Viser Technology Pte. Ltd.
Research and Application of Materials Science
DOI: 10.33142/rams.v3i2.6732

Research Article Open Access

Oxidation Resistance of Magnesium Alloyed by Different Elements: a Brief Review

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Abstract:
The application of magnesium (Mg) and its alloys in automotive and aerospace industry is promoted gradually because of its outstanding properties, such as light weight, high specific strength and excellent castability. However, as a chemically active metal, Mg and its alloys generally possess low oxidation resistance in air at high temperatures because of the high affinity of Mg for O. This has caused a lot of industrial waste and a short service life. In the present work, according to the relevant mechanism of Mg alloy oxidation in air at high temperature, the effect of alloying elements on the oxidation of pure Mg and Mg alloys as well as the research progress of oxidation resistant Mg alloys are briefly reviewed.

Keywords: *Magnesium; Magnesium alloy; High temperature; Oxidation; Alloying elements*

1 Introduction

Magnesium (Mg) and its alloys, as the lightest metallic structure materials, has been widely employed in industries from aerospace to automotive products due to their low density, high specific strength and stiffness, wonderful machinability and excellent castability[1-3]. However, Mg alloys are prone to fire in the process of casting and processing due to their high activity and strong affinity with oxygen, which poses a potential danger to the safe operation of equipment and further limit its widespread applications. Therefore, most commercial magnesium alloys cannot be used in aircraft cabins [4-6].

The oxidation behaviors of Mg alloys will be effected by many factors, such as grain size, the quantity and the distribution of the intermetallic compounds, the content and distribution of alloying elements, the impurity and the texture or twin for the wrought Mg alloys. Both coating and ion implantation also can improve the corrosion resistance of alloys, however, these methods have a single function, i.e., only oxidation resistance at high temperature or corrosion resistance in NaCl solution and they are more complex in contrast to the alloying for Mg alloys. Thus, among of these, alloying is an effective manner to improve the mechanical properties and corrosion resistances. The added alloying elements will modified the microstructure of the alloys, including grain size of α-Mg, the secondary phases and the distribution of alloying elements dependent on the fabrication processes. Thus, review the effect of alloying elements on the oxidation behaviors of Mg alloys at high temperature is critical to enlarge their application.

Usually, the alloying elements added in Mg alloys can be classified into two categories: common elements (Al, Zn, Ca) and rare earth (RE) elements (La, Ce, Nd, Y, Er and Gd). When Mg and its alloys are exposed to air at room temperature, they easily react with oxygen to form oxide films. The structure of oxide divided into three layers as shown in Figure 1[7]. It is well known that MgO, as the dominant oxidation product of pure Mg and Mg alloys, has a low Pilling-Bedworth ratio (PBR) of 0.81 less than unity and plays non-protective roles at the temperature above 450℃ [8-9].

The growth rate of Mg oxide is dependent on the outward migration of Mg ions at a certain temperature for the same alloys [10]. The higher the oxidation temperature, the faster the lattice diffusion of Mg ions. This results in the thickening of oxide film. At the same time, the holes will be produced at the matrix/oxide film interface by the accumulation and segregation of these defects, then lead to the cracks at the critical thickness, which further accelerate the diffusion of Mg ions and promote oxidation [11].

Meanwhile, the microstructure and the physical

Received on October 24, 2021; Accepted on December 10, 2021

properties of oxide film formed on the surfaces of Mg alloys during oxidation also affect the oxidation resistance of the alloys oxidized at high temperature. If the oxide films are compact with few defects, i.e., voids and cracks, the oxidation resistance will be improved. When the alloying elements are added in the Mg alloys, the oxides layer on the surfaces should contain the oxides of alloying elements besides MgO as mentioned.

It is well known that the processing of Mg alloy parts has to be experienced of heat-treatment processing, such as casting and deformation (extrusion and rolling), considered the difficult deformation ability at ambient temperature because of few independent slip systems in Mg alloys. So, understanding of alloying elements – oxidation resistance relationships, and the establishment of the database are all very important for the widespread application of Mg alloys.

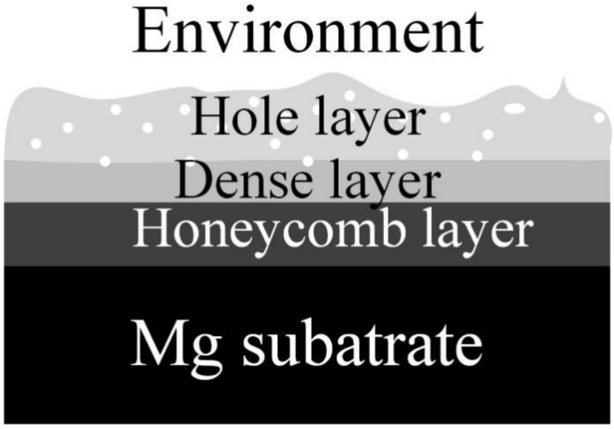


Figure 1 Natural oxide film structure formed on the surface of Mg alloy after oxidation [7]

Hence, the effect of common alloying elements and rare earth elements on the oxidation behaviors of Mg alloys and the research progress of antioxidant Mg alloys are briefly reviewed in this paper.

2 Common Alloying Elements Affect the Oxidation Resistance of Mg Alloys

2.1 Al

Mg-Al serials alloys with good castability, high strength and low price are the most commonly used casting Mg alloys [12-13]. Since the PBR of Al is larger than unity unlike that of Mg aforementioned, the compactness of Al_2O_3 oxide film is better than that of MgO oxide film against the high temperature oxidation [14]. However, it was reported that the oxidation resistance of pure Mg is better than that of Mg-Al alloy at high temperature above the eutectic temperature of Mg-Al alloys mainly due to the low melting point of β-$Mg_{17}Al_{12}$ eutectic phase, in which the β phase is very easy to be evaporated and oxidized therefore caused the poor oxidation resistance [15-16].

The high evaporation rate of Mg described by K_{evap} = 0.6 exp(-25,000/RT) g/cm^{2}s, meaning that the higher the reaction temperature, the more evaporation beats oxidation kinetically. [16] In case of AZ91D alloy, the oxide layers are comprised of the dominant MgO and few of $MgAl_2O_4$. The oxidation mechanism of AZ91D alloy is different at different oxidation temperatures. At temperatures of solid state, the spheroidization and dissolution of second phases take place simultaneously with Al preferential oxidation near the areas enriched in Al, resulting in the high oxidation resistance. However, at the semi-solid temperatures, the incipient melting of the Al-rich fraction, selective evaporation of Mg from liquid islands and condensation within the scale pores and cracks are superimposed on the reaction with oxygen, leading to the weak oxidation resistance.

Barrena [17] reported that weight gain of AZ91 Mg alloys is always higher than that of AM60 through the cyclic oxidation tests at 500-700℃ for 60 min and 540-590℃ for 10 min (Figure 2). This suggests that the oxidation resistance of Mg-Al based alloys is not dependent on only the Al content but the other alloying element addition (Mn and Zn). Conversely, Leontis and Rhines [18] reported that exceeding Al (＞ 1.1 wt.%) addition will accelerate the oxidation rates of Mg not as expected that the addition of Al lower the oxidation rates of Mg alloys. Even at 400℃, over 10 wt.% Al also decrease the oxidation resistance of Mg alloys probably related to the thicker oxide layer [18].

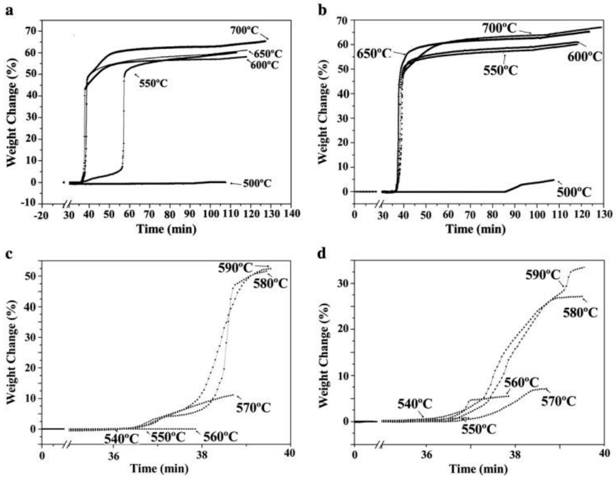


Figure 2 Curves of weight gain versus time for AM60 and AZ91 alloys in air for two different isothermal times: a) AM60 for 1 h, b) AZ91for 1 h, c) AM60 for 10 min and d) AZ91 for 10 min [18]

2.2 Zn

Zn is a useful alloying element in Mg alloys, such as AZ, ZK and Mg-Zn-Y serials alloys, can improve the mechanical properties of Mg alloys by refining the microstructures. However, Zn addition will also reduce the oxidation resistance of pure Mg at high temperature predominantly owing to the following reasons [18]. Firstly, as the melting temperature of Mg-Zn phases is low, a large amount of Mg will evaporate and produce selective oxidation at high temperature [19]; Secondly, Zn is easy to sublimate at high temperature and to penetrate the alloy substrate and oxide film results in the formation of holes at the oxide/alloy interface, which causes the defective oxide films and therefore accelerates the oxidation rate. Although binary Mg-Zn alloy is rarely used because of its instability at high temperature, it has attracted much attention because of its good mechanical properties for the alloys with the other alloying elements additions, such as Mg-Y-Zn alloys, where the oxidation resistance of Mg-Zn alloys was enhanced with the increasing addition of Y (Figure 3) [20].

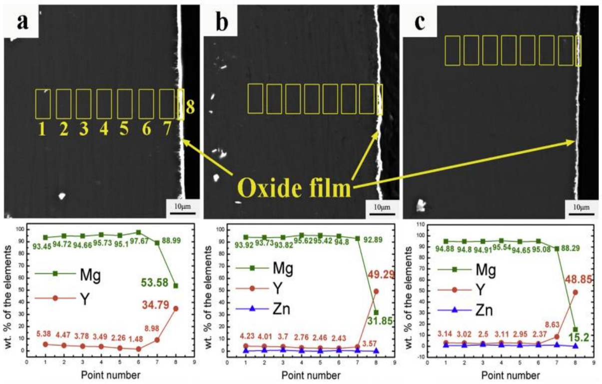


Figure 3 Backscattered electron images of the cross-sectional micrographs of the oxide films of (a) Mg-3Y, (b) Mg-3Y-0.5Zn and (c) Mg-3Y-1Zn alloys with their corresponding EDS analyses of Mg, Y and Zn elements [20]

2.3 Ca

It was found that Ca addition in Mg can lower the oxidation rate at high temperature [21-22]. Small amounts of Ca addition (0.5、1.5 and 3.0 wt.%) decreases the oxidation rates of pure Mg oxidized at 440℃ and 500℃ by thermo-gravimetric experiments, indicating that the addition of Ca is beneficial to the oxidation resistance of pure Mg [21]. However, the oxidation rate of Mg-3Ca alloy is higher than that of Mg-0.5Ca alloy at all tested temperatures. It was explained by the low melting point Mg-Mg_2Ca eutectic structure formed in Mg-Ca alloys with Ca content exceeding 0.5 wt.%, which will be melt leading to a liquid island structure at a higher temperature. Meanwhile, the mass gain results suggested that these liquid islands promote the evaporation of Mg [21]. Further investigation by Auger electron spectroscopy (AES) on the element distribution in the surface layer of alloys after oxidation at 440℃ and 500℃ showed that only MgO layer formed on pure Mg with uniform distribution of Mg and O. However, the element of Ca has been detected on the surface for Mg-3Ca alloy except for MgO. It is also noted that the composition of the oxide layer changed with the different oxidation temperatures. At 440℃, the surface layer consists of the outer Mg oxide layer and the relative inner oxide layer containing Ca. However, when the oxidation temperature reached 500℃, the outer oxide layer is CaO, followed by the mixed oxide layer containing Mg and CaO.

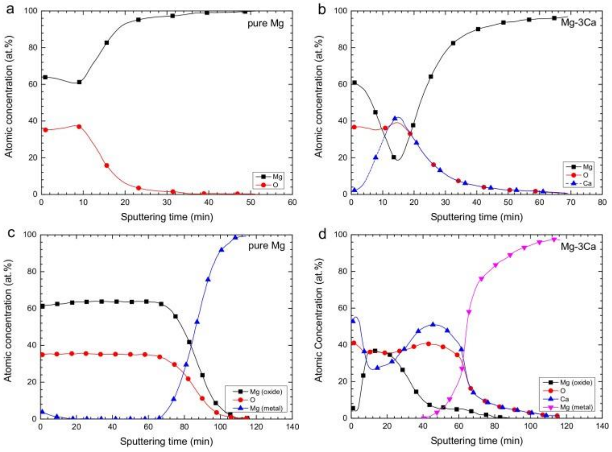


Figure 4 AES depth profiles sputtered from the surface of (a) pure Mg at 440℃; (b) Mg-3Ca at 440℃; (c) pure Mg at 500℃ and (d) Mg-3Ca at 500℃ for 1 h[21]

The improvement in the oxidation resistance of pure Mg by small amounts of Ca addition is possibly related to the following reasons: (1) during the oxidation process, a thin, dense and composite oxide layer composing of CaO and MgO is formed, which plays a good protection roles by hindering the diffusion of O and Mg; (2) the addition of Ca will partially replace Mg in MgO during oxidation process, which increases the density of MgO in the oxide film; (3) The highly thermal-stable second phase formed. Pure Mg alloyed with Ca will produce Mg_2Ca eutectic structure along the grain boundaries. When the oxidation temperature is lower than the melting temperature of Mg-Mg_2Ca eutectic structure, Mg^{2+} ions are difficult to diffuse along the dendritic grain boundaries and therefore inhibit the oxidation.

2.4 Sr

The different ignition temperatures of various Mg-Sr alloys are dependent on the amount of Sr, which is easily segregated to the surface prior to ignition because of its high activity. The ignition temperature of pure Mg is increased by 214℃ from 640℃ of pure Mg to 854 ℃ of Mg-6 wt% Sr alloys. [23] For the high temperature oxidation behavior of Mg–Sr hypoeutectic alloys, a non-uniform two-phase structure oxide scale formed over the interdendritics after oxidation at 500 °C in dry air flow for 12 h.

Note that the secondary phases oxidize primarily causing the formation of MgO and SrO on the surface and continued, which is related to Sr surface activity affecting the initial oxidation sequence. [24] It is highlighted that 75 ppm Sr addition exhibits the similar oxidation resistance of 9 ppm (wt) Be addition in molten AZ91 alloys at 680℃ for 300 min [25], suggesting that Sr could substitute the toxic element of beryllium (Be) usually used in casting of Mg alloys.

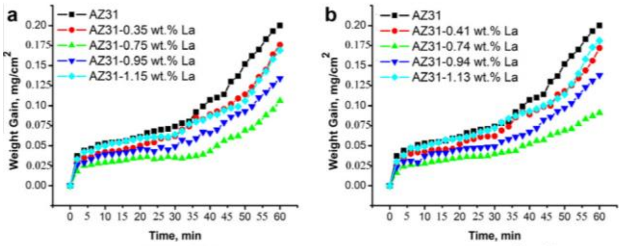
3 Oxidation Resistance of Mg Alloyed with Rare Earth Elements

In the past decade, the effect of rare earth (RE) elements on the microstructure, mechanical performance, creep resistance and corrosion resistance of pure Mg has been studied extensively. The addition of RE could enhance the mechanical properties, formability and creep resistance of Mg alloys at high temperature. Furthermore, the addition of RE can improve the oxidation resistance of Mg alloys. Note that the rare earth elements are divided into two subgroups, light rare earth elements and heavy rare earth elements. We will review the effects of light rare earth elements of La, Ce and Nd, heavy metal elements of Gd, Y and Er on the oxidation resistance of Mg alloys.

3.1 Light RE elements

3.1.1 La

As the P-B ratio of La_2O_3 (1.11) is larger than that of Mg [26], the dense oxide layer will be formed on pure Mg by adding of La compared to that of pure Mg, and this can improve the oxidation resistance of Mg alloys. The addition of not only La but La_2O_3 could improve the oxidation resistance of AZ31 Mg alloys, in which the addition of 0.9 wt.% La alloy exhibits the best oxidation resistance of all samples (Figure5) [27]. When the practical La content over 0.9 wt.%, the oxidation resistance of AZ31 is worsened due to the loose and porous oxide film formed and the reduced adhesion to the substrate. As a RE element, the addition of a trace amount of La has a very positive role in improving the oxidation resistance of Mg alloys.



a. AZ31 with La

b. AZ31 with La_2O_3

Figure 5 Oxidation kinetic curves of AZ31 with both additions. (a) AZ31 with La addition. (b) AZ31 with La_2O_3[24]

3.1.2 Ce

Cerium (Ce) is one kind of light RE elements usually as the alloying elements in Mg alloys. Wang pointed that the Ce addition lowered the mass gained of AZ31 alloy oxidized at 500℃ compared to that of AZ31 alloys [28]. It was found the addition of 0.25 wt.% Ce could increase the ignition points of AZ91 and AM50 alloys by 50℃ primarily ascribed to the dense Ce_2O_3 layer formed [29]. Once this layer is mixed with MgO, the composite oxide film will be denser and be free of defects [30]. However, if the addition of Ce exceeds 0.25 wt.%, the oxidation rate will increase related to the limited solid solubility of Ce in both alloys. The surface morphologies of AM50 alloys with and without Ce addition after oxidation at 500℃ for 1 h were examined shown in Figure 6, where the loose and porous oxide film could be observed on the base alloy. When 0.25 wt.% Ce was added, the surfaces of oxidized alloys were relative smooth with very few holes and defects. Further increasing addition of Ce to 0.45 wt.%, the distinct holes and defects can be seen on the surface of the alloy again (Figure 5C). Thus, a small amount of Ce addition can improve the oxidation resistance of Mg alloy. These dense Ce oxide layer on the surface can protect the substrate from further oxidation. In addition, the existence of Ce in Mg alloy has no adverse effect on mechanical properties. Consequently, from a technical point of view, Ce-containing Mg alloys are worthy of further study.

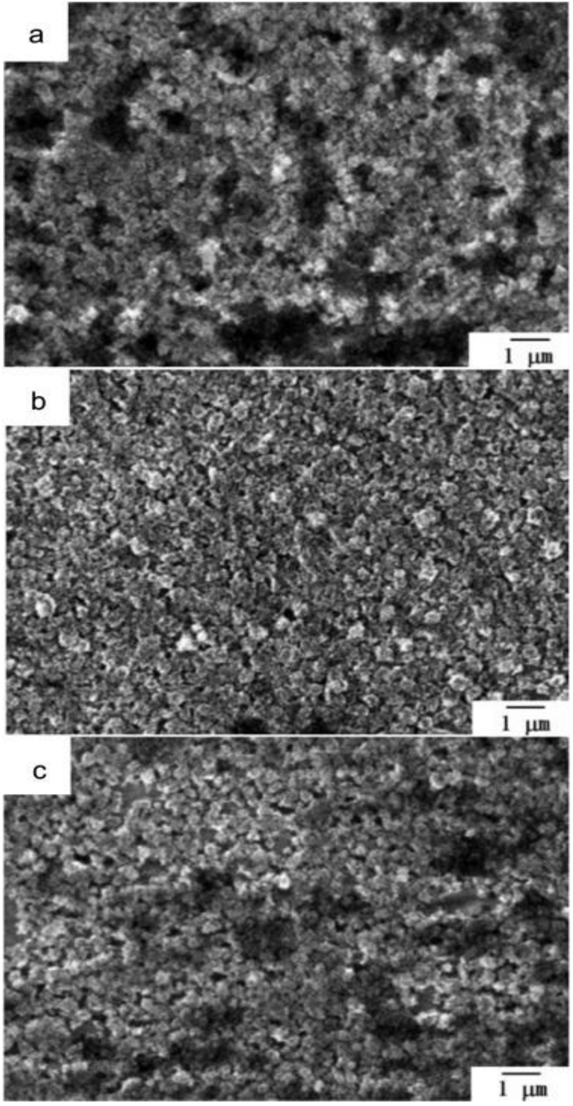


Figure 6 SEM micrographs on the surface of the AM50 Mg alloy after isothermal oxidation in air at 500℃ for 60 min: (a) 0 wt.% Ce with high porosity; (b) 0.25 wt.% Ce showing dense oxide layer; (c) 0.45 wt.% Ce showing porous oxide layer[29]

3.1.3 Nd

To improve the oxidation resistance of alloys, alloying element of Nd could be considered. Small addition (2.87 and 4.5 wt.%) of Nd can improve oxidation resistance of Mg oxidized at 500℃, while the oxidation resistance decreased significantly when Nd content is 11.2 wt.% and 25 wt.%, which are related to the cracks or pores on the surfaces of alloys during oxidation (Figure7) [31-32]. Moreover, MgO will be formed prior to Nd_2O_3 at the initial stage during oxidation via thermodynamic calculation. The initially formed MgO reduces the content of Mg elements on the surface of α-Mg as shown in Figure 8. Correspondingly, the surface activity of Nd increases leading to the formation of Nd_2O_3 [33]. As the PBR ratio of Nd_2O_3 is 1.13 [34], the dense composite oxide film can effectively protect the alloy matrix from further oxidation.

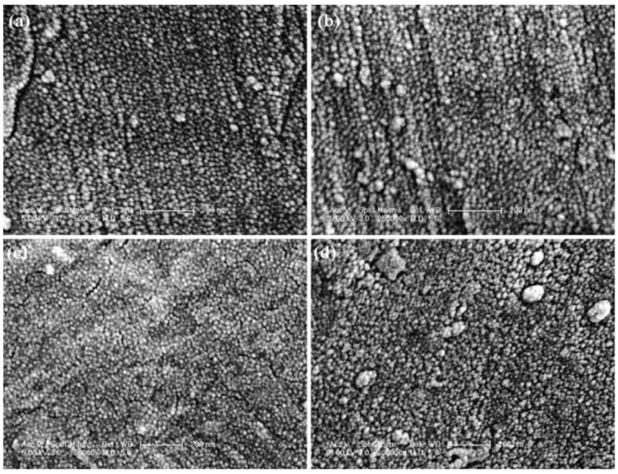


Figure 7 Surface morphologies of MgNd alloys (Nd = 25 wt.%) oxidized in pure O_2 up to 90 min at (a) 723 K, (b) 773 K, (c) 823 K and (d) 873 K[33]

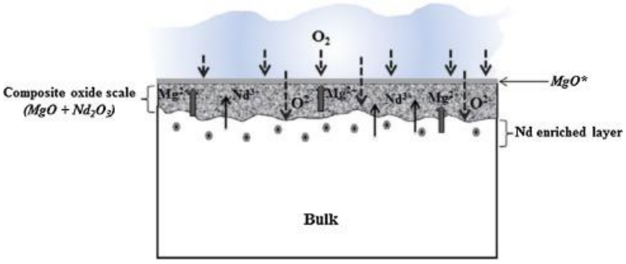


Figure 8 The schematic depiction of the oxidation in dilute Mg–Nd alloys at 500℃ for 12 h in dry air [33]

The oxidation mechanism of Nd in ternary Mg alloy is very complex. Arrabal highlighted that 1.4 wt.% Nd addition could improve the oxidation resistance of AZ91D alloys at 410℃, where the oxidation rate of Nd-containing alloy is 72% compared to the base alloy [35-36]. The improvement can be interpreted by the increasing volume of Al-Nd intermetallic compounds and the decreasing of $Mg_{17}Al_{12}$ phase by Nd addition as shown in Figure 9 (a) and (b). Compared with other rare earth elements, Nd is an ideal addition element that can protect Mg alloy from oxidation. The oxidation kinetics curves of Mg-Nd (Nd = 25 wt.%) alloy follow the parabolic oxidation at 723-873K. [31] The oxide layers show a triplex structure: the outer layer is composed of MgO, Nd_2O_3 and $Nd(OH)_3$, the middle layer chiefly consists of MgO and Nd_2O_3, and the inner layer is made of MgO, Nd_2O_3 and the content of the substrate, where the Nd_2O_3/MgO layer contributed to the high oxidation resistance of Mg-Nd alloys acted as a barrier to retard the outward Mg^{2+} diffusion.

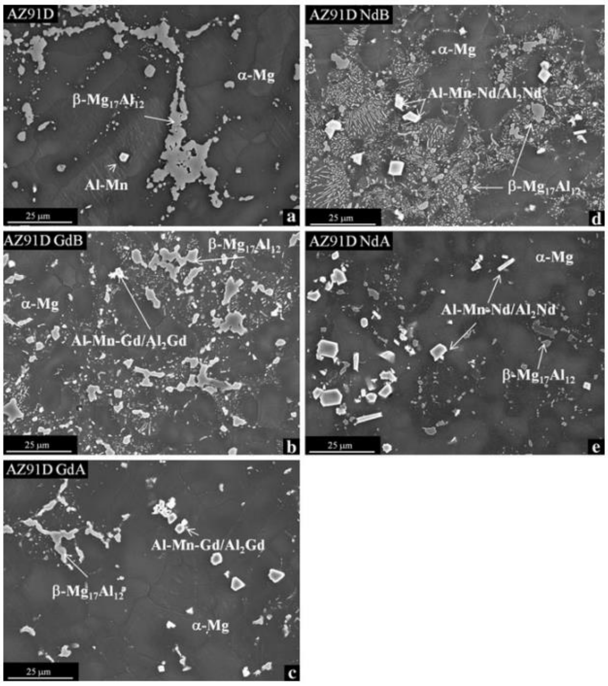


Figure 9 SEM micrograph of AZ91D (a) AZ91D, (b) AZ91D-0.2Gd, (c) AZ91D-0.7Gd, (d) AZ91D-0.7Nd and (e) AZ91D-1.4Nd[36]

3.2 Heavy RE elements

3.2.1 Gd

Gd is one kind of heavy RE elements, which often was used as an alloying element in improving mechanical properties of Mg alloys. Moreover, it can also improve the oxidation resistance of Mg alloys [18]. Arrabal [37] concluded that the 0.7 wt.% Gd addition can improve the oxidation resistance of AZ91 alloy because the addition of Gd could effectively suppress the formation of unstable $Mg_{17}Al_{12}$ phase during casting. Liu et al. [38] studied the oxidation resistance of Mg-Gd-Y-Zr alloy in oxygen or mixed gas of oxygen and water vapor in the temperature range of 230℃ to 300℃. It was found that weight gain is negligible after exposure to the atmospheres for 10 h, meaning that the oxidation rate of Mg alloy containing Gd and Y is low in contrast with the matrix.

The good oxidation resistance is that the surface is composed of MgO, Y_2O_3 and Gd_2O_3, which hinders the diffusion of Mg and further protects the alloy from further oxidation. Wang et al. [39] studied the high temperature oxidation behaviors of Mg-10Gd-3Y alloy. The oxidation kinetics curves follow the parabolic law but not linear even oxidation at 600℃ for 90 min as shown in Figure 10, indicating that this protection is also applicable to semi-molten alloys due to the protective effect of Y and Gd additions.

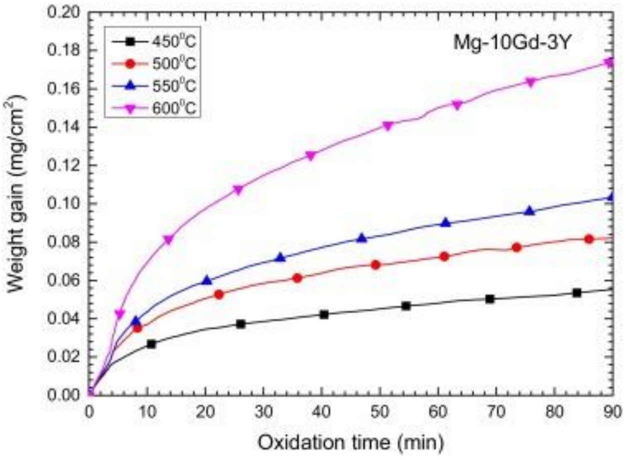


Figure 10 Weight gain curves of the Mg–10Gd–3Y alloys oxidized at 450-600℃ for 90 min [39]

Kim [36] attributed the better oxidation resistance of Gd-containing Mg alloys to its high solid solubility (23.5 wt.%) in Mg. With the increasing temperature, the solute concentration in the matrix increases due to the dissolution of the intermetallic phases. Moreover, the high solid solubility of Gd in Mg promotes the formation of dense layer during the oxidation process, and therefore improves the oxidation resistance. Thus, it is an effective method to develop a new Mg alloys via Gd addition. And these alloys show the desirable oxidation and flame resistance, which can be used at high temperature in future.

3.2.2 Y

The effect of various addition of Y on the Mg alloys has been studied [40-42]. For Y containing Mg alloys during oxidation, the following reactions are possible occur. [43]

$$2Mg + O_2\,(g) = 2MgO \tag{1}$$

$$2Y + 3/2\ O_2\,(g) = 2Y_2O_3 \tag{2}$$

$$2Y + 3MgO = Y_2O_3 + 3Mg \tag{3}$$

The calculated results show that the element Y is much more active than Mg, the selection oxidation of Y is very possible due to the larger affinity of Y with O, and the outward diffusion of Y accelerating with the increasing temperature based on reaction (3), that makes MgO deoxidize associated with the formation of Y_2O_3 as the outer layer oxide film. Yu attributed the improved oxidation resistance of Mg with 3.7 wt.% Y addition to a composite oxide film of Y_2O_3 and MgO (Figure 11). Further investigation by XPS shows that Y_2O_3 is mainly concentrated in the inner layer (Figure12). However, 8 wt.% Y addition cause the significant embrittlement [43-44]. For $Mg_{3.08}Y_{0.68}Ce$ alloy oxidized at 673 and 773 K, The oxidation dynamics curve follow the parabolic-line law, which can be fitted as:

$x^2 + 3.38308\ x = -2.92579 + 0.02548\ t$, R-square = 0.99963, for T= 673 K, and

$x^2 + 1.08078\ x = -1.03408 + 0.03089\ t$, R-square = 0.99920, for T= 773K, where t and x represent the time and mass gained, respectively. [43]

Except single Y addition, combination Y and Al addition in Mg alloys demonstrates that the addition of Al exceeds 2.5 wt.% could improve the oxidation resistance of Mg-2.5Y alloys. For low Al addition (< 2.5 wt.%), the oxidation resistance of alloys is weak due to the high-melting-point Al_2Y formed during casting that inhibits the formation of Y_2O_3 on the surface [43]. While for high Al addition (> 2.5 wt.%), the oxidation resistance is better owing to the compact Al_2O_3 films formed on the surface by the reaction between the remaining Al and oxygen during oxidation, and hindered the diffusion of Mg and O ions.

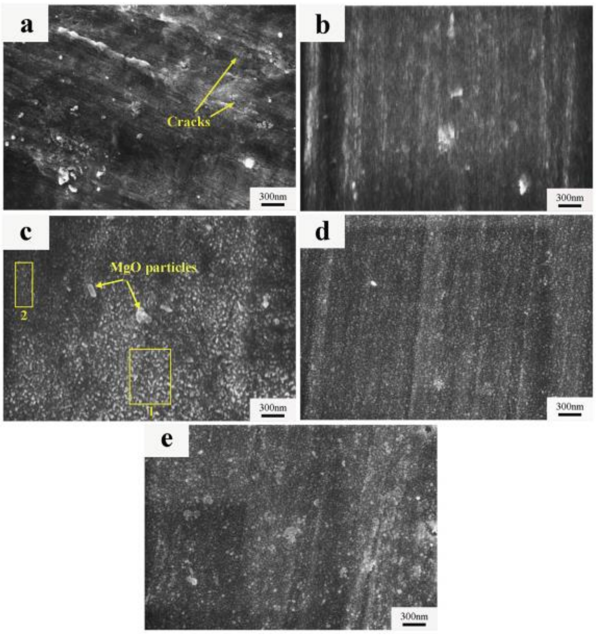


Figure 11 Surface morphologies of (a) Mg-0.5Y, (b) Mg-1.0Y, (c) Mg-1.7Y, (d) Mg-3.7Y and (e) Mg-5.5Y alloys oxidized at 550℃ for 6 h in dry air [40]

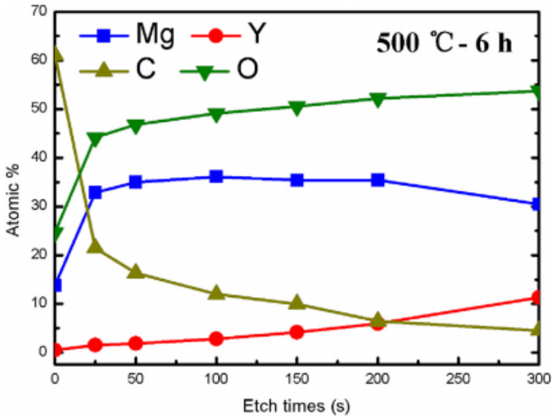


Figure 12 The variation in the atomic contents of elemental Mg, O, Y, and C along the depth direction in the oxide film of Mg-3.7Y alloy [40]

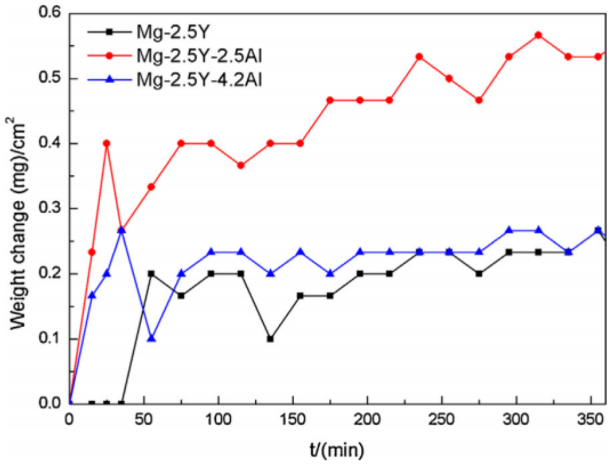


Figure 13 The weight gain curves of Mg–2.5Y, Mg–2.5Y–2.5Al and Mg–2.5Y–4.2Al alloys oxidized at 500℃ for 360 min in the dry air [43]

3.2.3 Er

Few studies evaluated the oxidation behaviors of Mg-Er Mg alloys at high temperature compared to Mg-Al and Mg-Y alloys. The Er_2O_3 in the intermediate layer and the dense fine-grained Er_2O_3 in the inner layer dominantly improve the oxidation resistance of Mg alloys. Additionally, the impurities Ca in the matrix generated the discontinuous CaO in the outer layer hardly affect the oxidation resistance of alloys. And the schematic diagram of the multi-layer oxide film structure shown in Figure 14 (a)～(d).

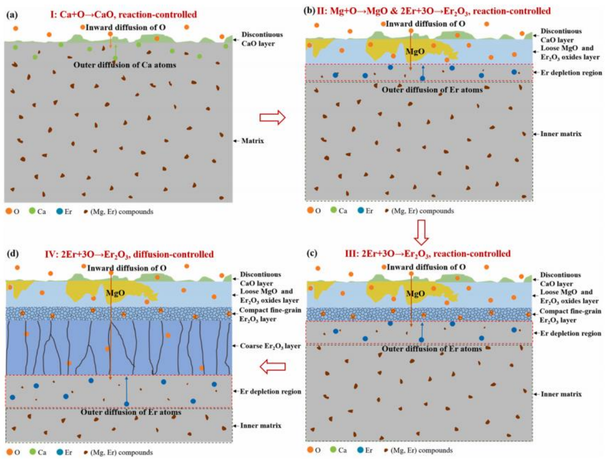


Figure 14 Schematic diagrams of multi-layered oxide film structures formed on the Mg-8Er alloys at 500℃ in air[44]

4 Conclusions

Oxidation resistance is a critical issue for Mg alloys because the oxidation is not inevitable during fabricating and processing of these alloys due to its high affinity with O. Alloying is an effective manner to improve the oxidation resistance of Mg and its alloys. Hence, the effect of alloying elements (Al, Zn, Ca, Sr, La, Ce, Nd, Y, Gd and Er) on the oxidation resistance of Mg and Mg alloys was addressed. The conclusion and the future development were given as follows.

(1) For common alloying elements, the proper addition of Al, Ca and Sr is beneficial to improve the oxidation resistance of Mg alloys, while the addition Zn will decrease the oxidation resistance.

(2) With respect to the rare earth elements X (X: La, Ce, Nd, Gd, Y and Er), small amounts of addition of X enhance the oxidation resistance of Mg alloys related to their solubility in Mg, the relative large PBR compared to that of Mg and the compact X_2O_3 film formed on the surface. These factors work together to improve the oxidation resistance of Mg alloys.

(3) Combination additions of common alloying elements with rare earth elements can further enhance the oxidation resistance of Mg alloys compared to those of binary Mg alloys.

(4) Tailoring the alloying elements added in Mg alloys should be careful because these elements are favorable to strengthen the mechanical properties, the formability and the corrosion resistance in ambient surroundings. How to coordinate these factors among the alloying elements should be further studied as well as the other alloying elements, such as Sr, Mn, Sm and Sn and so on, also should be further investigated to enlarge the applications of Mg alloys.

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