Microstructural Evolution and Mechanical Properties of As-Cast Mg-12Zn Alloys with Different Al Additions

Yu Zhang\textsuperscript{a,b,*}, Ming Li\textsuperscript{a,b}, Wen-Long Yang\textsuperscript{a,b}, Zong-Gang Wang\textsuperscript{a,b}, Xue-Zhi Wang\textsuperscript{a,b}

\textsuperscript{a} Hexi University, College of Physics and Electromechanical Engineering, Zhangye, China
\textsuperscript{b} Hexi University, Institute of Advanced Material Forming Technology, Zhangye, China

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In this study, Mg-12Zn magnesium alloys alloyed with Al additions (0, 2, 4, 6, 8 and 10, wt.%) were fabricated by permanent mould casting. The Al content on their microstructure and mechanical properties were systematically examined with an optical microscope (OM), a scanning electron microscope (SEM), an X-ray diffractometer (XRD) and mechanical tests at room temperature. The experimental results indicate that the microstructure of the alloys is mainly composed of \( \alpha \)-Mg and semi-continuous or continuous eutectic phases. A higher addition of \( \text{Al}( \geq 6\%) \) causes the generation of the \( \text{Mg}_{57}\text{Al}_{12} \) phases. Notably, the grain sizes of the alloys gradually decrease, whilst the partial morphology of some eutectic phases is modified into lamellar structure with increasing of Al addition. Mechanical properties characterization manifested that, the alloys with different Al additions reveal distinguishing tensile properties. Among them, the alloy with 4\% Al provides an excellent mechanical properties, i.e., a UTS of 206 MPa and an EL of 7.92\%, which is respectively higher 28 MPa and 1.08\% than that of ZA120 alloy. The deterioration in the tensile properties for the higher Al-bearing alloys is possibly related to the lamellar structure, coarse and continuous net-work morphology and \( \beta\)-\( \text{Mg}_{57}\text{Al}_{12} \) phases, respectively.

\textbf{Keywords:} Mg-12Zn magnesium alloy, Al addition, microstructure, mechanical property, \( \beta\)-\( \text{Mg}_{57}\text{Al}_{12} \) phase.

1. Introduction

Magnesium and its alloys are considered to be the lightest metallic structural materials at present. They have the advantages of low density, high specific strength and stiffness, good damping, shock absorption and mechanical processing performance, and have been widely used in rail transit, aerospace, electronic communication industry and other fields\textsuperscript{1-5}. In recent years, the nations of the world attaches great importance to the research of magnesium and magnesium alloys, and has made plans for the research, development and application of magnesium alloys. Among them, there are many studies on Mg-Al-Zn (AZ) series and its multi-component magnesium alloys, such as AZ31, AM50A, AM60B and AZ91, were developed for industry applications. However, the existence of many \( \beta\)-\( \text{Mg}_{57}\text{Al}_{12} \) phases in these alloys seriously affects the properties of the alloys mentioned. As a result, their widespread applications in engineering are severely restricted. The previously published findings manifest that, the brittle \( \beta\)-\( \text{Mg}_{57}\text{Al}_{12} \) phase is prone to crack and the crack usually propagates along the interface between \( \beta\)-\( \text{Mg}_{57}\text{Al}_{12} \) phase and Mg matrix, resulting in large cracks on the fracture surface\textsuperscript{5}. Similar investigation has also been mentioned\textsuperscript{6}, \( \alpha \)-Mg matrix has a HCP structure, while \( \beta\)-\( \text{Mg}_{57}\text{Al}_{12} \) phase possesses a BCC structure, which causes the interface between \( \alpha \)-Mg matrix and \( \beta\)-\( \text{Mg}_{57}\text{Al}_{12} \) phase to become fragile, leading to the formation of micro-cracks at \( \text{Mg}/\beta\)-\( \text{Mg}_{57}\text{Al}_{12} \) phase interface. It is worth mention that \( \beta\)-\( \text{Mg}_{57}\text{Al}_{12} \) phase in Mg-Al system alloy will undergo softening and cannot play a role in hindering grain boundaries slide when the service temperature exceeds 393 K\textsuperscript{7}. From that it can be concluded that, \( \text{Mg}_{57}\text{Al}_{12} \) phase is not only sensitive to crack generation, but also a weakened phase in these alloys. Therefore, aiming at improving of microstructure and properties of the Mg-Al-Zn system alloys has become a critical issue at present. Fortunately, it is reported that Mg-Al alloy containing high content of Zn exhibits fine castability and mechanical properties at ambient\textsuperscript{7}.

As is known to all, Mg-Zn-Al (ZA) magnesium alloys with high Zn and low Al have been proposed as a low-cost, creep-resistant and diecastable alloy\textsuperscript{8,9}, which has a remarkable heat treatment strengthening characteristics. Meanwhile, the main precipitated phases of the alloys include \( \text{Mg}_{57}(\text{Al}, \text{Zn}) \)\textsubscript{0.9} and/or MgZn, which have a good strengthening effect. It is further reported\textsuperscript{10} that the ZA series alloys show mechanical properties superior to that of the AZ series alloy in both room temperature and high temperature, and have a broad commercial application prospect comparable to that of the AZ system alloys. Currently, plentiful researches have been carried out on the design, microstructure and properties of the ZA series alloy at home and abroad. Abundant investigations on ZA series alloy seem to suggest that it is a promising candidate for developing high performance magnesium alloys. According to our previous study\textsuperscript{11}, the Zn/Al ratio has a significant influence on the formation of the phase in ZA series alloys, which affects the properties of the alloys. Wan\textsuperscript{12} et al. investigated the microstructure, mechanical properties and creep resistance
of Mg-(8%-12%)Zn-(2%-6%)Al alloys and pointed out that, the ZA82, ZA102 and ZA122 alloys are mainly composed of α-Mg, ε-Mg51Zn49 and τ-Mg12(Al, Zn)49 phase, the alloys ZA84, ZA104 and ZA124 contain α-Mg and τ phases, the ZA86, ZA106 and ZA126 alloys consist of α-Mg, τ precipitates, ϕ-Al,MgZn, eutectics and β-Mg24Al17 compounds. Although the previously mentioned studies about ZA series alloys have been reported abundantly. But up to now, very little information pertaining to the microstructural evolution and mechanical properties of Mg-12Zn based alloys with the addition of Al has been reported so far. Furthermore, it is of great interest to explore the Zn/Al ratio possible cumulative effects on phase composition of ZA series alloys. Hence, based on the reports of ZA series alloys and previous studies, Mg-12Zn-xAl alloys (0, 2, 4, 6, 8 and 10, wt.%) alloyed with different additions of Al were designed and investigated systematically, so as to provide a reference for the research and development of new type ZA alloys.

2. Experimental procedure

The investigated alloys were prepared from commercial high purity Mg (>99.99%), Zn (>99.999%), Al (>99.99%) ingots. Six sample alloys with the nominal compositions were shown in Table 1 and noted ZA120, ZA122, ZA124, ZA126, ZA128 and ZA1210 (all compositions are in wt.% hereinafter), respectively. The smelting plays a significant role in the alloy fabrication, to ensure the purity of the studied alloys, the Mg, Zn and Al ingots were polished off by a steel brush before preheating. The weight of 2.0 kg of each alloy was melted in a mild steel crucible placed in a temperature of about 200 °C with dimensions of length (210 mm), height (130 mm) and breadth (85 mm).

For the best results, the specimens for OM and SEM observation were mechanically polished with grinding and polishing papers, and then etched by a 4 vol % HNO3 solution at room temperature. The microstructures of the specimens were subsequently observed by an optical microscope (OM, MeF-3) and a scanning electron microscopy (SEM, 450) equipped with an energy dispersive spectroscopy (EDS). The phase compositions in the experimental alloys were analyzed by a D/max-2400 X-ray diffraction (XRD) at 40 kV and 40 mA using Cu Ka radiation with a scanning velocity of 5 °/min and a step scanning 20 from 10° to 90°. The tensile specimens with a gauge section of 15 mm × 2 mm × 3 mm were machined from the bottom of obtained ingots by using a computer numerical-controlled wire-cutting machine, as shown in Figure 1. The mechanical tests were carried out by WDW-100D type electromechanical universal testing machine with a loading rate of 1 mm/s at room temperature. Three specimens were tested for each alloy and the average values were presented as the results of ultimate tensile strength (UTS) and elongation to failure (EL). The transverse fracture surfaces of the tensile specimens were also examined by SEM.

3. Results and discussion

3.1 As-cast microstructure

The XRD profiles of the Mg-12Zn alloys containing different additions of Al are presented in Figure 2. It can be seen from Figure 2a that both α-Mg phase and MgZn2 phase exist in ZA120 alloy. In contrast, the new diffraction peaks of MgZn4 and Mg51(Al, Zn)49 phase are observed for the Al-containing alloys. The ZA122 and ZA124 alloys consist of four phases, i.e., α-Mg, MgZn2, Mg51(Al, Zn)49 and Mg8Zn7 phases. According to the previous investigation13, the MgZn2 binary phase will generate in Mg-Zn-Al alloy when the mass ratio of Zn/Al is more than 2. As for mentioned Mg3ZnAl intermetallic compound, it has a body-centered cubic structure (space group Im3, a=1.416 nm3), meanwhile possesses a high melting point and good thermal stability. With Al addition further increasing, as observed from Figure 2 that, additional diffraction peaks of Mg13Al12_13 phase emerge

Table 1. Chemical compositions of the investigated alloys

| Alloy code | Nominal compositions (wt.%) |
|------------|-----------------------------|
| ZA120      | Mg-12Zn                     |
| ZA122      | Mg-12Zn-2Al                 |
| ZA124      | Mg-12Zn-4Al                 |
| ZA126      | Mg-12Zn-6Al                 |
| ZA128      | Mg-12Zn-8Al                 |
| ZA1210     | Mg-12Zn-10Al                |

Figure 1 - Schematic for permanent mold, ingot and dimension of tensile sample (unit: mm).
in ZA126, ZA128 and ZA1210 alloys, and peaks emerge corresponding to the Mg$_2$Al$_2$ phase tends to decrease with increasing of Al addition. It is confirmed that Mg$_2$Al$_2$ phase has a body-centered cubic structure with a lattice parameter $a=1.06$ nm$^{15}$. Despite the above analysis, the reason for the formation of the intermetallic compounds in the Al-bearing alloys is not completely clear and further study is needed. Through the above analysis, it can be summarized that the Zn/Al mass ratio has a significant influence on the phase composition of the investigated alloys. Namely, if the mass ratio of Zn/Al is less than 2, the Mg$_2$Zn$_7$, Mg$_{52}(Al,Zn)$_{49}$, and Mg$_{17}$Al$_{12}$ intermetallic compounds exist in the as-cast microstructure alloy. The reason is that the addition of excessive Al makes the Al atom at the front of solid-liquid interface combine with Zn atom under non-equilibrium solidification conditions, leading to the generation of form Mg$_2$Al$_2$ phases. On the contrary, when the Zn/Al mass ratio more than 2, Mg$_{32}$Zn$_{49}$, Mg$_{52}(Al,Zn)$_{49}$ and Mg$_{17}$Zn$_{32}$ phases are be detected in the as-cast microstructure alloy. The eutectic compounds of the investigate alloy with Zn/Al mass ratio 2 consist of Mg$_{32}$Zn$_{49}$, Mg$_{52}(Al,Zn)$_{49}$ and Mg$_{17}$Al$_{12}$.

The OM and SEM micrograph images of as-cast Mg-12Zn alloys with different Al additions are demonstrated in Figure 3, where it can be found that the alloys have a typical dendrite configuration with interphases at interdendritic regions, the volume fraction of which increases with increasing of Al addition in the Al-containing alloys. The resultant microstructures of the alloy are mainly composed of primary Mg, non-equilibrium eutectic phase with continuous or semi-continuous precipitated along grain boundaries and tiny irregular granular phases in the interior of the grain. The morphology of eutectic phases of the alloys is gradually evolved from an isolated island into a coarse and continuous netlike, the size and the number of the secondary phases gradually increases, whereas the size of the grains gradually decreases with an increment of Al addition.

It is well known that the size and morphology of dendrite grains are mainly determined by heterogeneous nucleation and solute segregation$^{16}$. As shown in Figure 3a and b, the microstructures of the ZA120 alloy are mainly comprised of coarse α-Mg grains and intermetallic compounds with an isolated island and granular form. When the 2% Al is added to Mg-12Zn alloy, the corresponding microstructural analysis shown in Figure 3c and d. It indicates that the microstructures of the alloy reveal still developed dendritic morphology. Moreover, the eutectic phases in ZA122 alloy are aggregated in some regions, the isolated particles and grain sizes tend to decrease, as compared to the ZA120 alloy. With 4% Al addition, it is observed from Figure 3e and f that the grain size of the ZA124 alloy is dramatically smaller than that of the ZA120 alloy, the morphology of eutectic phase distributed along grain boundary tends to be fine, and the number of eutectic phase is more than that of ZA120 alloy. According to the XRD patterns, the increased phases should be Mg$_2$Zn$_7$, Mg$_{52}(Al,Zn)$_{49}$ and Mg$_{17}$Zn$_{32}$. In contrast, with 6% Al addition, as illustrated in Figure 3g and h, it is distinct that most of the eutectic phases exist as networks along the grain boundaries, while some particles are dispersed inside the grains. Interestingly, it is necessary to note that the distribution of the secondary phases has prominent dendrite segregation (marked by blue elliptic region), and the number of secondary phases increases significantly. Additionally, one can see that the grain size of the alloy further decreases. When the 8% Al is added to Mg-12Zn alloy, it can be found from Figure 3i and j that the morphology of eutectic phases appears to be similar to that of the ZA126 alloy. Apparently, the grain size decreases considerably while the number of eutectic phases of the ZA128 alloy increases significantly (comparing Figure 3a, c, e, g). Meanwhile, the presentation of dendrite segregation trends to be more obvious, and grain boundaries seem to be demonstration conspicuous trend of broadening. When the addition of Al is further increased to 10%, as shown in Figure 3k and l, where further grain refinement effect and the highest value of the volume fraction of the secondary phase can be obtained with the Al addition higher than 8% in the alloy, meanwhile the grain size of the alloy is the smallest among those of the studied alloys. This is mainly due to a higher addition of Al results in the Al enrichment during the solidification, then induces constitutional undercooling in a diffusion layer ahead of the solid/liquid interface and suppresses grain growth thus leading to the grain refinement. Similarly, the primary grains with different morphologies can still be found. Furthermore, the distribution of the secondary phases penetrates the interdendritic regions and demonstrates coarsely and continuously network. Simultaneously, the evident dendrite segregation (marked by blue elliptic region) still can be observed in the ZA1210 alloy.

It should be emphasized that the phase structure and its component depend mainly on the composition of the alloy. Figure 4 displays the high magnification SEM micrograph images of the investigated alloys, where the elementary composition of the typical ZA120, ZA124 and ZA1210 alloys was point-analyzed by EDS and the EDS results are presented in Table 2. By comparison, it can be seen from Figure 4, after being modified by adding different Al, the morphology of the secondary phases is altered predominantly. Evidently, the local regions of eutectics compounds of the Al-bearing alloys reveal obvious lamellar structure, whose volum fraction...
Figure 3 - Optical (a, c, e, g, i, k) and SEM (b, d, f, h, j, l) micrographs of the as-cast alloys: (a, b) ZA120 alloy; (c, d) ZA122 alloy; (e, f) ZA124 alloy; (g, h) ZA126 alloy; (i, j) ZA128 alloy; (k, l) ZA1210 alloy.
tends to be increasement with increasing of Al addition. Furthermore, the partial gray or black precipitates along the grain boundary were replaced by bright precipitates unevenly dispersed in the edge of the whole eutectic phases.

For the ZA120 alloy without Al, as shown in Figure 4a, the eutectic compound presents a smooth island morphology, which is composed of bright phase and gray one. On the basis of XRD results (Figure 2a) and EDS analysis, the gray precipitate (marked by A) can be confirmed as α-Mg and MgZn$_2$ phase. The bright precipitate (marked B) is rich in Zn. Combined with the XRD results, the bright one is identified as MgZn$_2$ phase. With 2% Al addition, it is seen from Figure 4b that the partial eutectic phases of the alloy show lamellar morphology (marked by elliptical circle). Obviously, there are existing three main phases in the eutectic compound: the black, grey and bright phases.
respectively. After adding 4% Al, it also observed that from Figure 4c, similar eutectic morphology that of the ZA122 is also exhibited in ZA124 alloy. As seen in Table 2, the black precipitate (marked A) and gray one (marked C) includes Mg, Zn and Al, combining with the XRD results (Figure 2c), they are regarded as α-Mg and $\text{Mg}_6\text{Al}_2\text{Zn}$ intermetallics. Duley\(^9\) pointed out that brittle particles often act as stress raisers and thereby, as crack initiation sites during deformation, resulting in inferior mechanical properties. Similar literature has been reported that coarse β-Mg$_{17}$Al$_{12}$ phase is commonly considered to be detrimental to the plastic deformation of magnesium alloys. Furthermore, Nie\(^4\) found out that brittle Mg$_{17}$Al$_{12}$ phase cracks easily and the cracks usually propagate along the interface of Mg$_{17}$Al$_{12}$ phase and Mg matrix. The above outcome means that the Mg$_{17}$Al$_{12}$ compounds play an adverse role in the increasing of tensile properties of the studied alloys. It is well known that the presence of fine and uniform phases distributed along grain boundaries is easier to act as an effective straddle to the dislocation motion thus improving the properties\(^1\). It can, thereby, be inferred that the increasement of tensile properties is mainly attributed to the grain refinement strengthening and secondary phase strengthening effects. Also, the fine and homogeneous distributed eutectic phases in the alloy act as obstacles to the movement of dislocations during the deformation process. Nevertheless, it is clearly found that from Figure 5, the addition of Al which is got up to 6% does not maintains continuous increase in UTS and EL except an apparent decrease (from 206 MPa to 171 MPa and from 7.92% to 5.78%), respectively. Indicating that fine grain strengthening does not play a leading role in improving mechanical properties, especially in ZA1210 alloy. It is well accepted that the mechanical properties of the alloy have an important relationship with the distribution, morphology, size and category of eutectic phase\(^1\). It is believed that the decrease of tensile properties of the Al-containing alloys can be explained by the following three aspects: (1) When the addition of Al exceeds 4%, with increasing of Al addition, the grains of the investigated alloys are gradually refined, but the primary α-Mg grains are almost surrounded by the coarsely and continuously interdendritic eutectic compounds, which can induce generation of the cracks and stress concentration at the interface between intermediate phase and matrix during deformation, thus leading to the deterioration of the tensile properties. (2) A higher addition of Al (≥6%) causes the generation of the Mg$_{17}$Al$_{12}$ intermetallics. Duley\(^9\) pointed out that brittle particles often act as stress raisers and thereby, as crack initiation sites during deformation, resulting in inferior mechanical properties. Similar literature has been reported\(^9\) that coarse β-Mg$_{17}$Al$_{12}$ phase is commonly considered to be detrimental to the plastic deformation of magnesium alloys. Furthermore, Nie\(^4\) found out that brittle Mg$_{17}$Al$_{12}$ phase cracks easily and the cracks usually propagate along the interface of Mg$_{17}$Al$_{12}$ phase and Mg matrix. The above outcome means that the Mg$_{17}$Al$_{12}$ compounds play an adverse role in the increasing of tensile properties of the studied alloys. It is

Figure 5 - Mechanical properties of the as-cast Mg-12Zn alloys with different Al content (a) tensile strength and (b) elongation.
reported that the lamellar eutectic microstructures possibly act as crack initiation sites during tensile test, thus leading to the relatively poor tensile properties\textsuperscript{21}. (3) When the Al addition exceeds 4\%, the local morphology of the eutectic phase is evolved into lamellar shape, which is possibly related to the deterioration of the mechanical properties. However, this needs to be further investigated.

By combining above the analysis, it can be summarized that the moderate addition of Al (2 ~ 4\%) has an advantageous effect on the tensile properties of the investigated alloys at room temperature. More important, a higher addition of Al (\geq 6\%) causes the grain refinement does not play a dominant role in improving mechanical properties.

The SEM images of tensile fracture surfaces of the studied alloys with different Al additions are shown in Figure 6. It is well accepted that the cleavage fracture, quasi-cleavage fracture and inter-granular fracture are the main fracture modes of magnesium alloys\textsuperscript{22}. As shown in Figure 6, a number of

![Figure 6 - SEM fractographs acquired from the investigated alloys (a) ZA120 alloy; (b) ZA122 alloy; (c) ZA124 alloy; (d) ZA126; (e) ZA128; (f) ZA1210.](image-url)
cleavage facets with cleavage steps of various sizes, tearing ridges, a few porosities and micro-cracks can be clearly seen, respectively. Furthermore, some river patterns at some places are observed as well, indicating that all the tensile fracture surfaces have mixed fracture characteristics of cleavage and quasi-cleavage fractures. As shown in Figure 6a, some cleavage facets, tearing ridges, visible cracks and a few porosities are observed in the Al-free alloy. The generation of the porosity is ascribed to the developed primary dendrites. According to the report\textsuperscript{11,12}, the porosities induce the origination of cracks, and then the cracks grow and propagate along the dendrite boundaries to the final failure. As shown in Figure 6b, c, the fracture surfaces of the ZA122 and ZA124 alloys too reveal cleavage facets, tearing ridges and a few porosities. This manifesting that the additions of 2 ~ 4% to the binary alloy do not dramatically modify the fracture regime of the alloys. However, the area of the cleavage facets on the fracture surface of the ZA124 alloy tend to increases compared to that of the ZA122 alloy (see Fig. 6b and c). In short, the ZA120, ZA122 and ZA124 alloys show typical quasi-cleavage fracture and cleavage fracture mode. A higher content Al (\( \geq 6\% \)) addition the Mg-12Zn alloy does significantly change the fracture regime of the alloys. As observed in Figure 6d, e and f, the number of cleavage facets and tearing ridges decreases considerably, which are replaced by micro-cracks and numerous fractured secondary phases. This indicated that the fracture regime transformed to brittle fracture. On the basis of existing literature\textsuperscript{11,12}, the Mg\textsubscript{32}Al\textsubscript{9} phase may facilitate the generation of micro-cracks and severe cracking of the particles at the grain boundary due to the their incompatibility with the \( \alpha \)-Mg matrix.

4. Conclusion

(1) The ZA120 alloy mainly consists of \( \alpha \)-Mg and MgZn\textsubscript{2} phase. Al addition results in not only the formation of Mg\textsubscript{2}Zn\textsubscript{17} and Mg\textsubscript{32}Zn\textsubscript{9} phase, but also the production of Mg\textsubscript{32}Al\textsubscript{9}Zn\textsubscript{9} phase within the range of 2 ~ 4% Al. In addition, a higher addition of Al (\( \geq 6\% \)) causes the generation of the Mg\textsubscript{32}Al\textsubscript{9}Zn\textsubscript{9} compound.

(2) The addition of Al has a prominent effect on the morphology of eutectic phases of the investigated alloys. Namely, after adding Al to the ZA120 alloy, leading to the partial morphology of some eutectic phases is evolved into lamellar structure, and this evolution trend becomes more and more evident with increasing of Al addition. Furthermore, the lamellar structure play a detrimental role in the tensile properties.

(3) The Zn/Al mass ratio has a significant influence on the mechanical properties of the investigated alloys. When the Zn/Al mass ratio more than 2, the addition of Al can enhances the mechanical properties, while the Zn/Al mass ratio less than 2, it deteriorates the mechanical properties. The alloy with Zn/Al ratio 2 exhibits excellent mechanical properties, i.e., a UTS of 206 MPa and an EL of 7.92\%, which is respectively higher 28 MPa and 1.08\% than that of the free-Al alloy. Therefore, the amount of Al addition to the binary alloy must be limited within a rational range.

(4) The microstructural parameters of these alloys, such as grains size, eutectic morphology, secondary phase distribution, and intermetallic compounds, demonstrate conspicuously different from each other. It is considered that the distribution, morphology, composition of eutectic phases, are a ominous factor that affects the tensile properties of the investigated alloys.

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