion coating	pollution, high cost of waste liquid treat- ment, uneven and brittle film, porous, easy	Mg-Zn-Ca alloy	Lump-like mi- cro-clusters	159°	-	[188]
	to form cracks	AZ91D alloy	Microspheres	160.19°	1.5°	[189]
Wire electrical discharge ma-	Low cost, high efficiency, high removal efficiency of electro-corrosion prod- ucts/relatively large electrode wire vibra-	AZ91D magnesium	Big etching pits, mi- cro/nanopits, debris particles, pores and microcracks	146°–150°	-	[190]
chining (WEDM)	tion, low accuracy of the machined surface, and serious material waste	AZ31B magnesium alloy	Micro-/nano petal-like structure	$151^\circ\!\!\pm 0.5^\circ$	$4^\circ\!\pm 0.5^\circ$	[191]

CA = contact angle, SA = sliding angle, - indicates no mention.

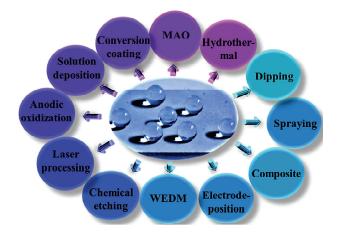


Fig. 9 Schematic diagram of various methods for preparing superhydrophobic surface^[188].

superhydrophobic coating with chemical stability and durability on the surface of AZ31 magnesium alloy by hydrothermal synthesis method. The static water contact angle was 156.7°, and the superhydrophobicity could be maintained for more than one year when exposed to air. In addition, the superhydrophobic coating in 3.5 wt% NaCl solution had good corrosion resistance. Zhang *et al.*^[194] prepared a Mg(OH)₂/Mg-Al composite coating on the AZ31 alloy substrate by co-deposition and hydrothermal methods. The surface was modified by stearic acid, and the maximum static contact angle was 153.5°, and the superhydrophobic surface showed good stability in electrochemical test, hydrogen evolution test and immersion test, significantly improving the corrosion resistance of AZ31 alloy (Fig. 10). But even so, this technique requires to be carried out at high temperature and pressure conditions, and therefore equipment must meet a number of strict requirements, which inhibits the development and application of the hydrothermal method in more fields. Moreover, it is not feasible for mass production due to poor preparation conditions and technical difficulties^[195,196].

4.2 Electrochemical deposition method

Electrochemical deposition is a simple and efficient method to prepare superhydrophobic surfaces on magnesium alloy, which relies on the reduction reaction of cathode to deposit metal or composite layer on the surface of material^[197–199]. Cui *et al.*^[200] prepared

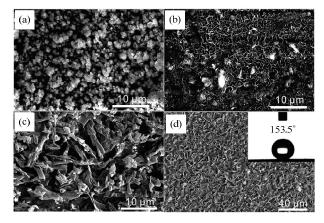


Fig. 10 SEM morphology of the sample after 14 days of immersion test. (a) AZ31; (b) LDH coating; (c) LDH/SA1 coating; (d) SEM morphology and contact angle (inset) of LDH/SA1 coating^[194].



superhydrophobic micro-arc oxidation/zinc stearate (MAO/ZnSA) coating with micro plate-like structure on Mg-4Li-1Ca alloy by electrochemical deposition method, and the static contact angle was $153.5^{\circ} \pm 0.5^{\circ}$. The superhydrophobicity of the MAO/ZnSA composite coating effectively sealed the surface of MAO, and hence prevented the contact between the corrosion solution and the substrate, significantly enhancing the corrosion resistance of the Mg-4Li-1Ca alloy (Fig. 11). Li and Kang^[201] prepared superhydrophobic a coating on AZ31 magnesium alloy by electrochemical deposition and surface modification. The static contact angle and sliding angle were $156.2^{\circ} \pm 0.6^{\circ}$ and 1.0° . Superhydrophobic coatings showed excellent corrosion resistance and chemical stability when immersed in 3.5 wt% NaCl solution and corrosive liquids. After 900 mm and 1100 mm mechanical wear tests, the coating maintained superhydrophobic property and corrosion resistance.

The process can be achieved by employing compact equipment and simple process flow in a short cycle with high metal deposition rate, and is feasible for mass production because it is a low-cost, energy-efficient and easy-to-control method. On the other hand, the film obtained by this process shows weak cohesion strength with the substrate, and in addition to this, heavy metal pollution and adverse manufacturing conditions are unavoidable problems during processing^[202,203].

4.3 Micro-arc oxidation

Micro-arc oxidation (MAO), also known as micro-plasma oxidation or anodic activation deposition, is a surface modification technology that produces ceramic coatings on metal surfaces. By controlling the micro-arc oxidation electrical parameters and the electrolyte system, coatings with different morphologies and structures could be prepared. The prepared ceramic coating had

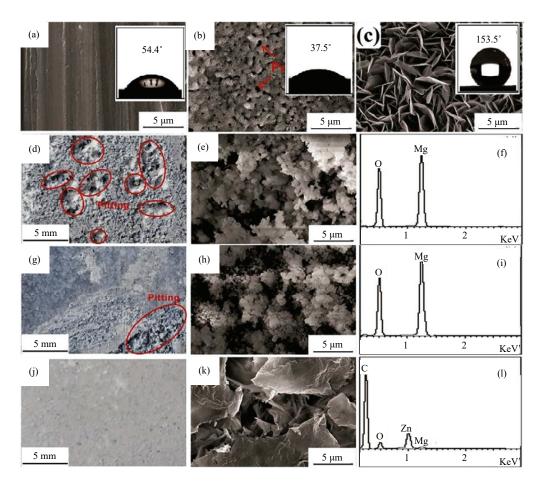


Fig. 11 SEM morphology and CA (inset) of the sample before immersion test. (a) Mg-4Li-1Ca, (b) MAO, (c) MAO/ZnSA coating. Macrograph, scanning electron microscope and energy spectrum of the sample after the 85-hour immersion test. (d – f) Mg-4Li-1Ca, (g - i) MAO, (j - l) MAO/ZnSA coating^[200].



the advantages of high hardness, good wear resistance and strong corrosion resistance^[204,205]. Zhang *et al*.^[206] prepared a superhydrophobic coating on the surface of Mg-1Li-1Ca alloy by MAO and stearic acid modification, and the static contact angle was 155.5° (Fig. 12a). In the potentiodynamic polarization, EIS, and 3.5 wt% NaCl solution immersion tests, the MAO/SA-7h coating showed excellent corrosion resistance and the corrosion current density was significantly reduced (Figs.12b – 12e). Liu and Xu^[207] prepared an AZ31 magnesium alloy superhydrophobic coating in a stearic acid ethanol solution using a two-step method of MAO and superhydrophobic treatment. The static contact angle of the surface was 156.96°. Compared with the AZ31 alloy substrate, the corrosion current density of the superhydrophobic AZ31 alloy was reduced by several orders of magnitude, the amount of hydrogen evolution was greatly reduced, and the corrosion resistance was obviously improved.

4.4 Spraying method

Spraying method is a technology that the coating particles impact the alloy substrate at high speed and then deposit on the alloy surface by aerodynamic force. Li *et al.*^[208] sprayed a fluorine-free suspension on a magnesium alloy substrate to prepare a strong superhydrophobic coating, and the contact angle and sliding angle were 159.5° and 3.8° (Fig. 13a). After a series

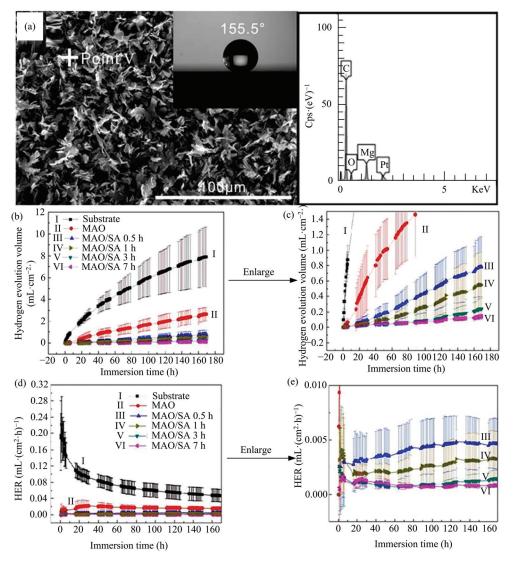


Fig. 12 (a) SEM morphology and CA (inset) of MAO/SA7h coating; (b) hydrogen evolution volume and (c) the corresponding enlarged view; (d) hydrogen evolution rates and (e) the corresponding enlarged view. (I) Mg-1Li-1Ca substrate; (II) MAO coating and (III) MAO/SA 0.5h, MAO/SA 1h, MAO/SA 3h, MAO/SA 7h coatings (IV–VI)^[206].

of mechanical damage tests and exposure to harsh environmental conditions, the coating still maintained good superhydrophobicity. In addition, the coating exhibited excellent self-cleaning performance and corrosion resistance in air and oil (Fig. 13b), and self-healing resistance to O₂ plasma etching (Figs. 13c and 13d). Shi et al.^[209] prepared a polyphenylene sulfide-polytetrafluoroethylene/SiO₂ (PPS-PTFE/SiO₂) coating on the AZ31 magnesium alloy by spraying. The morphology, composition, contact angle, abrasion behavior and corrosion performance of the composite coating were tested by scanning electron microscopy, infrared spectroscopy, contact angle test, abrasive paper wear, and electrochemical tests. The static contact angle of PPS-PTFE/SiO₂ coating was in the range of $(152^{\circ}-145.5^{\circ}) \pm 0.3^{\circ}$, and the sliding angle was less than 5°. PPS-PTFE/SiO₂ coating had good abrasion resistance and excellent corrosion resistance. This process is an environmentally friendly and easy-to-use means of preparing large area coating on different types of substrates with low cost. However, uneven coating occurs during the spraying process, and hazards exist that pose a potential danger to operators' health^[210,211].

4.5 Dipping method

The superhydrophobic surface can be obtained directly by immersing the magnesium alloy into the solution, so the surface that used this method does not need to be modified with low surface energy materials again, which is beneficial to the rapid and large-scale production of superhydrophobic surfaces^[212]. Xun et al.^[213] prepared a coating with low adhesion and superhydrophobic properties on the surface of AZ31B alloy by a two-step in-situ dipping method. The coating had good mechanical stability and ultra-low water adhesion, which gave AZ31B alloy excellent corrosion resistance (Fig. 14). In addition, compared with the AZ31B substrate, the coating had anti-bioadhesion properties, which greatly reduced the adhesion of biomolecules (proteins, bacteria and cells). Ishizaki et al.[214] prepared myristic acid modified micro/nano structure on the surface of AZ31 alloy by one-step dipping method, and the

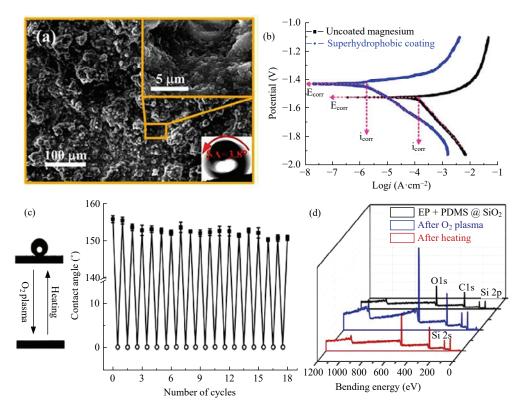


Fig. 13 (a) SEM morphology and rolling angle (inset) of EP+PDMS@SiO₂ coating, (b) potential polarization curves of magnesium alloy substrate and EP+PDMS@SiO₂ coating, (c) self-healing cycles of superhydrophobic coating, (d) XPS analysis of EP+PDMS@SiO₂ coating during self-healing^[208].



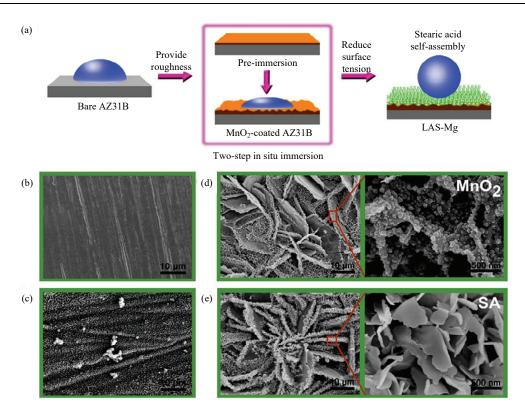


Fig. 14 SEM morphology of the LAS-Mg superhydrophobic coating during its preparation. (a) process flow diagram, (b) AZ31B substrate, (c) pre-immersed AZ31B, (d) MnO₂-coated AZ31B, (e) LAS Mg^[213].

static contact angle of the surface was more than 150°. The superhydrophobic samples were immersed into solutions of pH 4, 7 and 10 for 12 h, and the average static contact angles were $90^{\circ} \pm 2^{\circ}$, $119^{\circ} \pm 2^{\circ}$, and $138^{\circ} \pm 2^{\circ}$, indicating that superhydrophobic coatings had certain chemical stability. Besides, the superhydrophobic coatings showed good corrosion resistance in electrochemical test.

4.6 Chemical etching

Chemical etching method is a manufacturing process that uses strong acid, strong base or concentrated salt solution to remove materials from magnesium alloys to produce rough micro/nano structures^[215,216]. Feng *et* $al.^{[217]}$ prepared a superhydrophobic coating on the surface of AZ91 magnesium alloy by using sulfuric acid etching, AgNO₃ treatment, and dodecyl mercaptan modification. The water contact angle and sliding angle were 154° and 5°. Electrochemical experiments showed that superhydrophobic surfaces had good corrosion resistance. Wang *et al.*^[218] used chemical etching to generate nano-scale three-dimensional porous structures on the surface of AZ31 magnesium alloy (Fig. 15e), and made the surface superhydrophobic by oleic acid modification to obtain a coating with a static water contact angle of 155° (Figs. 15b and 15d). The air cushion effect in the superhydrophobic coating effectively isolated the contact between the corrosive medium and the substrate (Fig. 15c), resulting in improved corrosion resistance of the magnesium alloy substrate. After 6 months of storage in the air, it still demonstrated a good superhydrophobicity (Fig. 15f). Advantages of this process are that it is easy and simple and doesn't require complicated equipment for etching, and capable of producing surface textures with good controllability without applying current and voltage. However, waste stream generated from the process poses dangers to the environment and witnesses high chemical disposal costs^[219,220].

4.7 Wire electrical discharge machining

Wire Electrical Discharge Machining (WEDM) is one of the nontraditional machining processes for removing the material from the workpiece surface by using a continuous moving wire electrode which is usually

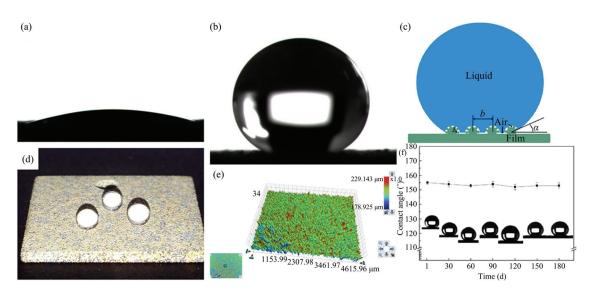


Fig. 15 (a) Static contact angle of magnesium alloy surface, (b) static contact angle of superhydrophobic coating, (c) superhydrophobic diagram, (d) superhydrophobic coating, (e) a 3D profile scanning image of superhydrophobic coating, (f) effect of changes in air storage time on contact angle ^[218].

made of copper or molybdenum as a machining tool electrode to establish a discharge channel via the application of impulse voltage between positive and negative electrodes of the workpiece and the wire electrode backed by the pulsed power supply of the machine tool. Making use of the instantaneous high temperature induced in the discharge channel by collisions of charged particles, the material on the surface melts and vaporizes, resulting in removal of material from the workpiece^[221,222]. Xu et al.^[190] studied the influence of the number of power tubes on the performance of a workpiece surface by machining an AZ91D magnesium alloy using a high-speed wire electrical discharge machine (WEDM-HS). The results showed that the surface treated by WEDM-HS was covered with a carbon layer and exhibited high hydrophobicity (the contact angle range between 146° and 150°). Moreover, an increase in the number of power tubes caused the micro-pits and cracks on the surface of the magnesium alloy to be reduced and resulted in improved corrosion resistance. Qiu et al.[191] fabricated a superhydrophobic surface with micro-nano petal-like structure on an AZ31B magnesium alloy by combining WEDM-HS process and surface modification method, with a contact angle and a sliding angle of 151° $\pm 0.5^{\circ}$ and $4^{\circ} \pm 0.5^{\circ}$, respectively (Fig. 16). The experimental results indicated that the superhydrophobic surface exhibited excellent corrosion resistance and wear

almost an order of magnitude. During the friction-wear test, the superhydrophobic surface demonstrated a lower coefficient of friction. WEDM is a low-cost and highly efficient option for manufacturing as well as has high efficiency in removing electro-corrosion products while violent vibration of the electrode wire, low accuracy of the machined surface and serious material waste are problems to be addressed^[223,224].

resistance, and the corrosion current density decreased by

4.8 Composite method

The composite method is essentially a combination of two or more processing methods to obtain a micro-nano structure superhydrophobic coating on the surface of the magnesium alloy^[225–227]. The two or more methods are used together in order to combine best properties and make the prepared coatings easy to control and exhibit stronger superhydrophobicity and durability than those fabricated by a single processing method. Zang et al.[228] prepared a bio-inspired lotus seed-like superhydrophobic coating on the surface of AZ91D alloy by a combination of hydrothermal synthesis and sonication assisted electroless plating. The static water contact angle was $153.9^{\circ} \pm 2.7^{\circ}$, and the sliding angle was less than 5°. The superhydrophobic coating had good corrosion resistance, which could effectively isolate the corrosion solution and protect the

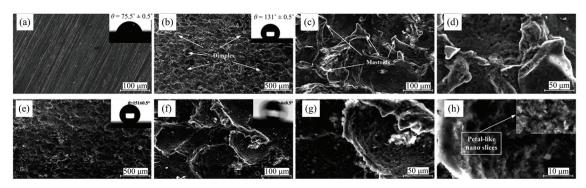


Fig. 16 Scanning electron microscope morphologies and water contact angle (inset) of (a) the bare magnesium alloy, (b - d) the high-speed wire electrical discharge machining (HS-WEDM) surface, and (e - h) the HS-WEDM/stearic acid composite surface^[191].

magnesium alloy substrate. Superhydrophobic coatings showed thermally induced reversible wetting transitions between superhydrophilic and superhydrophobic states, and had excellent fatigue resistance (Fig. 17). Ding et al.[229] prepared an anti-corrosion coating with superhydrophobic and self-repairing on the surface of AZ31B magnesium alloy by hydrothermal synthesis and spraying. The static water contact angle of the composite coating was 163°. The chemical test, immersion method and scanning vibrating electrode method were used to study the corrosion resistance of the coating. Compared with Layer Double Hydroxides (LDHs) coatings, superhydrophobic composite coatings had good corrosion resistance, durability and self-healing properties, which is of great significance for expanding the potential applications of magnesium alloys.

5 Preparation technology of magnesium-based SLIPS

In practical applications, it is found that the superhydrophobic property of superhydrophobic coating is not stable, which will lead to the failure of superhydrophobic coating under high temperature, high pressure or surface damage^[230]. Based on the bio-inspired principle, the researchers studied the special surface properties of the pitcher's mouth area, and synthesized a SLIPS by injecting a low-surface-energy lubricant into the micro/nano-structured substrate^[231–233]. SLIPS lubricants can effectively isolate the corrosion of the substrate by the corrosive medium. Especially in the liquid, the lubricants and the aqueous solution are incompatible with each other, and can be stored for a long time, which has a long-term protection potential for the substrate to reduce corrosion^[234–236]. Table 3 summarizes the innovative technologies applied to the surface processing of magnesium alloys.

SLIPS repels liquids based on the lubricating fluid layer and solid substrate with special microstructure. The solid substrate with special microstructure firmly locks the lubricating liquid layer, and the low-surface-energy lubricating liquid layer has a repellent effect on the liquids. The liquids have a larger contact angle and smaller contact angle hysteresis on the SLIPS^[245].

Jiang et al.^[238] used plasma electrolytic oxidation

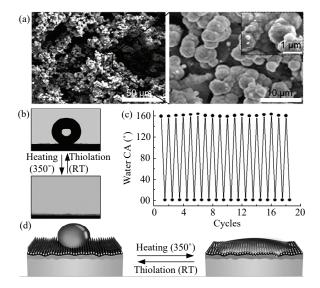


Fig. 17 (a) SEM morphology of the superhydrophobic coating after heating; (b) wettability process of superhydrophobic coating; (c) contact angle measurements after a transition period, (d) schematic diagram of the wettability transition of a superhydrophobic coating^[228].



Technique	Advantage/disadvantage	Material	CA	CAH	Refs.
Hydrothermal meth- od	Coating structure is dense/ poor conditions, not suitable for large-scale production prepa- ration	AZ31B magnesium alloy	123.1°	2.7°	[237]
		AZ91D magnesium alloy	121°	-	[238]
		AZ31B magnesium alloy	122°	12°	[239]
		AZ31 magnesium alloy	105°	3°	[240]
		AZ31B Mg alloy	115°	-	[241]
Anodic treatment	Simple operation, low cost /environmental pollution, high cost of waste liquid treatment	Magnesium alloy	117°	8°	[242]
Spraying method	Large-area preparation, suitable for a variety	Magnesium alloy AZ31B	≤110°	$\leq 10^{\circ}$	[243]
	of substrates, simple operation, low cost/uneven coating	AZ31B magnesium alloy	$106^\circ\pm1.0^\circ$	-	[244]

Table 3 Summary on the processing methods of SLIPS on magnesium alloys for corrosion resistance

CA = contact angle, CAH = contact angle hysteresis, - indicates no mention.

method and hydrothermal method to generate MgAl-LDH coating on the surface of AZ91D alloy, and chemically modified and injected lubricant to prepare PEO-LDH-SLIPS. In the immersion test and electrochemical test, the PEO-LDH-SLIPS showed long-term water-repellency and self-healing ability of surface damage. The water-repellency and self-healing properties made the AZ91D magnesium alloy have excellent corrosion resistance (Fig. 17). Zhang et al.^[239] used a hydrothermal method to form a barrier layer on the surface of AZ31B alloy, chemically modified and injected lubricant to prepare a double-layer anti-icing and corrosion resistant SLIPS. Compared with superhydrophobic coatings, SLIPSs had smaller rolling angles, long-lasting corrosion resistance and anti-icing performance. Zhang et al.^[242] anodized the magnesium alloy in the choline chloride-ethylene glycol based deep eutectic solvent, changed the external anode current density, and formed a conversion film with porous network and jagged nanorod arrays on the surface of the magnesium alloy. After surface modification and injection of lubricating oil, superhydrophobic surface and SLIPSs were obtained. In the electrochemical test, superhydrophobic surface and SLIPSs exhibited better corrosion resistance.

6 Summary and outlook

The corrosion resistance of magnesium alloy is extremely poor, which severely restricts its application range in different fields. Improving the corrosion resistance of magnesium alloy has become an urgent problem that needs to be solved. This article summarizes the research progress on corrosion resistance of magnesium alloys with bio-inspired water-repellent properties in recent years. By analyzing the factors and types of corrosion affecting magnesium alloys, based on the bio-inspired principle, the superhydrophobic surface and SLIPS are prepared on the surface of magnesium alloy to improve the corrosion resistance of magnesium alloy. Nevertheless, during the preparation and application of the magnesium-based bio-inspired water-repellent surfaces, there are still many problems to be solved:

(1) The bio-inspired superhydrophobic surface of magnesium alloy has gradually become a new idea to solve the problem of poor corrosion resistance of magnesium alloy due to its unique interface characteristics and advantages. As mentioned earlier, various processing technologies for constructing superhydrophobic coatings on the surface of magnesium alloys have been proposed, but these technologies still face some problems that need to be solved urgently. For example, the micro-arc oxidation method causes high power consumption, produces a porous surface, and requires a composite packaging treatment. The hydrothermal method has poor preparation conditions and is not suitable for large-scale production. Volatile organic solvents are used in spraying method, which is harmful to human body and creates uneven coating. The preparation process of the composite method is complex and unsuitable for mass production. In the electrochemical deposition method, the adhesion between the film and the substrate is weak and the heavy metal pollution will occur. The dipping method/chemical etching method/solution deposition/conversion coating all can pollute the environment and the cost of waste liquid treatment is high. Laser processing technology is costly and inefficient. The surface accuracy of WEDM is low and the material waste is serious. The anodized film is

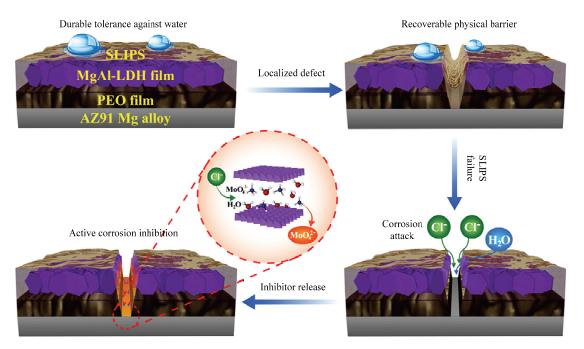


Fig. 18 Schematic protection mechanism for the smart anticorrosion system on Mg alloy^[238].

brittle and porous, making it difficult to process complex workpieces.

(2) Superhydrophobic coating has a wide application in the fields of improving corrosion resistance of magnesium alloys due to its unique properties. On the other hand, some issues still need to be addressed such as high manufacturing cost and limited large-scale production. Furthermore, low-surface-energy modifiers used during manufacturing are expensive, and fluorine-containing substances such as fluoroalkyl silanes and fluoroacrylic copolymers pose significant risks to human health and the environment. In addition, it is found in the practical application of the superhydrophobic coating that its superhydrophobicity was not stable and failed at high temperatures and pressures, or on damaged surface. It is thus considered to be of great significance to develop environmentally friendly, cost-effective and efficient modifiers and to design simple but effective manufacturing process ensuring superhydrophobic coating with self-healing performance on the surface of magnesium alloys can be obtained.

(3) Compared with the superhydrophobic surface, SLIPS has more excellent corrosion resistance and durability, but there are still some problems in the preparation process of SLIPS. The microstructure of the substrate surface is too simple to store lubricant adequately, and the processing technology is unsuitable for large-scale preparation because of its complex operations and long cycle time. Besides, problems such as expensive lubricant, volatile lubricant and poor durability still need to be solved. To solve this problem and extend the service life and create greater value of SLIPS in practical applications, researchers have tried to produce regular-shaped nanostructures by increasing the complexity of the surface microstructure, and have considered replacing liquid lubricants by solid ones. So far, there are few studies on the preparation of SLIPSs magnesium alloys, which needs to be further promoted and studied.

(4) Both superhydrophobic surface and SLIPS can effectively improve the corrosion resistance of magnesium alloy and extend its service life, making it possible to speed up production and use of magnesium alloy in various fields, but there are some problems that still have not been solved. Since the environment in practical applications is complex and diverse, the corrosion resistance of magnesium alloys is more demanding. Especially in biomedical and industrial production, the corrosion resistance and biocompatibility of magnesium alloys need to be considered comprehensively. Therefore, the application research and development technology of magnesium alloy with multi-functional needs to be urgently proposed.

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