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Modification and refinement effects of Sb and Sr on $Mg_{17}Al_{12}$ and Mg_2Si phases in Mg-12Al-0.7Si alloy

Zhi-wen Wang [1,2], * Hong-xia Wang [1,2], Jia-lin Gong [1,2], Ming Li [1,2], Wei-li Cheng [1,2], and Wei Liang [1,2]

1. Shanxi Key Laboratory of Advanced Magnesium Based Materials, College of Materials Science and Engineering, Taiyuan University of Technology, Taiyuan 030024, Shanxi, China

2. Key Laboratory of Interface Science and Engineering in Advanced Materials, Taiyuan University of Technology, Ministry of Education, Taiyuan 030024, Shanxi, China

Abstract: The effects of Sb and Sr on the modification and refinement of $Mg_{17}Al_{12}$ and Mg_2Si phases in Mg-12Al-0.7Si alloy were investigated and compared. The microstructure and mechanical properties of Mg-12Al-0.7Si alloy and its modification mechanism by Sb and Sr were investigated using a scanning electron microscope (SEM), an energy dispersive spectrometer (EDS), X-ray diffraction (XRD) and differential thermal analysis (DTA). The results indicate that by adding 0.5wt.% Sb to the Mg-12Al-0.7Si alloy, the $Mg_{17}Al_{12}$ phase was refined and broken into some discontinuous island structures. However, some network $Mg_{17}Al_{12}$ phases still can be detected in Mg-12Al-0.7Si-0.09Sr alloy. Therefore, Sb performs better in modification and refinement of $Mg_{17}Al_{12}$ phase than does Sr. Small amounts of fine polygonal shaped Mg_2Si phases were found in Mg-12Al-0.7Si-0.5Sb alloy, while the morphology of Mg_2Si phases in Mg-12Al-0.7Si-0.09Sr alloy changed from the coarse Chinese script shapes to fine granule and irregular polygonal shapes, indicating that the effects of modification and refinement on Mg_2Si phase are more significant by adding 0.09wt.% Sr than 0.5wt.% Sb. The ultimate tensile strengths of the Sb and Sr modified Mg-12Al-0.7Si alloys were considerably increased both at room temperature and at 200 ºC.

Key words: magnesium alloy; Sb; Sr; $Mg_{17}Al_{12}$ phase; Mg_2Si phase

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Magnesium alloys, as the lightest structural metal, show great potential in the automotive, aerospace and electronics industries due to their perfect combination of properties, including low density, high specific strength and specific stiffness, super damping capacity and good castability [1-4]. Among all the magnesium alloys, Mg-Al alloys are most widely used due to their low cost and good mechanical properties at room temperature [5]. However, when the content of Al exceeds 9wt.%, the β-$Mg_{17}Al_{12}$ phase with low melting point tends to form a network along the grain boundaries, which is detrimental to the mechanical properties. Previous study suggested that the network structure of β-$Mg_{17}Al_{12}$ phase in high-Al magnesium alloys (Al>9%), such as Mg-15Al and Mg-12Al, can be broken by the equal channel angular pressing (ECAP), making the plasticity and strength increase simultaneously [6-7]. In addition, with the increase of Al content, the corrosion resistance of Mg-Al alloys was improved [8-9]. Therefore, it is of great significance to study how to improve the properties of high-Al Mg-Al alloys.

In order to improve the high temperature performance and creep resistance of Mg-Al alloys, alloying elements, such as Zr and Nd [10-11], are introduced so that they can form second phases with high thermal stability. Particularly, more attention has been paid to the low cost Mg-Al-Si alloys due to the existence of the Mg_2Si phase with high melting point, high hardness and low thermal expansion coefficient. However, the Mg_2Si phases in Mg-Al-Si-based alloys are prone to forming coarse Chinese script shapes under low solidification rates [12,13]. Hence, the key point is to modify the coarse Chinese script shape Mg_2Si phase in Mg-Al-Si alloys. Recent research [14-16] has indicated that Sr and Sb can assist in refining and modifying the Mg_2Si phase of magnesium alloys. But some disagreement regarding the Sb modification still exists. Yuan et al.[14] reported that the Mg_2Si phases changed from coarse Chinese script shapes to small polygons in Mg-5Al-1Zn-1Si-0.5Sb alloy due to the Mg_3Sb_2 particles formed in the alloy acting as nuclei for the Mg_2Si phase. Yang et al. [15] proposed that the addition of 0.4%Sb (mass fraction) to AZ61-0.7Si alloy can refine but not modify the Mg_2Si

*** Hong-xia Wang**

Female, born in 1974, Ph.D, Professor. Her research interests mainly focus on magnesium alloys.

E-mail: wanghxia1217@163.com

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phase. Alternatively, the Mg_2Si phase can be modified by the addition of 0.12%Sr to AZ61-0.7Si alloy. Srinivasan et al [16] reported that the coarse Chinese script Mg_2Si precipitates were refined by adding Sr to a Si-containing AZ91 Mg alloy. Previous study shows that both the best microstructural refinement and the highest mechanical properties (tensile strength and elongation) are obtained in Mg-10Al-0.5wt.%Sb [17] and in Sr-modified Mg-Al-Si alloys when content of Sr is 0.09wt.% [18-19]. However, to date, few investigations can be found concerning the Sr effects on high Al Mg-Al-Si alloys (Al>wt.9%). Especially, the modification and refinement mechanism of Sr on high Al magnesium alloys is not completely clear. Therefore, in the present study, the alloy Mg-12Al-0.7Si was modified with 0.5Sb and 0.09Sr. The modification and refinement effects of Sb and Sr on $Mg_{17}Al_{12}$ phase and Mg_2Si phase in Mg-12Al-0.7Si magnesium alloy, as well as the modification and refinement mechanism, are investigated.

1 Experimental procedure

Alloy ingots of about Φ40 mm × 200 mm were obtained by melting pure Mg (99.8wt.%), Al (99.9wt.%), Al-6wt.%Si master alloy, Sb (99.9wt.%) and Mg-30wt.%Sr master alloy in an electrical resistance furnace with a steel crucible, under a protective flux at melting temperature of about 700 ºC; and then poured into a steel mold, which was preheated to 280 ºC. The chemical compositions of these specimens were determined by inductively coupled plasma atomic emission spectroscopy (ICPAES) and are listed in Table 1. Tensile specimens with a gauge dimension of 13 mm×3.5 mm×1.5 mm were machined from as-cast billets. The tensile tests were carried out using a WDW-100KN machine at room temperature and 200 ºC, with a speed of 0.5 mm·min^{-1}. The samples for microstructure observation were ground and polished, following standard metallographic procedures, and etched at room temperature using a solution of 2 g picric acid, 10 ml $C_2H_6O_2$, 70 ml C_2H_5OH and 10 ml distilled water. The microstructures of the as-cast alloys were identified using a Leica DM2500M optical microscope (OM) and a JSM-6700F scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS). The phase constitutions were analyzed using TD-3000 X-ray diffraction (XRD).

Table 1: Chemical compositions of experimental alloys

Alloy	Al	Mn	Fe	Be	Si	Sb	Sr	Mg
Mg-12Al-0.7Si	12.87	0.27	0.22	0.05	0.723	-	-	Bal.
Mg-12Al-0.7Si-0.5Sb	12.74	0.21	0.40	0.05	0.724	0.48	-	Bal.
Mg-12Al-0.7Si-0.09Sr	12.91	0.19	0.25	0.05	0.723	-	0.092	Bal.

2 Results and discussion

2.1 Microstructures

Figure 1 shows the XRD patterns of the Mg-12Al-0.7Si, Mg-12Al-0.7Si-0.5Sb and Mg-12Al-0.7Si-0.09Sr alloys. It is well known that the Mg-12Al-0.7Si alloy is composed of α-Mg, $Mg_{17}Al_{12}$ and Mg_2Si phases. No new phase was found after adding 0.5wt.% Sb or 0.09wt.% Sr to Mg-12Al-0.7Si alloy, as shown in Fig. 1(b and c).

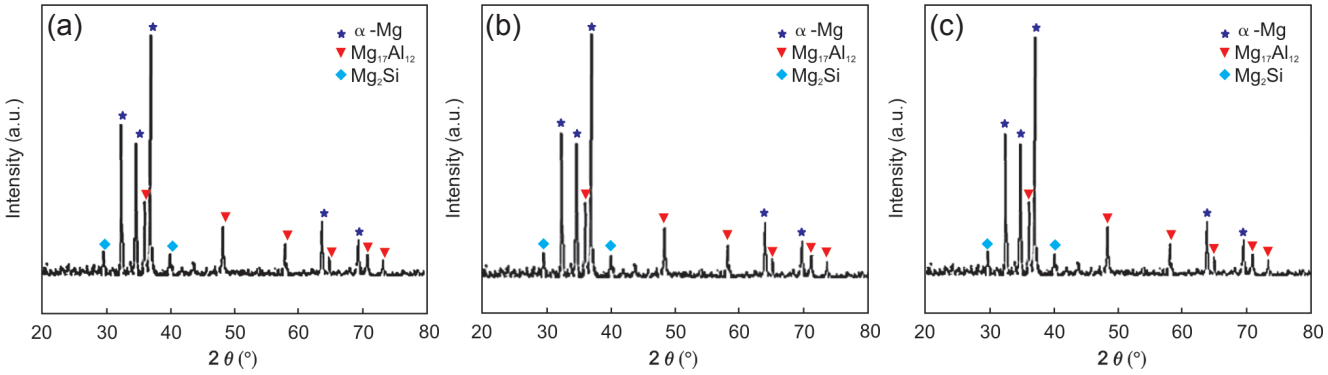

Fig. 1: XRD results of experimental alloys for (a) Mg-12Al-0.7Si alloy, (b) Mg-12Al-0.7Si-0.5Sb alloy, (c) Mg-12Al-0.7Si-0.09Sr alloy

Figures 2, 3 and 4 show the microstructures and EDS results of the experimental alloys. As shown in Fig. 2(a, b), the microstructure of Mg-12Al-0.7Si alloy consisted of primary Mg, coarse divorced eutectic $Mg_{17}Al_{12}$ particles and network $Mg_{17}Al_{12}$ precipitates at the grain boundaries, as well as coarse Chinese script-shaped Mg_2Si phase. Under low solidification rates the coarse Chinese script-shaped Mg_2Si phases form easily in Mg-12Al-0.7Si alloy [4,20], which would damage the mechanical properties of the alloys.

Figure 2(c) shows the micrograph of Mg-12Al-0.7Si-0.5Sb alloy. Figure 3 presents the results of EDS analysis of the regions marked in Fig. 2(c). Combining Fig. 2(c) with Fig. 3,

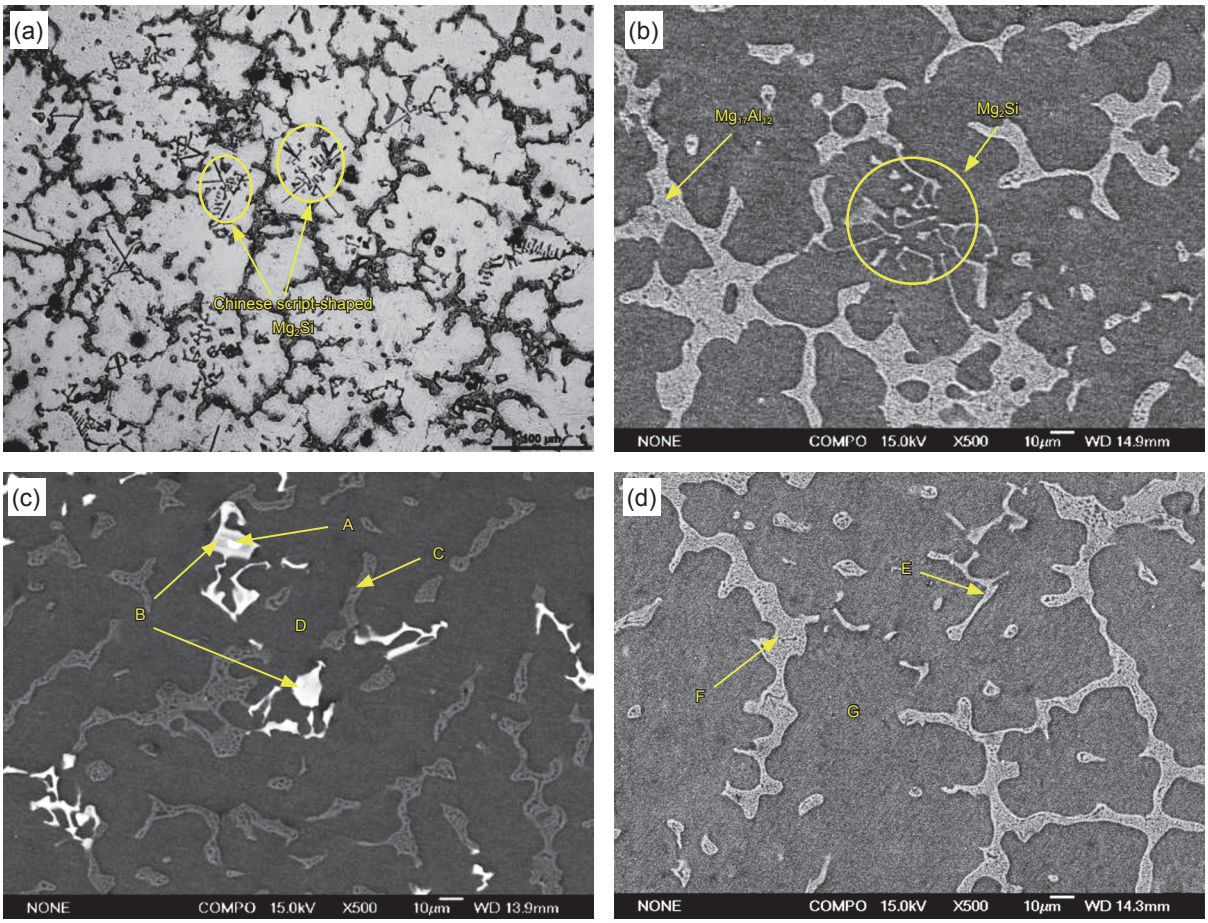

Fig. 2: SEM images of experimental alloys for Mg-12Al-0.7Si (a, b), Mg-12Al-0.7Si-0.5Sb (c), Mg-12Al-0.7Si-0.09Sr (d)

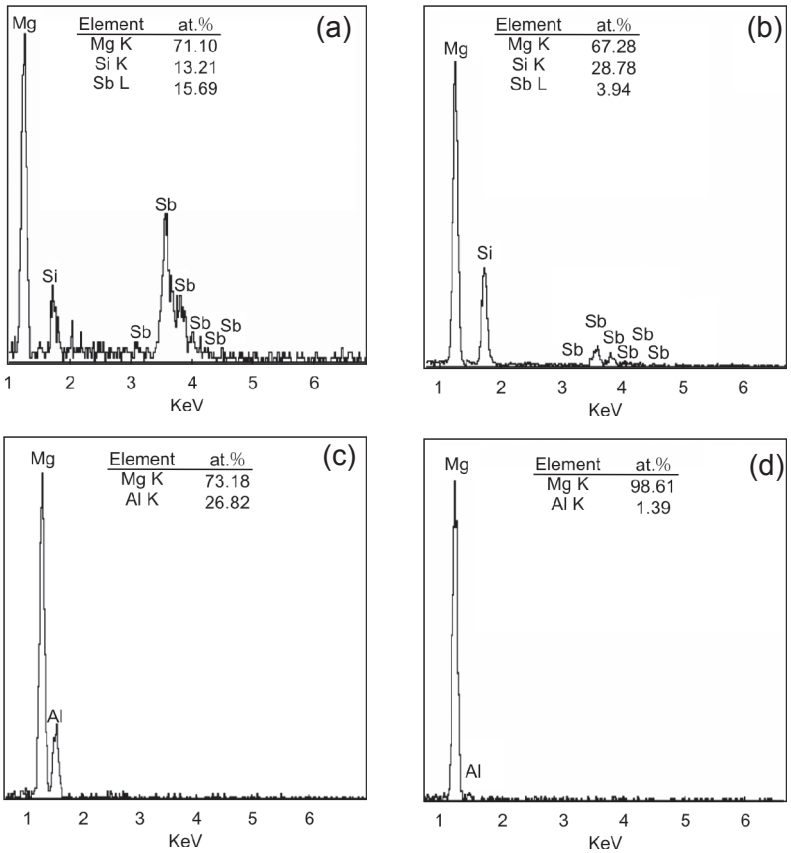

Fig. 3: EDS spectrum of phases in Mg-12Al-0.7Si-0.5Sb: (a) region A; (b) region B; (c) region C and (d) region D in Fig. 2(c)

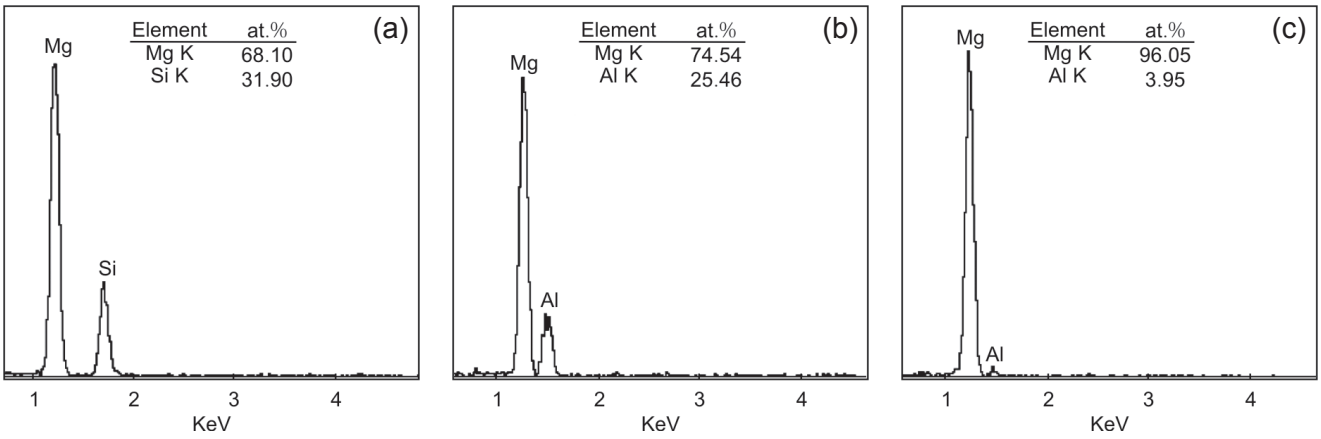

Fig. 4: EDS spectrum of phases in Mg-12Al-0.7Si-0.09Sr: (a) region E; (b) region F; (c) region G marked in Fig. 2(d)

it can be demonstrated that B in Fig. 2c is the Mg_2Si phase, C is the $Mg_{17}Al_{12}$ phase and D is α-Mg. It can be clearly seen that some fine polygonal-shaped Mg_2Si precipitated around the grain boundary while a few were still in the form of Chinese script-shape. However, the massive $Mg_{17}Al_{12}$ phase was refined, and the network structure was broken into some discontinuous island structure and became more dispersive. A clear white phase marked as A was detected in the Mg_2Si phase. The atomic percentages of Mg, Si and Sb elements in the white phase are 71.10%, 13.21% and 15.69%, respectively, and a (Mg, Sb) compound is a possibility. The (Mg, Sb) compound is extremely small, which explains why no new phase was detected by XRD. Previous investigations showed that the modification and/or refinement of Mg_2Si phases in Si-containing micro-alloyed magnesium alloys were mainly related to the formation of nuclei for Mg_2Si precipitates [21]. In the present work, the EDS results confirm that it is the (Mg, Sb) compound in the Mg_2Si intermetallics, which acts as a nucleus [22, 23]. According to the above-mentioned mechanism, the main reason for the modification and refinement of Mg_2Si phases in Mg-12Al-0.7Si-0.5Sb alloy is related to the heterogeneous nucleation.

Figure 2(d) shows the microstructure of the Mg-12Al-0.7Si-0.09Sr alloy. Figure 4 presents the results of EDS analysis of the regions marked in Fig. 2(d). As shown in Fig. 4(a), the atomic percentage of Mg is nearly twice that of Si, indicating that E is the Mg_2Si phase. In addition, F is the $Mg_{17}Al_{12}$ phase and G is α-Mg. It is remarkably observed that, after adding 0.09wt% Sr to Mg-12Al-0.7Si alloy (Fig. 2d), the massive $Mg_{17}Al_{12}$ phase was refined, but some net-shaped $Mg_{17}Al_{12}$ phase still can be detected. The Mg_2Si phases in the Mg-12Al-0.7Si-0.09Sr alloy became extraordinarily fine, and their morphology changed from initial Chinese script-shape to granular and/or irregular polygonal shapes, indicating that adding 0.09wt.% Sr can effectively modify and refine the Chinese script-shaped Mg_2Si phase, which is consistent with the results of Yang Ming-bo et al. [15] and Srinivasan et al. [16]. However, no new phase was found in the alloy according to the above information from the XRD and EDS results (Fig. 1(c) and Fig. 4), indicating that the heterogeneous nucleation mechanism is not suitable for the modification and refinement of Mg_2Si phases in the Mg-12Al-0.7Si-0.09Sr alloy.

It is observed from the DTA results in Fig. 5 that there are three endothermic peaks in all of the three alloys, including Mg transform peak (1), α-Mg+Mg_2Si eutectic peak (2) and $Mg_{17}Al_{12}$ eutectic peak (3). The effect of adding 0.09wt.% Sr on the onset crystallizing temperatures of peak (1) and peak (2) is very obvious, decreasing from 588.76 ºC and 559.35 ºC to 580.17 ºC and 554.31 ºC, respectively. However, after adding 0.5wt.% Sb, the onset crystallizing temperatures decreased slightly to 585.47 ºC and 558.37 ºC. As known, the under-cooling degree is a very vital factor affecting the microstructural refinement during solidification process. According to the classic solidification theory, the relationship between the critical nucleus radius and the under-cooling degree is given as follows [24]:

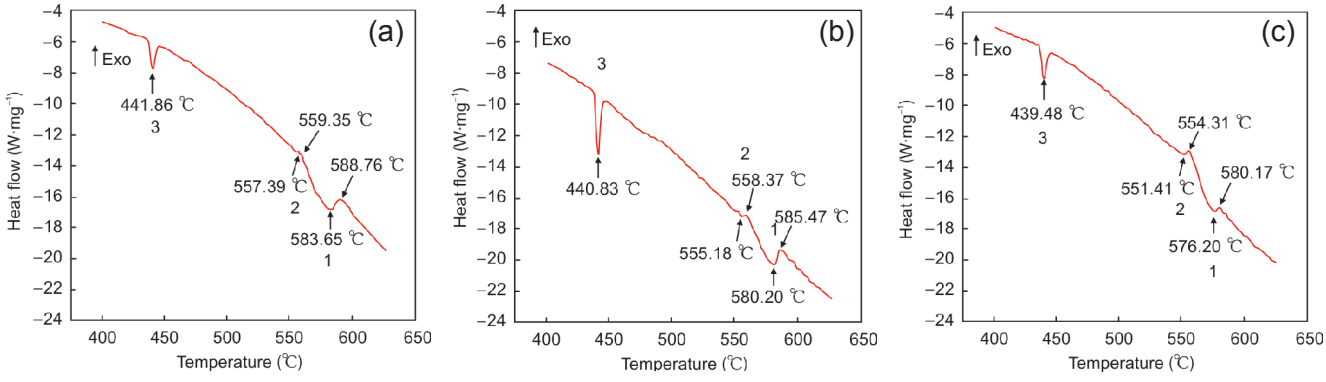

Fig. 5: DTA result of experiment alloys (a) Mg-12Al-0.7Si, (b) Mg-12Al-0.7Si-0.5Sb, (c) Mg-12Al-0.7Si-0.09Sr

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

where r^* is the critical nucleus radius, ΔGr is the variation of volume free energy, σ is the interfacial energy of unit surface area, T_m is the equilibrium crystallizing temperature, L_m is the crystallizing latent heat and ΔT is the under-cooling degree, T_i is the onset crystallizing temperature. According to the equation above, the degree of under-cooling increases with the decrease of Ti, then the critical nucleus radius decreases, the nucleation energy of crystal nucleus reduces and the probability of nucleation increases, which would result in grain and precipitate refinement. Obviously, the under-cooling degree mechanism can explain the modification and refinement of the Mg_2Si phase in the Mg-12Al-0.7Si-0.09Sr alloy. The under-cooling degree of Sr-modified Mg-12Al-0.7Si alloy is higher than that of the Sb-modified Mg-12Al-0.7Si alloy. Hence, the Mg_2Si phases in Sr-modified alloy are effectively modified and refined. Based on the above analysis, the main reason for the modification and refinement of Mg_2Si phases in Mg-12Al-0.7Si-0.09Sr alloy is related to the increase of the under-cooling degree.

In addition, after adding 0.5wt.% Sb to Mg-12Al-0.7Si alloy, the $Mg_{17}Al_{12}$ phase is not only refined, but also broken into some discontinuous island structure. However, there is still some network $Mg_{17}Al_{12}$ phase that can be found in Mg-12Al-0.7Si-0.09Sr alloy. The difference of Sb and Sr in the modification and refinement of $Mg_{17}Al_{12}$ phase in Mg-12Al-0.7Si alloy is possibly related to the following two aspects: Firstly, Sb and Sr are both surface-active elements. The solubility of Sb in the magnesium matrix is lower than that of Sr (0.11wt.%). As the primary α-Mg formed, more redundant Sb solute atoms in the melts enrich the $Mg_{17}Al_{12}$ growing interface than those of Sr, restricting the $Mg_{17}Al_{12}$ growth during the solidification process. Secondly, the Mg_2Si phases in different morphologies at the interface of the liquid-solid phase during solidification can restrict the precipitation of $Mg_{17}Al_{12}$ phase. Some Chinese script-shaped Mg_2Si phase in the Mg-12Al-0.7Si-0.5Sb alloy can restrict the growth of $Mg_{17}Al_{12}$ phase and the formation of network structure. However, since most of Mg_2Si phase has changed to granular and irregular polygonal shapes in Sr-modified alloy, the $Mg_{17}Al_{12}$ phase precipitated along the grain boundary still has a certain growth space. Therefore, some network $Mg_{17}Al_{12}$ phase can be found in Mg-12Al-0.7Si-0.09Sr alloy.

According to the above analysis, it is inferred that the under-cooling degree plays a more important role in the modification and refinement of Chinese script-shaped Mg_2Si phase in Si-containing magnesium alloys than does a heterogeneous nucleus. Accordingly, it is found from Fig. 2(a-d) that as for Mg_2Si phases, the refinement and modification results of adding 0.09wt.% Sr are better than that of adding 0.5wt.% Sb. For the $Mg_{17}Al_{12}$ phase, the results are opposite.

2.2 Mechanical properties

The tensile properties, including room temperature and 200 °C, of the experimental alloys are shown in Fig. 6. It can be seen that the ultimate tensile strengths (UTS) of Mg-12Al-0.7Si alloy under room and high temperature are 121 MPa and 96 MPa, respectively. The UTS of Mg-12Al-0.7Si-0.5Sb and Mg-12Al-0.7Si-0.09Sr are both higher than that of Mg-12Al-0.7Si, indicating that adding small amounts of Sb or Sr can improve the mechanical properties of the Mg-12Al-0.7Si alloy. At room temperature, the UTS of Mg-12Al-0.7Si-0.5Sb alloy is 149 MPa and that of Mg-12Al-0.7Si-0.09Sr alloy is 158 MPa, enhanced by 22.3% and 30.6%, respectively. At 200 °C, the UTS of Sb-modified alloy and Sr-modified alloy are increased by 39.6% to 134 MPa and 60.4% to 154 MPa, respectively. The improvement in mechanical properties of Sb or Sr modified alloys is attributed to the modification and refinement of $Mg_{17}Al_{12}$ and Mg_2Si phases. After adding 0.09wt% Sr to Mg-12Al-0.7Si alloy, though the Chinese script-shaped Mg_2Si phases in the alloys are modified and refined, some network $Mg_{17}Al_{12}$ phase is a disadvantage to the mechanical properties of the alloys at room temperature. Hence, the UTS value of Mg-12Al-0.7Si-0.09Sr alloy is just slightly higher than that of Mg-12Al-0.7Si-0.5Sb alloy, not as obvious as that at high temperature. At 200 °C, the network $Mg_{17}Al_{12}$ phase in Mg-12Al-0.7Si-0.09Sr reduces the diffusion in and around the boundary during tensile testing and hence provides an effective barrier for grain boundary sliding [25]. With the fine granular and irregular polygonal-shaped Mg_2Si, the Mg-12Al-0.7Si-0.09Sr alloy exhibits distinctly higher UTS values than that of Mg-12Al-0.7Si-0.5Sb alloy at high temperature.

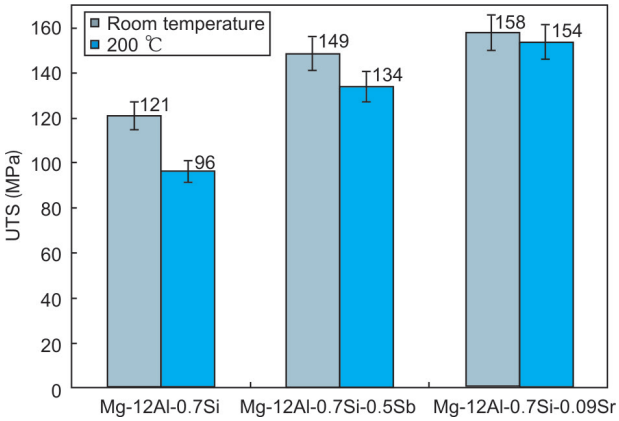

Fig. 6: Tensile properties of experimental alloys

3 Conclusions

(1) By adding 0.5wt.% Sb to the Mg-12Al-0.7Si alloy, the massive $Mg_{17}Al_{12}$ phase is refined and broken into some discontinuous island structure. However, some network $Mg_{17}Al_{12}$ phase can still be detected in the Mg-12Al-0.7Si-0.09Sr alloy. Therefore, Sb performs better in the modification and refinement of $Mg_{17}Al_{12}$ phase than Sr does.

(2) Some fine polygonal-shaped Mg_2Si precipitates are found at the grain boundary in Mg-12Al-0.7Si-0.5Sb alloy while a few are still in the form of Chinese script-shape. However, after adding 0.09wt.% Sr to Mg-12Al-0.7Si alloy, the morphology of Mg_2Si phase changes from the coarse Chinese script shapes to fine granules and irregular polygonal shapes. Hence, the effects of

modification and refinement on Mg_2Si phase are more significant by adding 0.09wt.% Sr than 0.5wt.% Sb.

(3) The room and elevated temperature properties of the Sb or Sr modified Mg-12Al-0.7Si alloy are greatly improved, especially at high temperature. Comparing Sb with Sr, it is detected that the UTS values are approximately similar to each other at room temperature, raised from 121 MPa to 149 MPa and 158 MPa, respectively, but show a large difference at elevated temperature, increased by 39.6% to 134 MPa and 60.4% to 154 MPa , respectively.

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