Research Progress in Magnesium Alloys as Functional Materials

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Abstract: Magnesium alloys have a significant advantage, low density over other structure metals currently and have been widely used in various fields such as transportation and aerospace. With the development of research and the enlargement of research scope, more advantages have been developed: high storage capacity, high theoretical volumetric energy density, extraordinarily high damping capacity, good biocompatibility, excellent shielding efficiency as well as impressive thermal conductivity. Therefore Mg alloys have the potential to be various functional materials, such as hydrogen storage material, rechargeable electrochemical batteries, damping material, biodegradable implant material, electromagnetic shielding material, and thermal conductive material. Unfortunately, each kind of functional material has bottlenecks needing to be broken through, and a lot of researches have to be carried out. This review comprehensively covers the research progress and the up-to-date summary of Mg and Mg alloys as functional materials in recent years. The six kinds of functional materials above all will be discussed.

Key words: magnesium alloys; functional materials; novel processing; properties; alloying

Magnesium alloys are getting speedy development in recent years because of their inherent advantages such as low density and high specific strength^[1]. With the development of researches, numerous appealing properties of Mg and its alloys are found and they are considered to be the promising candidates for functional materials in various applications because of the special advantages compared to other alloys^[1-5]. For example, Mg alloys have high storage capacity, so their hydrides are promising to be hydrogen carriers. Pure Mg, Mg-Ni, Mg-Al, and Mg-Re etc. have been developed as Mg hydrogen storage materials. In addition, Mg is expected to be an alternative to lithium in the future ion-transfer batteries due to its higher safety and lower cost. Mg-*M*-B (*M*=Co, Ni, Fe, ...) and Mg-*M*-SiO₄ (*M*=Mn, Fe, Co) were investigated as positive electrode materials for magnesium batteries. Mg alloys' damping capacity is excellent, so they can be used in anti-vibration and noise-reduction applications. Mg alloys also exhibit good biocompatibility and are ideal implant materials

because the content of magnesium in cells is the second largest, and the mechanical properties are close to those of natural bone. Mg-Ca and Mg-Sr series alloys coated by calcium phosphate or hydroxyapatite (HA) exhibit appropriate corrosion rate and good biocompatibility, and reach the requirement of an ideal implant material. What's more, Mg alloys are prominent candidate materials to reduce the harm of electromagnetic wave because of their high electromagnetic shielding performance. And the good thermal conductivity makes them have the potential to be radiator materials. However, there are still many problems to be solved for implementing functional Mg alloys widely. Considerable researches have focused on specific functional properties of Mg alloys and some alloy series have been investigated in particular areas. The present paper will focus on the research progress of magnesium alloys in these aspects, and the research hotspots and the development tendency of further research is summarized in detail. Such a review can provide

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important information for further development of Mg alloys as functional materials.

1 Hydrogen Storage Materials

Hydrogen gas will be the most promising fuel in the future because it is the most abundant resource and green in energy. Hydrogen can be stored as pressurized gas, cryogenic liquid, and solid state hydrogen. Solid-state storage is more economic and safer than gas and liquid storage methods, and many efforts have been made to research solid materials. The US Department of Energy (DOE) proposed a target: the optimal desorption temperature ranges from 60 to 120 °C as well as the hydrogen storage capacity is 6.5 wt% at least [6,7].

Mg is a promising candidate for hydrogen storage in various applications due to its light weight, high storage capacity, reversibility and recyclability. Mg alloys combine chemically with hydrogen to form hydrides that are known as Mg hydrogen-storage materials. In the past decade, a series of hydrides have been prepared, such as Mg_2NiH_4 , Mg_2FeH_6 , $\text{Mg}(\text{AlH}_4)_2$ and some unusual metal hydride Mg_2CoH_5 , Mg_3ReH_7 , Mg_3CrH_6 . However, Mg alloy hydrides are still far from meeting the need for practical applications because of high hydrogen discharge temperature, complicated synthesis process, slow desorption kinetics and high reactivity toward air and oxygen [7-9].

Corresponding to the challenges in application, lots of researches have been carried out to find viable solutions to optimize hydrogenation properties. The commercial hydrogen storage and utilization can be achieved by decreasing the temperature of hydrogen desorption, enhancing the kinetics and improving the cycle life span. Alloying and microstructure modification are the main approaches to improve the hydriding properties [10-12]. Shao et al reported that Mg-Co alloys present high hydrogen storage capacity of around 3 wt% [10], which is obviously higher than that of $\text{TiFe}_{0.86}\text{Mn}_{0.14}\text{Co}_x$ alloys (1.98 wt%) [13]. It was found that the hydrogenation kinetics and the H-storage capacity are dependent on the microstructure and phase composition of the alloys. Large grain boundary area as well as small particle sizes are beneficial to decrease the desorption energy and to reduce the desorption temperature [14-16]. Ballmilling is a main synthesis technique and widely applied to produce hydrogen storage materials. A large amount of fresh surface and defects produced during milling process are contributed to the kinetics enhancement. The nanostructured composites alloying with other transition metals and their oxides can also reduce the desorption temperature and improve the thermodynamic. However, nano-sized Mg is sensitive to oxygen, which is very dangerous and difficult to store and deliver [14-16]. A core-shell structured Mg based nano-composite is an ideal solution to protect the surface of nano-sized Mg for the sake of safety [10, 11]. J. X. Zou et al. developed a core-shell structured Mg based nano-composite with the surface of Mg particles covered by MgO , RE_2O_3

nano-grains, which significantly improved hydrogen storage thermodynamic, kinetic and anti-oxidation properties [6].

The absorption/desorption kinetics can also be improved by adding catalyst. It was known that some metals and transition metal oxides (such as Al, Fe, Ti, B, Zr, Nd_2O_3 and V_2O_5) could be efficient additives. For example, $\text{Mg}_{2-x}\text{Al}_x\text{Ni}$ ($x \leq 0.1$) has lower desorption temperature and faster desorption rate than Mg-Ni. Adding Nb_2O_5 especially nanocrystalline Nb_2O_5 in MgH_2 improves desorption capacity and cyclic stability significantly [16-18].

2 Rechargeable Magnesium Batteries

Lithium ion batteries play an important role in the field of portable electronic devices, electric vehicles (EV) and other energy storage systems. However, the safety and high cost are the main disadvantages that have not been resolved, since the electrolyte is easy to burn and Li is expensive [19, 20]. Consequently finding new types of batteries with greater safety and lower cost is urgent. The theoretical volumetric energy density of Mg-ion batteries is 3832 mAh cm^{-3} higher than that of Li (2061 mAh cm^{-3}), and Mg is relatively cheaper and safer than Li. Mg is expected to be an alternative to lithium in the future ion-transfer batteries. Since Aurbach et al. made a complete Mg rechargeable battery in 2000, more and more research institutions turned their sight to magnesium batteries [19-23].

Suitable electrodes for magnesium ion insertion, de-insertion and conducting electrolytes are critically important for Mg rechargeable batteries. While the Mg-ion is difficult to diffuse in solid-state electrode compared to the Li-ion, and the corrosion resistance of the magnesium battery is low. The reactions of the anode with electrolyte would produce a blocking layer, which limits the achievable battery voltage and leads to the poor cycling ability of Mg batteries. For Mg batteries, the choice of electrolyte is also very critical. There are two aims in present research activities: one is to find suitable cathodes for magnesium ion insertion and de-insertion or commercially viable active materials for Mg-ion batteries; the other is to prepare suitable magnesium ion conducting electrolytes with sufficiently ionic conductivity [23-25].

Although a lot of researches have been carried out, the development of magnesium batteries is still at a preliminary stage, and their poor catalytic activity and low corrosion resistance still restrict their practical applications. The initial research activities focused on various potentially promising metal oxides (vanadium and molybdenum oxides), sulfides and other insertion compounds in organic solvents and low-temperature salt melts; then manganese dioxide (MnO_2) and its polymorphs, Mg-M-B ($M=\text{Co}, \text{Ni}, \text{Fe}, \dots$), Mg-M-SiO₄ ($M=\text{Mn}, \text{Fe}, \text{Co}$) were investigated as a positive electrode material for magnesium batteries [23]. Nevertheless, there is no Mg battery system that is comparable to the Li-ion battery in the overall performance at present [26,27]. It was

reported that micrometer-sized electrode with a mesoporous structure had faster kinetics as well as better morphological stability, because the mesoporous structure could act as a buffer layer and the damage caused by volume expansion during the cycling process was alleviated. MgCoSiO₄ with a mesoporous structure prepared by Yupei Zheng et al. showed a larger discharge capacity and a better recyclability compared with conventional bulk forms^[28-30].

Since magnesium is very active in aqueous solution, the battery is difficult to keep stable. Solid, gel-polymer as well as molten electrolytes were the promising electrolytes in Mg ion batteries^[26,28,31]. It was reported that magnesium achieved reversible deposition in Grignard reagents, but the preparation process was complicated. In addition, Jinjie Zhu et al. demonstrated that the halogen-free electrolyte indeed lessened the corrosion to conventional metal current collectors. They prepared a new type of halogen-free boron based electrolyte (Mg[Mes₃BPh]₂/THF), which possessed high ion conductivity (1.5×10^{-3} S cm⁻¹) and good Mg deposition dissolution reversibility^[19-21,29,30].

3 Damping Materials

Materials with high damping capacity can suppress vibration, control noise and have good structural stability. Pure Mg and some Mg alloys such as Mg-Zr^[32], Mg-Si^[33] and Mg-Ni^[34] exhibit extraordinary high damping capacity. However, Mg-Ni alloys have poor corrosion resistance, Mg-Zr alloys are difficult to get alloying, and damping capacity of some commercial Mg alloys such as AZ31 and ZK60 is obviously lower than that of pure magnesium^[35,36]. Several methods have been employed to enhance their properties, including heat treatment, plastic deformation and alloying^[32,33,35,37]. But these developed alloys barely have both high damping capacity and good mechanical properties.

At present, the Granato-Lucke model is usually used to explain damping mechanism: the dissipation of mechanical energy is connected with the vibrations of shorter dislocation segments and the break away from weak pinning points. Therefore, the movement of internal defects is beneficial to improve damping capacity of materials, while there is no benefit to mechanical strength. The strength and damping capacity seem to be a contradiction for Mg alloys. In 2003, K. Nishiyama et al. sintered Mg-Cu-Mn alloy by powder metallurgy (PM) that showed higher damping capacity at strain amplitudes above 4×10^{-5} as well as tensile strength (290 MPa) than pure magnesium^[38]. But the technique of power metallurgy is expensive and complicated. Efforts are still needed to balance the damping capacity and mechanical strength.

Some researches were still focused on balancing the damping properties and mechanical properties by heat treatment, alloying with other elements and plastic deformation. Alloys with favorable strength such as Mg-Zn-Y-Zr and alloys with high

damping capacity like Mg-Cu-Mn were the research hotspot^[32,39-41]. A new damping mechanism called 'LPSO mechanism' was systematically investigated in the series alloy, because the long period stacking ordered (LPSO) structure existing in Mg-Zn-Y-Zr alloys could optimize the mechanical properties as well as damping capacities simultaneously^[42,43]. Adding appropriate amount of Y and Zn in Mg-Cu-Mn alloys led to the increase of mechanical properties in different degrees and had no damage to their damping performance^[41]. Moreover, the equal channel angular processing (ECAP) and small tensile strain increased the damping capacity significantly at elevated temperatures^[40]. Other researchers emphasized that more attention should be paid to the basic research, since a clear understanding on damping mechanism is beneficial to design high damping-high strength Mg alloys. They devoted themselves to the mechanism of the damping capacities of Mg alloys with very simple structures (such as pure Mg) or with no (or very less) solute and un-solute atoms, second phases such as Mg-Ni and Mg-Mn^[35, 36]. It was reported that the addition of Mn improved not only the corrosion resistance and strength, but also their damping capacity because the long dislocations were connected to the dispersed α -Mn^[36]. Even so, there is no breakthrough in the field of damping theory of Mg alloys, and further studies are still needed^[37, 41, 43].

4 Biodegradable Implant Materials

Metal implants such as titanium alloys, stainless steel and cobalt-chromium alloys have been used as tiny tubular mesh-like structures extensively. However, these materials show higher Young's modulus than bones and poor biocompatibility. So the bio-active and biodegradable implant shave received much attention^[44, 45]. Magnesium and its alloys have been considered to be safe as implantable materials, because magnesium is largely found in bone tissue and the mechanical properties of Mg are close to those of natural bone. Furthermore, Mg alloys are able to degrade gradually in physiological environment, avoiding the secondary operation after being implanted^[44,45]. Additionally, magnesium-based materials have received considerable attention as a promising candidate for a new generation of biodegradable vascular stent materials, owing to their in vivo degradation characteristics and non-toxicity^[46]. However, there are several shortcomings for Mg alloys as implants. Damage will be born duo to the higher corrosion rate of Mg in physiological environment. The hydrogen and magnesium ions generated in the corrosion process are harmful to health, and rapid corrosion is a major reason for the failure of magnesium implants^[46,47]. Controlling the degradation rate of bioactive magnesium implants is the focus of all researches.

In recent years, Mg alloys with various chemical compositions have been evaluated on biocompatibility, strength and corrosion rate. In order to prevent the toxicity and other adverse tissue reactions, the alloying elements existing

in human body or having beneficial effects on tissue regeneration were chosen when designing alloys^[48-52]. There are some new Mg alloys developed for implant application, mainly including Mg-Ca, Mg-Zn and Mg-Sr alloys, because Ca, and Sr are found to be essential elements for bone remodeling, metabolism and regeneration^[53-55]. The proper alloy design can improve the corrosion resistance. The corrosion rate is characterized by pH change or the hydrogen volume released from samples during immersion testing^[56]. The corrosion resistance of Mg-0.3Sr-0.3Ca alloy is obviously higher compared with pure Mg.

Surface modification is considered to be an effective method to decrease the corrosion rate of magnesium alloys. For example, the bio-active and biodegradable calcium phosphate or hydroxyapatite (HA) covered on the surface of Mg alloys improved the corrosion properties as well as the biocompatibility^[47,48]. Micro-arc-oxidation (MAO), electrochemical deposition and magnetron sputtering were applied to form a protective layer on the alloy surface^[49-51]. The volume of hydrogen evolved was much lower for the MAO-treated Mg-Ca alloy sample than the untreated one^[51,52].

5 Electromagnetic Shielding Materials

The accurate, reliable and safe output is critically important for equipment, but they are suffering from unwanted electromagnetic interference (EMI) from other communication systems. Moreover, the radiation coming from devices also generates health trouble. Consequently, the development of EMI shielding materials is in urgent need in order to diminish the trouble produced by electromagnetic radiation^[57,58].

Metals (such as copper and steel) are widely used for EMI shielding due to their high shielding effectiveness of 40~100 dB. The primary mechanism of EMI shielding is reflection because free electrons in them can be the mobile charge carriers. Metals with good electrical conductivity such as copper are all the common candidates for EMI shielding materials. Nevertheless, these metallic materials suffer from their heavy weight, which restricts their applications^[59,60]. Magnesium not only possesses low density, but also has relatively good conductivity and shielding capacity. In recent years, some researches have been performed on shielding properties of Mg and its alloys^[59, 61]. Zhihua Zhang et al. compared the electromagnetic shielding effectiveness of magnesium sheets with that copper, nickel, and aluminum^[62]. In the whole frequency range, the EMI effectiveness of Mg ranged from 60 dB to 75 dB higher than that of Al with the same thickness. IBM has made laptop shells with AZ91D alloy. The thickness of these shells was about 1.4 mm and the shielding capability was stable at 90~100 dB in the 30~200 MHz frequency range. Various commercial Mg alloys were evaluated on the strength and EMI shielding effectiveness, and methods that could improve the EMI shielding of Mg alloys were put forward. Xianhua Chen et al. found that the shielding

effectiveness of ZK60 alloy increased gradually with the increasing of second phase precipitates, and artificial aging treatment following solid solution enhanced the shielding capacity of extruded ZK60 obviously^[63,64]. The present researches on EMI shielding properties of Mg alloys are far from the wide application of Mg alloy shielding materials. The shielding mechanism of Mg alloys still needs to be investigated comprehensively and thoroughly.

6 Thermal Conductive Materials

In the field of the electronic devices and semiconductors, the thermal conductivity property is demanded highly and the thermal conductivity becomes a critical index when selecting a material. Thermal conductivity of metals is higher than others such as composite materials and polymer materials. However the heavy weight of some metals restricts their application in portable electronic devices. Mg is an exception: the thermal conductivity of Mg alloys is better than that of the resin and plastic products; besides their light weight makes them more suitable in the field of electronic packaging^[65,66].

The thermal conductivity of the Mg alloys has been investigated by some researchers. It was found that the thermal diffusivity and thermal conductivity of Mg alloys were sensitive to the microstructure and closely related with the atomic sizes of solute atoms^[65-67]. The addition of alloying element (Al, Sc, Re etc.) reduced the thermal conductivity, while the addition of Zn to Mg matrix had smaller effects on the thermal conductivity^[67-69]. The thermal conductivity of ZM51 alloy at room temperature was measured to be 125 W/m K, almost twice higher than that of Mg-Al series and Mg-Re series^[70]. In addition, grain refinement could be considered as a meaningful approach to obtain high thermal conductivity. Equal channel angular extrusion, accumulative roll bonding and back extrusion were employed to refine microstructure, but these procedures are too complicated and far from the application in mass production. The current research work on thermal conductivity of Mg alloy is still at the preliminary stage, and there is no systematic theory to guide the design of Mg alloys. According to the Wiedemann-Franzlaw model, electrical conductivity is linearly proportional with thermal conductivity. Hucheng Pan et al. tried to find a function between the electrical conductivity and thermal conductivity^[71,72]. The experimental results showed the electrical resistivity for both Mg-Al and Mg-Zn alloys varied linearly with composition, but the thermal conductivity did not show such a case.

7 Summary

1) The Mg alloy hydrides are still far from meeting the need for commercial applications. Efforts are required to design new alloys and to find efficient preparation methods.

2) There have been dramatic increases in both power and energy density of secondary batteries at the lab scale. However,

the current electrolyte composition of Mg-ion battery is complicated, and the cost for preparing the magnesium electrode is high. Magnesium ion batteries with simple production process and low cost are quite necessary to be developed.

3) The properties of Mg alloys for biomedical applications in simulated body fluids should be improved unceasingly. The time for dissolution of the alloys in simulated body environments is still short. Surface modification and new alloy design should be carried out to slow the corrosion speed.

4) The previous researches on Mg alloys as damping materials, electromagnetic shielding materials and thermal conductive materials are insufficient and lack of systematicness. The accurate and prevalent theories should be concluded to guide the actual research activities.

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镁合金功能材料的研究进展

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摘要: 与其它结构金属材料相比, 镁合金具有质轻的显著优点, 因此被广泛应用于交通运输和航空等领域。随着研究的深入和研究范围的扩大, 镁合金更多的优点被开发利用: 较高的理论能量密度, 较大的阻尼容量和屏蔽效能以及良好的生物相容性。因此镁合金具有作为储氢材料、可再充电电池、阻尼材料、可生物降解的植入材料、电磁屏蔽材料、导热材料的潜力, 但是每一种功能材料都存在未解决的瓶颈问题。本文针对近年来大量的研究工作进行了概括, 总结了镁及镁合金作为以上 6 种功能材料的主要研究方向及研究进展, 讨论了未来主要的研究方向。

关键词: 镁合金; 功能材料; 新工艺; 性能; 合金化

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