Rare Metal Materials and Engineering
Volume 49, Issue 4, April 2020
Available online at www.rmme.ac.cn

Science Press

Cite this article as: Rare Metal Materials and Engineering, 2020, 49(4): 1151-1158.

ARTICLE

Effect of Potassium Fluorozirconate Salts Mixture on the Microstructure and Mechanical Properties of Mg-3Y-3.5Sm-2Zn Alloy

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Abstract: Three potassium fluorozirconate (K_2ZrF_6) salts mixture (M) were prepared and added to the as-cast Mg-3Y-3.5Sm-2Zn alloys at three different temperatures. The microstructures and mechanical properties of the alloys were investigated by differential thermal analysis (DSC), X-ray diffraction (XRD), optical microscopy (OM), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and tensile test. The grain refinement mechanism of K_2ZrF_6 salts mixture to Mg-3Y-3.5Sm-2Zn alloys was also discussed. The results show that the as-cast Mg-3Y-3.5Sm-2Zn alloy is composed of three phases, namely α-Mg, $Mg_{12}(Y, Sm)Zn$ and $(Mg, Zn)_3(Y, Sm)$. The addition of K_2ZrF_6 salts mixture does not lead to the formation of any new phase which can be detected by SEM and XRD. M3 salt (60 wt% K_2ZrF_6-20 wt% NaCl-20 wt% KCl) added at 780 °C exhibits the best grain refining effect. The mechanical properties of the as-cast Mg-3Y-3.5Sm-2Zn alloy are improved by three salts added at 780 °C, where the alloy refined by M3 salt has the best mechanical properties. The grain refinement mechanism of K_2ZrF_6 salts mixture in the as-cast Mg-3Y-3.5Sm-2Zn alloy is attributed to the grain refining effect of fine Zr particles and zirconium-rich zones obtained by the reduction reaction between Mg and K_2ZrF_6.

Key words: magnesium alloy; potassium fluorozirconate (K_2ZrF_6) salts mixture; grain refinement; microstructure; mechanical properties

Magnesium alloys are widely used in various fields such as aerospace, automotive, electronic communications and other industries because of their excellent properties, including low density, high specific strength and stiffness, etc[1-4]. Rare earth (RE) containing Mg (Mg-RE) alloys have attracted much more attention because of their remarkable mechanical properties at room and high temperatures[5]. Element Y plays an important role in strengthening and heat resistance of WE series alloys, and element Sm, one of the cheapest rare earth elements[6], has great potential for future industrial applications[7]. The mechanical properties of AZ61 magnesium alloy are improved with the addition of 1.0 wt% Sm[8]. A base precipitate or a long-period stacked ordered (LPSO) structural phase can be formed to further strengthen the magnesium alloy by adding element Zn into the Mg-RE alloy[9]. The addition of element Zn can significantly increase the yield strength of Mg-Y alloys[10]. In recent years, Mg-Y-Sm-Zn-based magnesium alloys have been extensively studied. It is reported that Mg-Y-Sm-Zn-based alloy exhibited higher mechanical properties and hardness than other high rare-earth Mg alloy[7,11].

Zirconium (Zr) is a powerful grain refiner for Mg-RE alloys. By adding Zr into magnesium alloy, the grain can be refined, the structure can be homogenized, and the strength, ductility and creep resistance can be improved[12]. Moreover, the addition of element Zr can reduce the grain size of the as-cast alloy by nearly two orders of magnitude, and promote the formation of nearly uniform equiaxed microstructure[13].

The addition of Zr to magnesium alloy is generally realized using a zirconium-rich Mg-Zr master alloy[14]. Mg-Zr master alloys are commonly made by the reduction reaction between pure magnesium and salts mixture based on zirconium

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Received date: April 25, 2019
Foundation item: Science New Star Foundation of Shaanxi Province (2012KJXX-33); Science and Technology Project of Xi'an City (CXYJZKD003); Science and Technology Project of Yulin City

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fluorides (ZrF_4) or zirconium chlorides ($ZrCl_4$)[15]. A substantially chloride-free master alloy was prepared by adding $ZrCl_4$ to the magnesium melt[16]. Zirmax®, a proprietary 33Zr alloy, was made by chemical reduction between ZrF_4 and magnesium[17].

Although the application of Mg-Zr master alloy is popular in industrial processing, there are still some drawbacks. Firstly, $ZrCl_4$ is usually reduced by magnesium to obtain Zr particles, and then those Zr particles form Mg-Zr master alloy with excess magnesium directly. During this process, $ZrCl_4$ may tend to volatilize and form a low valence compound such as $ZrCl_2$. Moreover, it is difficult to preserve and use because the powder of $ZrCl_4$ is deliquescent and hydrolysable. Secondly, ZrF_4 supplied by business is usually a hydrate, which makes it very difficult to remove water from ZrF_4 without oxidizing Zr[18]. What is more, the limited solubility of Zr in magnesium leads to the presence of large amounts of Zr particles in the Mg-Zr master alloy, and some undissolved Zr particles usually exist in the microstructure of magnesium after adding Mg-Zr master alloy to the magnesium melt[19]. The presence of a great number of undissolved Zr particles or clusters is generally harmful to the mechanical properties even though fine undissolved zirconium particles play an important role in grain refinement of magnesium alloys.

In the previous studies, potassium fluorozirconate (K_2ZrF_6) was proved to be non-volatile, stable in air and readily stored and handled[20]. The addition of K_2ZrF_6 salts mixture in Mg-10Gd-3Y magnesium alloy produced a significant grain refinement effect[21]. However, the grain refinement behavior of K_2ZrF_6 salts mixture in the Mg-Y-Sm-Zn-based alloy has not been studied too much.

In the present paper, the effect of K_2ZrF_6 salts mixture (M) on the microstructure and mechanical properties of the as-cast Mg-3Y-3.5Sm-2Zn alloy was investigated. The grain refinement mechanism of M to Mg-3Y-3.5Sm-2Zn alloy was also discussed.

1 Experiment

The melting point of K_2ZrF_6 (840 °C) is higher than the smelting temperature of magnesium alloy (usually under 800 °C). For the better application of K_2ZrF_6 in magnesium alloy, salts mixture must be used to reduce the melting point of K_2ZrF_6. The melting points of NaCl and KCl are 801 and 770 °C, respectively, while the melting point of 50 wt% NaCl-50 wt% KCl system is only 658 °C[22]. Moreover, the NaCl-KCl system is beneficial to the increase in the fluidity of the molten salt and the surface tension difference between molten salt and alloy phase. The composition of three K_2ZrF_6 salts mixture[21] are shown in Table 1. Fig.1 shows the DSC heating curves of M1, M2 and M3 salts. The melting temperature ranges of M1, M2 and M3 salts are 543.08~584.57 °C, 544.98~595.29 °C and 547.34~613.66 °C, respectively. It can be seen that the melting temperatures of the three salts do not exceed 620 °C, so they can be used in this work.

Pure Mg (99.99%) ingot, Mg-Y master alloy (with 30 wt% Y) and Mg-Sm master alloy (with 20 wt% Sm) and pure Zn (99.99%) ingot were used as raw materials to prepare the Mg-3Y-3.5Sm-2Zn alloy in an electric resistance furnace. During the melting process, a protective atmosphere of CO_2 and SF_6 with the volume ratio of 99:1 was employed. After melting Mg and Zn, M salt was added into the melt at designed temperature (750, 780, 810 °C) accompanied by stirring. After holding for 10 min, Mg-Sm and Mg-Y master alloys were added. Finally, the melt was held at 780 °C for 25 min and poured subsequently into a preheated metallic mold. Then the mold was cooled down to room temperature by water cooling.

The DSC curves were obtained by TGA/DSC3+ type synchronized thermal analyzer, and the heating rate was 20 °C/min. The as-cast samples were etched in a solution of 3~5 g picric, 5 mL glacial acetic acid, 10 mL distilled water and 90 mL ethanol, and then the microstructure was observed

Table 1 Chemical composition of K_2ZrF_6 salts mixture (wt%)

Material	M1	M2	M3
KCl	30	25	20
NaCl	30	25	20
K_2ZrF_6	40	50	60

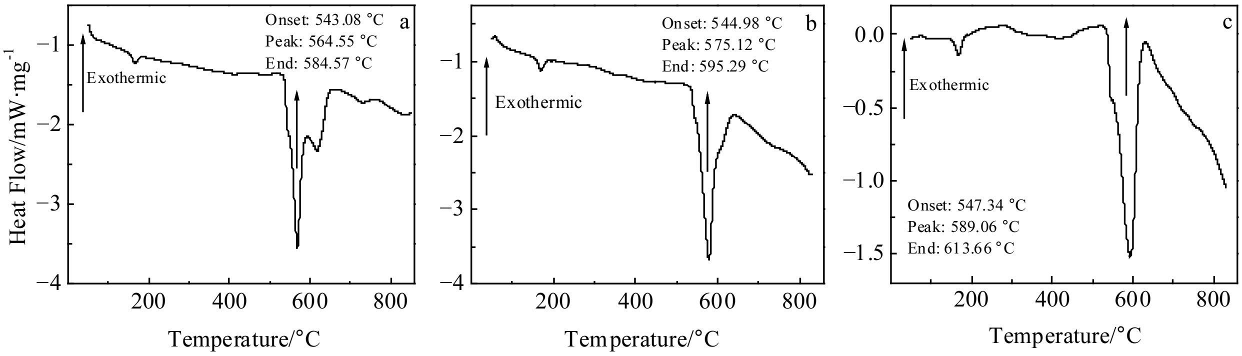

Fig.1 DSC heating curves of K_2ZrF_6 salts mixture: (a) M1 salt, (b) M2 salt, and (c) M3 salt

by OLYMPUS GX51 type metallographic microscope. The average grain size was measured by linear intercept method. The phases in the experimental alloys were analyzed by D/max-RA type X-ray diffraction (XRD) operated at 40 kV and 40 mA. The microstructure was also observed by JEOL JSM-6390A type scanning electron microscope equipped with energy dispersive X-ray spectrometer (EDS). The mechanical properties of the experimental alloys at room temperature were tested by INSTRON8801 type CNC servo stretching machine. The gage dimension of the specimens is 54.5 mm×15 mm×2.0 mm as shown in Fig.2.

2 Results and Discussion

2.1 Microstructures

The result of XRD analysis shown in Fig.3 indicates that the as-cast Mg-3Y-3.5Sm-2Zn alloy is composed of three phases, namely α-Mg, $Mg_{12}(Y, Sm)Zn$ and $(Mg, Zn)_3(Y, Sm)$. To show microstructure of the as-cast Mg-3Y-3.5Sm-2Zn alloy more clearly, SEM images of the alloy without M salts are shown in Fig.4. Table 2 presents the composition of those phases marked in Fig.4 which were obtained by EDS. Combining the SEM image (shown in Fig.4a) with the EDS result of point A (shown in Table 2), it can be seen that the molar content of Mg in the black area is close to 100%, and the molar contents of Y, Sm, and Zn are particularly small. Therefore, it can be determined that the black area is α-Mg phase. According to the result of EDS analysis of point B (gray strip phase) marked in Fig.4b, the phase with the mole ratio of (Y+Sm):(Zn) of roughly 1:1 exists in the as-cast Mg-3Y-3.5Sm-2Zn alloy. It can be indicated that it is the $Mg_{12}(Y, Sm)Zn$ phase. The EDS result of point C (triangular netted shape phase) marked in Fig.4c shows that the phase with the mole ratio of (Mg+Zn):(Y+Sm) of roughly 3:1 exists in the as-cast Mg-3Y-3.5Sm-2Zn alloy, meaning that this phase is $(Mg, Zn)_3(Y, Sm)$ phase. The results of EDS and XRD indicate that the as-cast Mg-3Y-3.5Sm-2Zn alloy is composed of three phases, namely α-Mg, $Mg_{12}(Y, Sm)Zn$ and $(Mg, Zn)_3(Y, Sm)$. The addition of K_2ZrF_6 salts mixture does not lead to the formation of any new phase which can be detected by SEM and XRD.

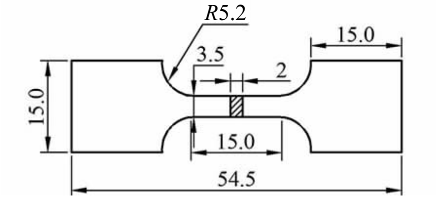

Fig.2 Gage dimension of rectangle tensile specimen

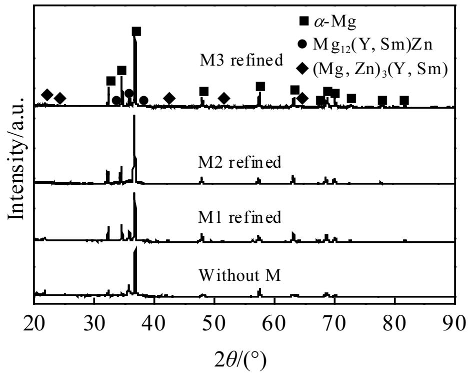

Fig.3 XRD patterns of the as-cast Mg-3Y-3.5Sm-2Zn alloys

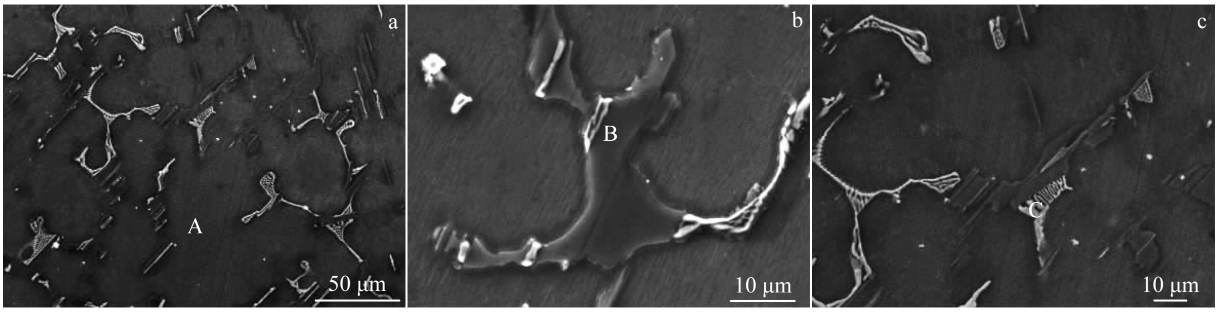

Fig.4 SEM images of the as-cast Mg-3Y-3.5Sm-2Zn alloys without M salts

Table 2 EDS analysis results of different phases marked in Fig.4 (at%)

Point	Mg	Y	Sm	Zn
A	98.37	0.91	0.60	0.12
B	81.34	5.95	3.66	9.05
C	64.23	5.45	15.10	15.22

Fig.5~7 show the microstructures of the as-cast Mg-3Y-3.5Sm-2Zn alloys which were smelted with the addition of M1, M2 and M3 salts at different temperatures of 750, 780 and 810 °C, where the addition of Zr is 0.6% at different temperatures.

As shown in Fig.5a, the as-cast Mg-3Y-3.5Sm-2Zn alloy without M salt exhibits a microstructure consisting of a large

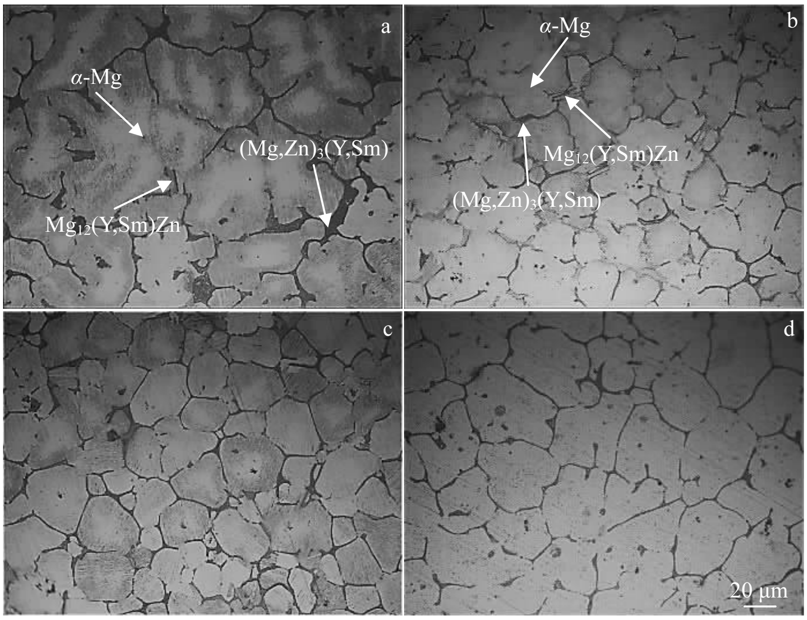

Fig.5 Microstructures of the as-cast Mg-3Y-3.5Sm-2Zn alloys without M salt (a) and refined by M1 at 750 °C (b), 780 °C (c), and 810 °C (d)

number of dendrites with a large dendrite spacing. Fig.5b shows that the microstructure mainly consists of equiaxed grains, rod-like precipitates ($Mg_{12}(Y, Sm)Zn$), and triangular network precipitates ($(Mg, Zn)_3(Y, Sm)$). Compared with the microstructure shown in Fig.5b, the microstructures of Fig.5c, 5d have no significant change. However, the roundness of the equiaxed grains increases and the grain size decreases with the increase of M1 salt addition temperature to 780 °C, as shown in Fig.5c. When the temperature rises to 810 °C, the grains grow up and the roundness of the grains decreases (Fig.5d). Fig.6, 7 show the microstructures of the alloys refined by M2 and M3 salts at different temperatures. From Fig.6a and Fig.7a, it can be seen that the shape of the grains in the alloy is equiaxed at 750 °C, and a few smaller equiaxed grains are mixed between the large grains. When the temperature rises to 780 °C, the grain size of the alloy decreases, as shown in Fig.6b and Fig.7b. Most of the grains in the alloy grow obviously and the grain size is not uniform at 810 °C, as shown in Fig.6c and Fig.7c.

Fig.8 shows the relationship between the average grain size of the as-cast Mg-3Y-3.5Sm-2Zn alloy and M salt addition temperature. It is obvious that the average grain size of the alloy decreases first and then increases with the increase of the M salt addition temperature, and the smallest average grain size of the alloy is obtained when M3 salt is added to the melt at 780 °C (24.93 μm).

Fig.9 shows the morphologies of element Zr in the as-cast Mg-3Y-3.5Sm-2Zn alloys refined by M1, M2 and M3 salts at 780 °C. EDS results of points A, B, and C in Fig.9 are shown in Table 3. It can be seen that there are different shapes of zirconium-rich zones in the alloys under different conditions. There are large zirconium-rich zones in the alloy treated by M1 salt, as shown in Fig.9a. The edges of these zones are bright white and the rest parts are gray. It can be seen from Fig.9b that the morphology of zirconium-rich zone in the alloy treated by M2 salt is bright white triangular aggregation. Compared with Fig.9a and 9b, the morphology of zirconium-rich zones in the

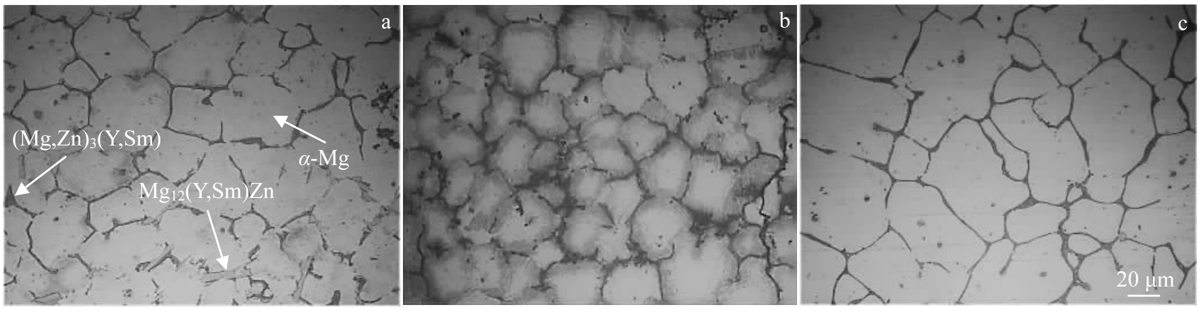

Fig.6 Microstructures of the as-cast Mg-3Y-3.5Sm-2Zn alloy refined by M2 at different temperatures: (a) 750 °C, (b) 780 °C, and (c) 810 °C

Fig.7 Microstructures of the as-cast Mg-3Y-3.5Sm-2Zn alloy refined by M3 at different temperatures: (a) 750 °C, (b) 780 °C, and (c) 810 °C

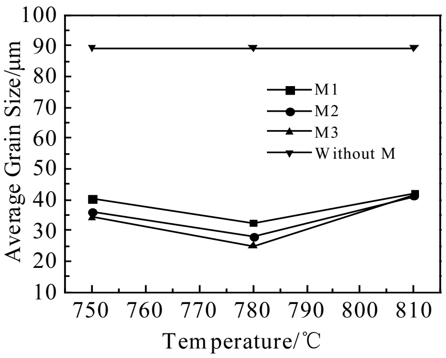

Fig.8 Relationship between the average grain size of the as-cast Mg-3Y-3.5Sm-2Zn alloy and M salt addition temperature

alloy treated by M3 salt changes from agglomeration to dispersion.

Fig.10 shows SEM image and distribution of the element Mg, Y, Sm, Zn, Zr of the as-cast Mg-3Y-3.5Sm-2Zn alloy refined by M3 salt at 780 °C. As shown in Fig.10, there are two distribution states of Zr in the alloy: Zr disperses in the matrix as the fine Zr particle; Zr distributes on the matrix in the form of zirconium-rich zone. Element Zr plays a role of grain refinement in the as-cast Mg-3Y-3.5Sm-2Zn alloy through these two ways.

Fig.11 shows the morphologies of inclusion in the as-cast Mg-3Y-3.5Sm-2Zn alloy. It can be seen that there are two kinds of inclusion morphologies in the alloy. One is the bright white flocculent aggregate, and the other is the gray massive aggregate. EDS results of points D and E in Fig.11 (shown in Table 3) show that these aggregates mainly contain O, F, Mg, Y, Sm, Zn and Zr, indicating that those agglomerates may be composed of oxides and fluorides. In addition, the mass percentage of element Y in these aggregates is high. This is due to the reaction of K_2ZrF_6 in M salt with Y to form YF and KF, which leads to the formation of those aggregates and the consumption of element Y in the alloy. Therefore, the formation of these inclusions is unfavorable to the properties of the as-cast Mg-3Y-3.5Sm-2Zn alloy.

2.2 Mechanical properties

Fig.12 shows the effect of adding M salt at 780 °C on the mechanical properties of the as-cast Mg-3Y-3.5Sm-2Zn alloy. The ultimate tensile strength (UTS), the yield strength (YS), and the elongation (EL) were obtained based on the average of three tests. It can be seen that there are some differences in the mechanical properties of the experimental alloys. Alloy without M salt addition has the poorest mechanical properties, including UTS of 81.67 MPa, YS of 76.52 MPa and EL of 0.93%. Alloy treated by the M3 salt has the best mechanical properties, including UTS of 184.00 MPa, YS of 147.24 MPa and EL of 2.35%. The improvement of mechanical properties

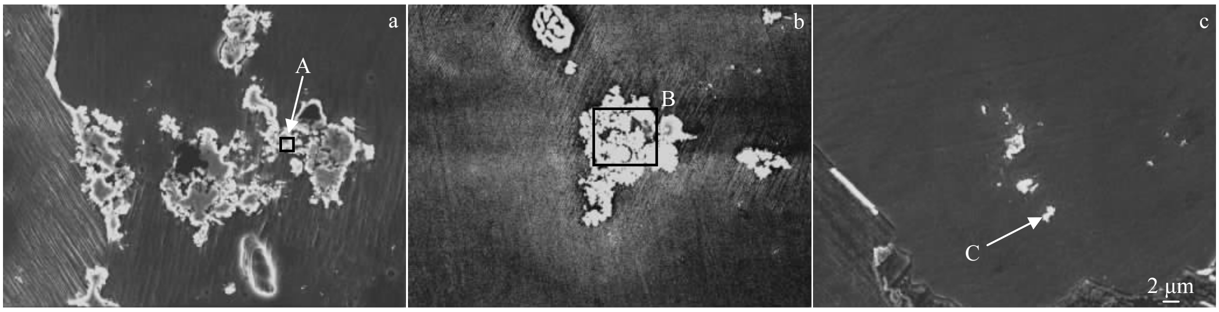

Fig.9 Morphologies of Zr in the as-cast Mg-3Y-3.5Sm-2Zn alloys refined by M1 (a), M2 (b), and M3 (c) at 780 °C

Table 3 EDS analysis results of different phases marked in Fig.9 and Fig.11 (wt%)

Point	Mg	Y	Sm	Zn	Zr	O	F	Na	Cl	K
A	36.42	6.39	4.43	4.69	41.31	5.74	0.73	0.25	0.00	0.04
B	46.63	0.00	10.39	5.94	30.59	5.96	0.14	0.12	0.21	0.02
C	40.59	5.75	1.01	3.30	41.71	6.72	0.31	0.61	0.00	0.00
D	28.71	36.47	6.90	2.86	4.21	18.71	1.71	0.19	0.17	0.07
E	9.44	43.10	5.84	0.26	19.74	18.65	2.97	0.00	0.00	0.00

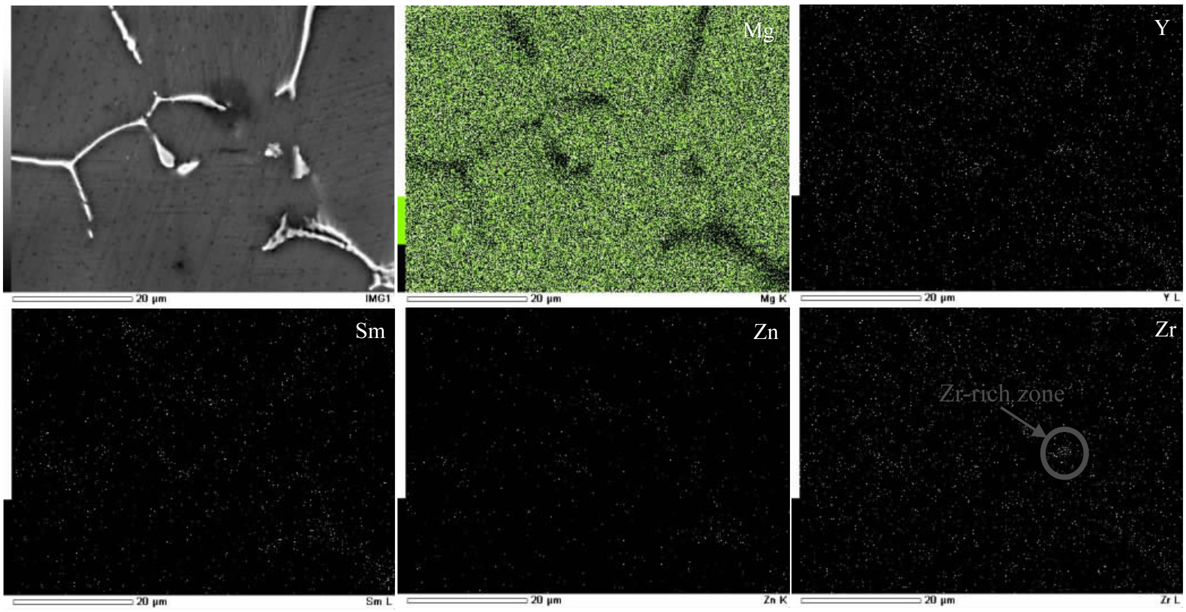

Fig.10 SEM image and distribution of the element Mg, Y, Sm, Zn, Zr of the as-cast Mg-3Y-3.5Sm-2Zn alloy refined by M3 salt at 780 °C

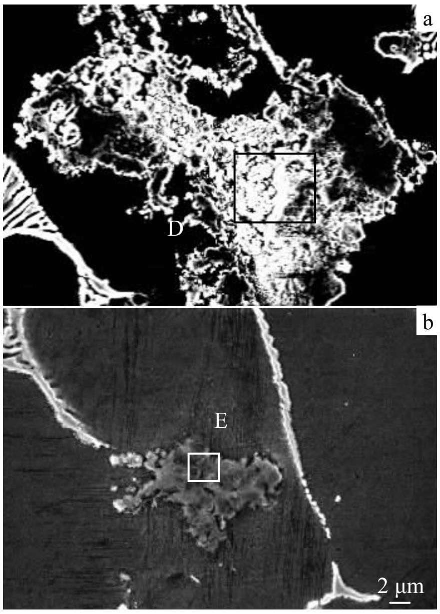

Fig.11 Morphologies of inclusion in the as-cast Mg-3Y-3.5Sm-2Zn alloy

is attributed to the element Zr which plays a role of fine grain strengthening in the as-cast Mg-3Y-3.5Sm-2Zn alloy.

3 Grain Refinement Mechanism Discussion

The peritectic mechanism proposed by Emley for magnesium alloys with Zr considers that the most significant feature of zirconium-containing magnesium alloys is the existence of zirconium-rich rings in the center of most grains in the microstructure[23]. However, no zirconium-rich ring appears in the microstructure of the experimental alloys under the experimental condition presented in this work. The formation of zirconium-rich rings is greatly related to the adding amount of Zr. According to Mg-Zr phase diagram[24], a peritectic reaction occurs at 650 °C and the maximum solubility of Zr in magnesium alloy is 0.6% at the peritectic temperature. In the present work, the addition of Zr in the experimental alloys is 0.6%, which means that the content of Zr in the melt is just equal to the saturated concentration of Zr in the equilibrium condition. In addition, it can be seen from Fig.9 that different forms of zirconium-rich zones are found when M salt is added to as-cast Mg-3Y-3.5Sm-2Zn alloy, indicating that the distribution of element Zr in the alloy is not

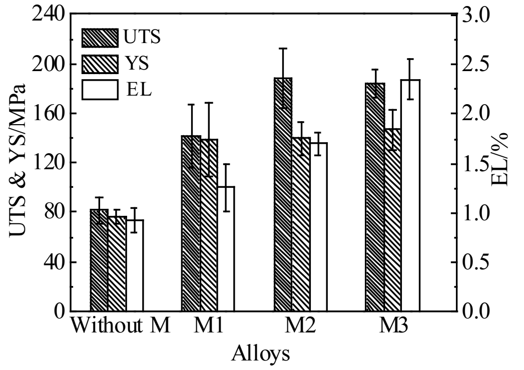

Fig.12 Effect of adding M salt at 780 °C on mechanical properties of the as cast Mg-3Y-3.5Sm-2Zn alloys

uniform, thus reducing the possibility of the occurrence of zirconium-rich ring in the matrix. Therefore, no zirconium-rich ring is found in this work.

Although zirconium-rich rings are not found in the as-cast Mg-3Y-3.5Sm-2Zn alloy, there are two distribution states of element Zr in the alloy: Zr disperses in the matrix as the fine Zr particle; Zr distributes on the matrix in the form of zirconium-rich zone, as can be seen from Fig.10. This is attributed to the reduction reaction between K_2ZrF_6 in M salt and Mg melt, as mentioned earlier. The specific reduction reaction is as follows:

It can be seen from the phase diagram of KF-ZrF_4 system that K_2ZrF_6 decomposes into K_3ZrF_7 and liquid phase $KZrF_5$ when the temperature is above 590 °C as Eq. (1)[25]:

$$2K_2ZrF_6 \rightarrow K_3ZrF_7 + L(KZrF_5) \quad (1)$$

The reaction between L($KZrF_5$) and Mg can produce intermediate products[26]. The specific reaction is shown as Eq.(2):

$$6KZrF_5+12Mg \Leftrightarrow 3K_2ZrF_6+6MgF_2+3Zr+6Mg \Leftrightarrow$$
$$2K_3ZrF_7+8MgF_2+4Mg+4Zr \Leftrightarrow 6KF+12MgF_2+6Zr \quad (2)$$

In the K_2ZrF_6-NaCl-KCl system, NaCl and KCl have high chemical stability and do not react with the magnesium liquid or element Zr. Therefore, K_2ZrF_6 can be restored by the liquid magnesium under the protection of NaCl-KCl salts system. The total reaction is shown as Eq.(3):

$$K_2ZrF_6+2Mg \rightarrow Zr+2KF+2MgF_2 \quad (3)$$

According to the results obtained from this work, those three reactions occur when M is added to the as-cast Mg-3Y-3.5Sm-2Zn alloy.

4 Conclusions

1) K_2ZrF_6 salt mixture can refine the grain size of the as-cast Mg-3Y-3.5Sm-2Zn alloy and improve its mechanical properties. Considering the grain size of the alloy refined by M1 salt (40 wt% K_2ZrF_6-30 wt% NaCl-30 wt% KCl), M2 salt (50 wt% K_2ZrF_6-25 wt% NaCl-25 wt% KCl), and M3 salt (60 wt% K_2ZrF_6-20 wt% NaCl-20 wt% KCl) at different temperatures, the optimum addition temperature of M salt is 780 °C.

2) M3 salt is the best grain refiner at 780 °C, and the grain size can be reduced to 24.93 μm. The alloy refined by M3 salt at 780 °C has the best mechanical properties (UTS: 184 MPa; YS: 147.24 MPa; EL: 2.35%).

3) The grain refinement mechanism of M salt in the as-cast Mg-3Y-3.5Sm-2Zn alloy is attributed to the grain refining effect of fine Zr particles and zirconium-rich zones obtained by the reduction reaction between Mg and K_2ZrF_6.

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氟锆酸钾盐混合物对 Mg-3Y-3.5Sm-2Zn 合金组织和性能的影响

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摘　要：制备了 3 种氟锆酸钾(K_2ZrF_6)盐混合物(M)，分别在 3 个温度下将 3 种盐混合物加入到铸态 Mg-3Y-3.5Sm-2Zn 合金中。通过差热分析(DSC)，X 射线衍射(XRD)，光学显微镜(OM)，扫描电子显微镜(SEM)，能量色散谱(EDS)和拉伸试验研究了合金的显微组织和力学性能，讨论了 K_2ZrF_6 盐混合物在合金中的晶粒细化机理。结果表明，铸态 Mg-3Y-3.5Sm-2Zn 合金由 α-Mg，$Mg_{12}(Y, Sm)Zn$ 和 $(Mg, Zn)_3(Y, Sm)$ 3 种相组成。SEM 和 XRD 检测发现，添加 K_2ZrF_6 盐混合物不会在合金中产生新的相。在 780 ℃下加入 M3 盐(60% K_2ZrF_6-20% NaCl-20% KCl，质量分数)时，合金的晶粒细化效果最佳。通过在 780 ℃下加入 3 种盐，铸态 Mg-3Y-3.5Sm-2Zn 合金的力学性能得到提高，由 M3 盐细化后的合金具有最佳的力学性能。K_2ZrF_6 盐混合物可细化铸态 Mg-3Y-3.5Sm-2Zn 合金，这是由于 Mg 和 K_2ZrF_6 之间还原反应得到的细 Zr 质点和富锆区在合金中起到的晶粒细化作用。

关键词：镁合金；氟锆酸钾盐(K_2ZrF_6)混合物；晶粒细化；显微组织；力学性能

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