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Materials

Effects of Sr on the microstructure, tensile and creep properties of AZ61-0.7Si magnesium alloy

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Abstract: The modification and refinement of Mg_2Si phase is thought to be one of the key aspects to improve the mechanical properties of Si-containing magnesium alloys. In this article, the effects of Sr on the microstructure, tensile and creep properties of AZ61-0.7Si magnesium alloy were investigated. The results indicate that adding small amounts of Sr to AZ61-0.7Si alloy can modify and refine Chinese script shaped Mg_2Si phases in the alloy. After adding 0.03wt%-0.09wt% Sr to AZ61-0.7Si alloy, the Mg_2Si phases in the alloy change from the coarse Chinese script shape to fine granule and/or irregular polygonal shapes. The modification and refinement mechanisms of Mg_2Si phases in Sr-containing AZ61-0.7Si alloys are possibly related to the reduction of growth rate and the enhancement of nucleation ratio for Mg_2Si particles during the solidification process. Owing to the modification and refinement of Mg_2Si phases, the tensile and creep properties of Sr-containing AZ61-0.7Si alloys are greatly improved.

Key words: magnesium alloy; Mg_2Si phase; Sr addition; mechanical properties; microstructure

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1. Introduction

Magnesium alloys are the lightest structural alloys commercially available and have great potential for applications in automotive, aerospace, and other industries. However, in recent years, improving the elevated temperature properties has become a critical issue for possible application of magnesium alloys in hot components. It has been seen that Mg-Al-Si based alloys are potential elevated temperature magnesium alloys [1-2], because Mg_2Si phase in Mg-Al-Si based alloys has high melting point, high hardness, low density, high elastic modulus, and low thermal expansion coefficient, and Mg_2Si phase is very stable and can impede grain boundary sliding at elevated temperatures [3]. However, under the lower solidification rates, Mg-Al-Si based alloys easily form undesirable, coarse, Chinese script shaped Mg_2Si phases, which will damage the mechanical properties of the alloys. Therefore, the modification and refinement of Mg_2Si phase are thought to be one of the pivotal factors to improve the mechanical properties of Mg-Al-Si based alloys.

Owing to the above-mentioned reason, the research on the modification and refinement of Mg_2Si phases in Mg-Al-Si based alloys by microalloying method has received considerable attention all over the world, and consequently, several researches have been carried out. It has been reported that Chinese script shaped Mg_2Si phases in Mg-Al-Si based alloys could be modified and refined by Sb [4-6], Ca, and P additions [7-9]. However, some researches also found that Sb was not an effective modifier of Mg_2Si phase [7], Ca resulted in cast defects such as hot-cracks [10], P addition produced ignition, and the amount of P addition was difficult to control [4]. Therefore, other microalloying elements for the modification and refinement of Chinese script shaped Mg_2Si phases need to be considered. Recent results indicated that the Sr element that has been used in industrial practice especially for the modification of Al-Si alloys [11-12], was an effective modifier and refiner for Chinese script shaped Mg_2Si

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phases in Mg-Al-Si based alloys [13-14]. For example, Srinivasan *et al*. [13] reported that the addition of Sr to a Si-containing AZ91-Mg alloy appeared to refine the microstructure by promoting a smaller grain size, and the coarse Chinese script Mg_2Si precipitates also appeared to be smaller and more uniformly distributed. Similar results were obtained by Song *et al*. [14]. In spite of the above research, the investigation about the effects of Sr on the microstructure and mechanical properties of Mg-Al-Si based alloys, especially about the effects of Sr on the modification and refinement of Chinese script shaped Mg_2Si phase in the alloys, is very limited. In order to provide a theoretical guide of Sr microalloying for Mg-Al-Si based alloys, the present study investigated the effects of Sr on the microstructure, tensile and creep properties of an AZ61-0.7Si magnesium alloy, especially on the modification and refinement of Mg_2Si phases in the alloy.

2. Experimental procedures

An AZ61-0.7Si alloy was prepared by adding the following materials: commercial AM60 alloy, pure Al, pure Mg, pure Zn, and a Al-30%Si master alloy. The experimental alloy was melted in a crucible resistance furnace and protected by a flux addition. When the melt temperature reached 740℃, the melts were respectively treated with 0.03wt%, 0.06wt%, and 0.09wt% Sr using an Al-10Sr master alloy. After held at 740℃ for 60 min, the melts were homogenized by mechanical stirring and then poured into a permanent mould, which was coated and preheated to 200℃ in order to obtain a casting. The specimens as shown in Fig. 1 were fabricated from the casting for tensile and creep tests. For comparison, an AZ61-0.7Si-0.4Sb alloy was also cast and machined into the same dimensions and tested under the same conditions as the above samples. The actual chemical compositions of the experimental alloys are listed in Table 1.

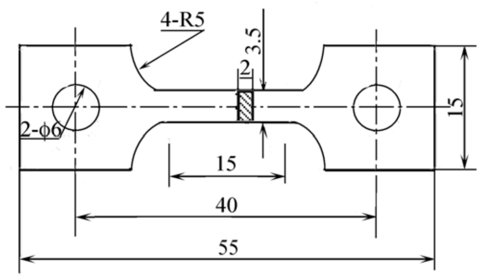

Fig. 1. Sample used for tensile and creep tests (unit: mm).

Table 1. Actual compositions of the experimental alloys **wt%**

Experimental alloy	Al	Zn	Mn	Si	Sb	Sr	Mg
AZ61-0.7Si	5.92	0.79	0.24	0.68	—	—	Bal.
AZ61-0.7Si-0.4Sb	5.89	0.82	0.23	0.67	0.40	—	Bal
AZ61-0.7Si-0.03Sr	5.91	0.83	0.21	0.66	—	0.027	Bal.
AZ61-0.7Si-0.06Sr	5.90	0.81	0.24	0.68	—	0.058	Bal.
AZ61-0.7Si-0.09Sr	5.92	0.85	0.22	0.69	—	0.086	Bal.

The microstructure analysis samples were etched with an 8% nitric acid distilled water solution, and then were examined by Joel/JSM-6460LV type scanning electron microscope (SEM) equipped with Oxford energy dispersive spectrometer (EDS). The phases in the experimental alloys were analyzed by D/Max-1200X type X-ray diffraction (XRD) operated at 40 kV and 30 mA. The differential scanning calorimetry (DSC) was also carried out using a NETZSCH STA 449C system. Samples weighted about 30 mg were heated in a flowing argon atmosphere ranging from 30 to 700℃ for 5 min before being cooled down to 100℃. The cooling curve was recorded at a controlling speed of 15℃/min.

The tensile properties at room temperature and 150℃ were determined from a complete stress-strain curve. The 0.2% yield strength (YS), ultimate tensile strength (UTS), and elongation to failure (elongation) were obtained based on the average of three tests. The constant-load tensile creep tests were performed at 150℃ and 50 MPa for creep extension up to 100 h. The total creep strain and minimum creep rate were measured from each elongation-time curve and averaged over three tests, respectively.

3. Results and discussion

3.1. As-cast microstructure

Fig. 2 shows the XRD results of AZ61-0.7Si-0.09Sr and AZ61-0.7Si-0.4Sb alloys. As seen in Fig. 2(a), the AZ61-0.7Si-0.09Sr alloy is mainly composed of α-Mg, $Mg_{17}Al_{12}$, and Mg_2Si phases. It is well known that the AZ61-0.7Si alloy is also composed of α-Mg, $Mg_{17}Al_{12}$, and Mg_2Si phases. Then, it is inferred that adding 0.09wt% Sr to the AZ61-0.7Si alloy does not cause the formation of any other new phases. This result is consistent with that of Zhao *et al*. [15]. However, adding 0.4wt% Sb to AZ61-0.7Si alloy will form small amounts of Mg_3Sb_2 phase, as seen in Fig. 2(b).

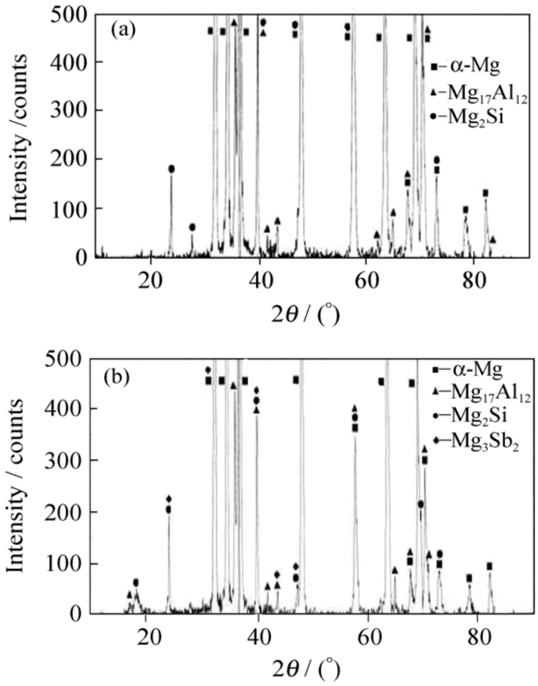

Fig. 2. XRD results of different alloys: (a) AZ61-0.7Si-0.09Sr; (b) AZ61-0.7Si-0.4Sb.

Figs. 3 and 4 show the SEM images of experimental alloys. Combining with EDS results (Table 2), it is found from Fig. 3 that Mg_2Si phases in the AZ61-0.7Si alloy exhibit coarse Chinese script shaped morphology. After adding 0.4wt% Sb to AZ61-0.7Si alloy, although the Chinese script shaped Mg_2Si phases in the alloy are still obvious, they become relatively fine, as seen in Fig. 4(a). The result is consistent with that of Quimby *et al*. [7], but it is contradictory to the results from Yuan *et al*. [4]. Further investigation is needed to understand the reason for the difference. At the same time, it is interestingly observed from Figs. 4(b)-(d) that after adding small amounts of Sr to the AZ61-0.7Si alloy, the Mg_2Si phases in the alloy become fine, and their morphology changes from the Chinese script shape to granule and/or irregular polygonal shapes, indicating that adding small amounts of Sr can modify and refine the Chinese script shaped Mg_2Si phase. Furthermore, it is found that adding 0.06wt% or 0.09wt% Sr appears to have a relatively higher modification and refinement efficiency. In addition, based on solid solution treatment at 400℃ for 12 h followed by water quenching, it is found that adding small amounts of Sr to the AZ61-0.7Si alloy can refine the as-cast microstructure of the alloy by promoting a smaller grain size, which is consistent with the previous results [16-22]. Considering that considerable investigation in this aspect has been carried out in Refs. [16-22], and the present study mainly focuses on the modification and refinement of Mg_2Si phase, therefore, the effect of Sr on the grain refinement of the AZ61-0.7Si alloy is not discussed in this article.

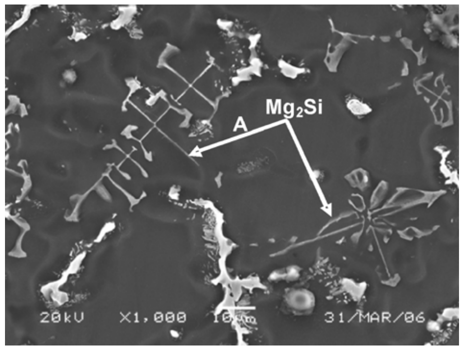

Fig. 3. SEM image of the AZ61-0.7Si alloy.

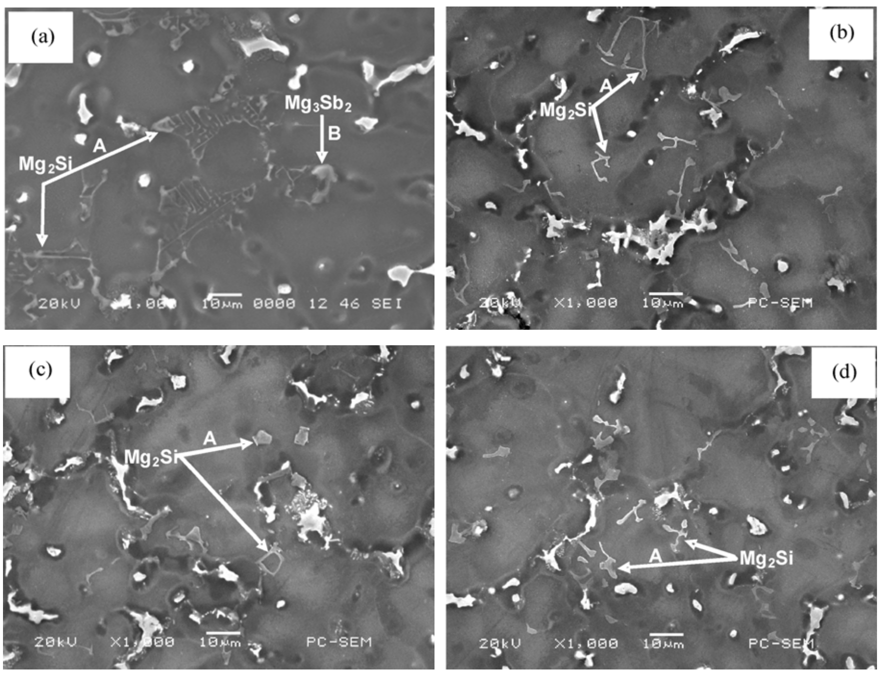

Fig. 4. SEM images of the experimental alloys: (a)AZ61-0.7Si-0.4Sb; (b) AZ61-0.7Si-0.03Sr; (c) AZ61-0.7Si-0.06Sr; (d) AZ61-0.7Si -0.09Sr.

Table 2. EDS results of the experimental alloys at%

Position	Mg	Al	Si	Sr	Sb	Total
Fig. 3-A	65.96	2.09	31.95	—	—	100
Fig. 4(a)-A	63.96	3.73	31.68	—	0.63	100
Fig. 4(a)-B	56.15	5.23	25.42	—	13.2	100
Fig. 4(b)-A	67.35	2.31	30.03	0.31	—	100
Fig. 4(c)-A	70.25	2.64	26.83	0.28	—	100
Fig. 4(d)-A	65.40	2.50	31.78	0.32	—	100

3.2. Mechanical properties

The tensile properties including ultimate tensile strength (UTS), 0.2% yield strength (YS), elongation (δ), and creep properties of experimental alloys are listed in Table 3. For comparison, the tensile and creep properties of the AZ61-0.7Si-0.4Sb alloy are also listed in Table 3. It is observed from Table 3 that the tensile and creep properties of the Sr-containing AZ61-0.7Si alloys are higher than those of the AZ61-0.7Si alloy, indicating that adding small amounts of Sr can improve the mechanical properties of the AZ61-0.7Si alloy. This situation is possibly related to the modification and refinement of Mg_2Si phases and grain refinement. Similarly, the AZ61-0.7Si-0.4Sb alloy also exhibits higher tensile and creep properties than the AZ61-0.7Si alloy owing to the refinement of Mg_2Si phases in the alloy. However, the elongation of the AZ61-0.7Si-0.4Sb alloy is relatively lower than that of the Sr-containing AZ61-0.7Si alloys. This situation is possibly related to the uncomplete modification of Mg_2Si phases in the AZ61-0.7Si-0.4Sb alloy. 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 mechanical properties of engineering alloys [23]. Apparently, Chinese script shaped Mg_2Si phases in the AZ61-0.7Si and AZ61-0.7Si-0.4Sb alloys will provide a detrimental effect on the mechanical properties of the two alloys since long cracks can easily nucleate along the interface between Chinese script shaped Mg_2Si particles and α-Mg matrix [8]. Conversely, after Sr microalloying, Mg_2Si phases in the Sr-containing AZ61-0.7Si alloys change from the coarse Chinese script shape to the fine granule and/or irregular polygonal shapes, and then the extending trend of microcracks will decrease. Accordingly, the mechanical properties of the Sr-containing AZ61-0.7Si alloys are improved, especially elongation. The situation can be further confirmed from Fig. 5. As seen in Figs. 5(a) and 5(b), the fractographs of the AZ61-0.7Si and AZ61-0.7Si-0.4Sb alloys show that their fracture surfaces exhibit relatively large cleavage-type facets (arrow 'A' in Figs. 5(a) and 5(b)), which presumably form along the interface between Chinese script shaped Mg_2Si particles and α-Mg matrix. On the other hand, in the fracture surface of the AZ61-0.7Si-0.09Sr alloy, the cleavage-type facets are relatively fine (arrow 'A' in Fig. 5(c)). In addition, owing to the higher modification and refinement efficiency of Mg_2Si phases in the AZ61-0.7Si-0.06Sr or AZ61-0.7Si-0.09Sr alloys, the alloys exhibit relatively higher tensile and creep properties than the AZ61-0.7Si-0.03Sr alloy, as listed in Table. 3.

Table 3. As-cast tensile and creep properties of the experimental alloys

Experimental alloy	Tensile properties						Creep properties	
	Room temperature			150℃			150℃ and 50 MPa for 100 h	
	UTS / MPa	YS / MPa	δ / %	UTS / MPa	YS / MPa	δ / %	Total creep strain / %	Minimum creep rate / (10^{-3} %·h^{-1})
AZ61-0.7Si	147	77	4	140	70	12	0.54	5.11
AZ61-0.7Si-0.4Sb	175	99	5	160	91	16	0.47	4.56
AZ61-0.7Si-0.03Sr	176	104	5.2	166	97	16.8	0.46	4.51
AZ61-0.7Si-0.06Sr	182	113	5.8	172	108	17.9	0.42	4.36
AZ61-0.7Si-0.09Sr	184	115	5.9	173	108	18.1	0.42	4.24

3.3. Discussion

Generally, Mg_2Si phases in the Mg-Al-Si based alloys unmodified are prone to forming the coarse Chinese script shape under lower solidification rates [1-2]. Therefore, under the present experimental conditions, Mg_2Si phases in the AZ61-0.7Si alloy exhibit typical Chinese script shaped morphology (Fig. 3). However, after Sr microalloying, Mg_2Si phases in the Sr-containing AZ61-0.7Si alloys change from the Chinese script shape to fine granule and/or irregular polygonal shapes (Figs. 4(b)-4(d)). Previous investigation showed that when the microalloying method was adopted, the modification and/or refinement of Mg_2Si phases in the Si-containing magnesium alloys were mainly related to the forming of nuclei for Mg_2Si precipitates. For example, Yuan *et al*. [4] reported that after adding 0.5wt% Sb to a Mg-5Al-1Zn-1Si alloy, the Mg_3Sb_2 particle that could act as a nucleus for Mg_2Si phases would form in the alloy, and then a morphology change in Mg_2Si particles occurred from the coarse Chinese script shape to a small polygonal type. In addition, Kim *et al*. [8] reported that after adding Ca and P to the AZ61-0.79Si alloy, the $CaSi_2$ and $Mg_3(PO_4)_2$ particles that could act as nuclei for Mg_2Si phases would also form in the alloy. However, according to the above information from XRD and EDS results, in the present study, adding small amounts of strontium (<0.09wt%) to the AZ61-0.7Si alloy does not cause the formation of any other new phases, indicating that the above-mentioned mechanism is not suitable for the modification and refinement of Mg_2Si phases in the Sr-containing AZ61-0.7Si alloys. Fig. 6 shows the cooling curves of AZ61-0.7Si and AZ61-0.7Si-0.09Sr alloys and Fig. 7 shows the surface scanning results of the AZ61-0.7Si-0.09Sr alloy. It is found from Fig. 7 and Table 2 that Sr not only exists in the α-Mg matrix but also incorporated in Mg_2Si precipitates. In addition, it is observed from the cooling curves of the AZ61-0.7Si and AZ61-0.7Si-0.09Sr alloys (Fig. 6) that after Sr microalloying, the onset crystallizing temperature of the AZ61-0.7Si alloy, T_l, decreases from 608.8 to 600.4℃. According to the classic solidification theory, the relationship between the critical nucleus radius and the undercooling degree is given as follows [21]:

$$r^* = \frac{2\sigma}{\Delta G_r} = \frac{2\sigma T_m}{L_m \Delta T} \quad (1)$$

where, r^* is the critical nucleus radius, ΔG_r the variation of volume free energy, σ the interfacial energy of unit surface area, T_m the equilibrium crystallizing temperature, L_m the crystallizing latent heat, and ΔT the undercooling degree, which can be expressed as $\Delta T = T_m - T_l$. According to Eq. (1), the critical nucleus radius decreases with the decrease of T_l, and then the nucleation energy of crystal nuclei reduces and the probability of nucleation increases, which result in grain and precipitate refinement. Based on the above analysis, the possible reason for the modification and refinement of Mg_2Si phases in the Sr-containing AZ61-0.7Si alloys may be mainly related to the following two aspects: (1) owing to the limited solid solubility of Sr in magnesium, redundant Sr will enrich in the liquid ahead of the Mg_2Si growing interface, which will restrict Mg_2Si growth during the solidification process; (2) Sr microalloying increases the undercooling degree, which will result in the increase of the effective number of potential Mg_2Si crystal nuclei. However, the exact reason is not completely clear. It is a subject for further study in our group.

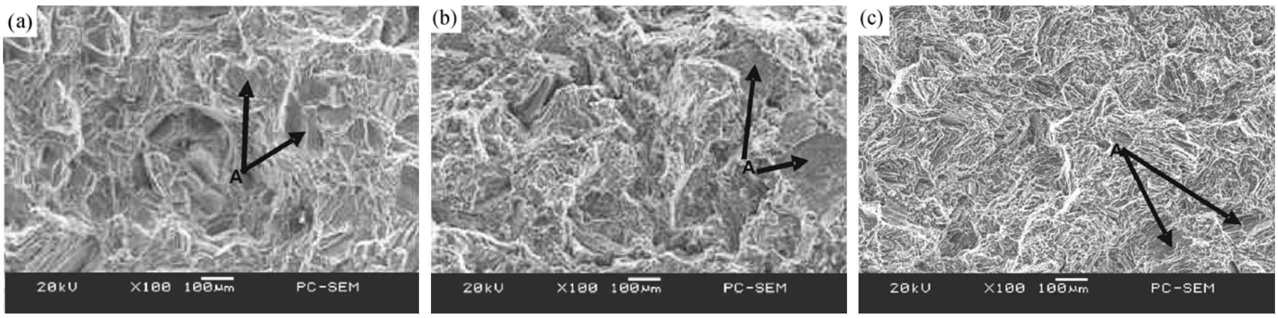

Fig. 5. SEM fractographs of the experimental alloys: (a) AZ61-0.7Si; (b) AZ61-0.7Si-0.4Sb; (c) AZ61-0.7Si-0.09Sr.

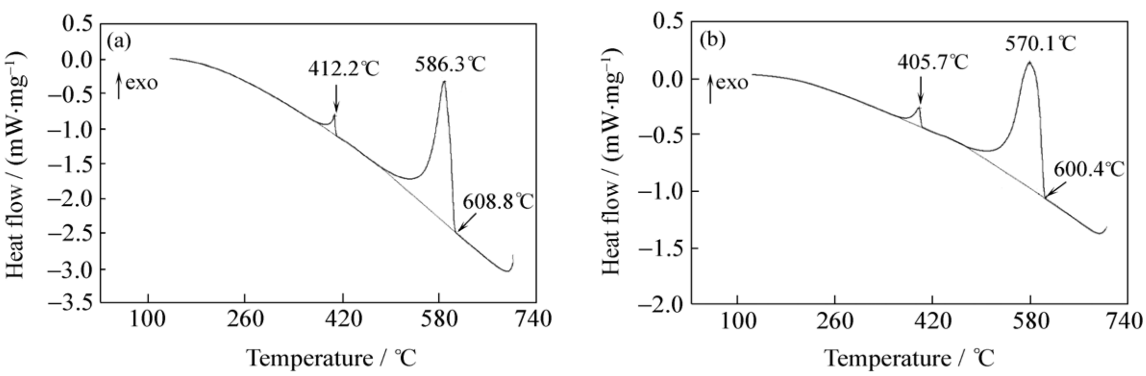

Fig. 6. DSC cooling curves of the experimental alloys: (a) AZ61-0.7Si; (b) AZ61-0.7Si-0.09Sr.

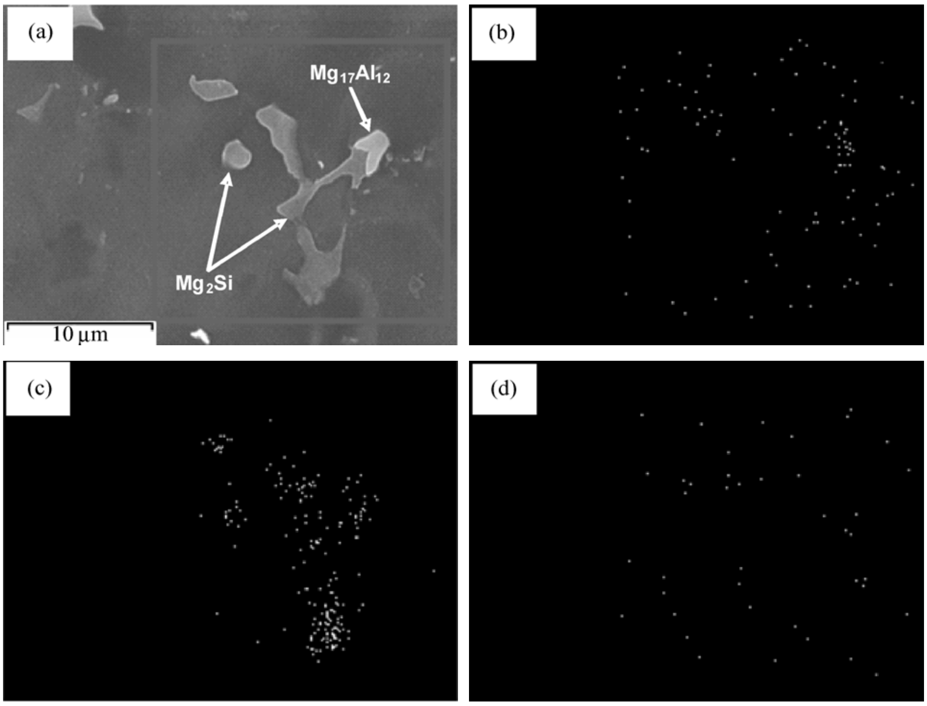

Fig. 7. Surface scanning results of the AZ61-0.7Si-0.09Sr alloy: (a) SEM image; (b) mapping of Al element; (c) mapping of Si element; (d) mapping of Sr element.

4. Conclusion

The results indicate that adding small amounts of Sr to the AZ61-0.7Si alloy can modify and refine Chinese script shaped Mg_2Si phases in the alloys. After adding 0.03wt%-0.09wt% Sr to the AZ61-0.7Si alloy, Mg_2Si phases in the alloy change from the coarse Chinese script shape to fine granule and/or irregular polygonal shapes. The modification and refinement mechanisms of Mg_2Si phases in the Sr-containing AZ61-0.7Si alloys are possibly related to the reduction of growth rate and the enhancement of nucleation ratio for Mg_2Si particles during the solidification process. Owing to the modification and refinement of Mg_2Si phases, the tensile and creep properties of the Sr-containing AZ61-0.7Si alloys are greatly improved.

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