Article

Thermal Conductivity of As-Cast and Annealed Mg-RE Binary Alloys

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Abstract: Thermal conductivity and phase identification of Mg-RE (Rare-Earth) alloy (Mg-La, Mg-Ce, Mg-Nd, Mg-Sm) in both as-cast and annealed states were investigated. The thermal conductivity was measured by the laser flash method in the composition range of 4 wt.% to 12 wt.% RE and the temperature range of 298 K to 673 K. Results demonstrated that homogenization treatment can increase its thermal conductivity. The thermal conductivity increases with the increasing temperature in two states, which is inconsistent with the pure magnesium, and the thermal conductivity of Mg-RE alloys decreases with the alloy additions. Compared with the atomic radius, valence, and intermetallic compounds, the solid solubility of the above four RE elements in α -Mg plays a vital role in the thermal conductivity of Mg-RE binary alloy. The reduction of thermal conductivity caused by the addition of Nd and Sm, which show observable solid solubility in α -Mg, is significantly greater than the addition of La and Ce with negligible solid solubility in α -Mg.

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1. Introduction

Magnesium alloys are promising materials in the automotive, electronic, and aviation field because of light weight, high specific strength, outstanding damping performance, and environmental protection [1–3]. Thermal conductivity is an important thermophysical properties, and it plays an essential role in the performance of alloy. High thermal conductivity of magnesium alloys provides fast heat conduction. It ensures that the temperature is evenly distributed, thus improving fatigue property and prolonging the service life [4–6]. Thermal conductivity is affected by solid solution atoms, second phases, temperature changes, and heat treatment [7–12]. Pan et al. [13] found that the influence on thermal conductivity of magnesium alloys for the six solute elements was $Zn < Al < Ca < Sn < Mn < Zr$.

Among different magnesium alloy types, Mg-RE (Rare-Earth) alloys have superior properties such as excellent creep properties, high strength, and heat and corrosion resistance [14–16]. In contrast, there are few studies on the thermal conductivity. Rudajewva et al. [4] measured the thermal conductivity of as-cast Mg-(3.2–19.0 wt.%) Sc from 293 K to 573 K, which has a negative correlation with Sc content, while it has a positive correlation with temperature. Zhong et al. [17] experimentally studied the effect on thermal conductivity that four elements added in magnesium in as-cast and as-solutionized states at 298 K. The order is $Ce < Nd < Y < Gd$. Su et al. [18] reported the influence of three typical Ce, Sm, and Y elements on magnesium alloys for both as-cast and as-solutionized at 298 K. Peng et al. [19,20] proved that adding Ce would obviously increase the strength of alloys without reducing the thermal conductivity. From these reports, we can see that these available measurements on the thermal conductivity of Mg-RE alloys are either in the as-cast state or at room temperature.

In order to comprehensively study the effect of various factors on the thermal conductivity of pure magnesium and obtain Mg-RE alloy with outstanding thermal conductivity, Mg-RE (RE = La, Ce, Nd, Sm) alloys were experimentally investigated. According to the solid solubility of the above four elements in α -Mg, they were divided into two groups. Group one, including La and Ce, shows negligible solid solubility, while the other group, including Nd and Sm, shows observable solid solubility. The effect of elements types, elements content, temperature, and homogenization treatment on the thermal conductivity of these four binary systems will be discussed in details.

2. Experimental Procedure

The nominal composition of Mg-RE (RE = La, Ce, Nd, Sm) alloys was listed in Table 1; the samples were prepared with pure magnesium (99.99 wt.%) and master alloys of Mg-12 wt.% La, Mg-25 wt.% Ce, Mg-20 wt.% Nd as well as Mg-35 wt.% Sm. Raw materials were placed in a graphite crucible, and the induction furnace was filled with high purity Ar to protect the raw materials from oxidation during the melting process. They were remelted many times to ensure the homogeneities of the samples, and then four alloy ingots were obtained. Each alloy ingot was cut into two disc-shaped samples with a diameter of 10 mm and a thickness of 3 mm. One piece was directly used for as-cast research, and the other piece was sealed in an evacuated silica capsule under vacuum (1 Pa) to wait for subsequent homogenization heat treatment. Mg-RE alloys were homogenized annealed at 673 K for 30 days, then quenched in water. Chemical analysis was performed with an inductively coupled plasma analyzer (IRIS Advantage 1000), and the detected composition is shown in Table 1. Scanning electron microscopy (SEM) (Zeiss Sigma SEM, Germany) and X-ray diffraction (XRD) (Rigaku D max/2550 VB+X-ray diffractometer) were used to observe the microstructure of the as-cast and annealed alloys and analyze their phases.

Table 1. Compositions of Mg-RE binary alloys.

Nominal Composition of Allys (wt.%)	Actual Composition of RE (wt.%)	Actual Composition of RE (at.%)	Mg
Mg-4La	3.96	0.71	Balance
Mg-6La	5.78	1.04	
Mg-8La	8.31	1.50	
Mg-10La	10.10	1.82	
Mg-4Ce	3.16	0.55	
Mg-6Ce	4.68	0.82	
Mg-10Ce	7.57	1.32	
Mg-12Ce	9.43	1.65	
Mg-4Nd	3.56	0.61	
Mg-6Nd	5.37	0.91	
Mg-8Nd	7.29	1.24	
Mg-10Nd	8.89	1.51	
Mg-4Sm	3.54	0.58	
Mg-6Sm	5.31	0.88	
Mg-8Sm	6.73	1.11	
Mg-10Sm	9.33	1.54	
Mg-12Sm	11.00	1.82	

The laser flash method (NETZSCH LFA 457 laser conductometer, ASTM E1461 Standard, Germany) was used to measure thermal diffusivities (α) of Mg-RE alloy samples from 298 K to 673 K, and repeated more than three times. The density at 298 K of all alloys was obtained by the Archimedes method, and it was obtained by Equation (1) [21] above 298 K:

$$\rho = \rho_0 - 0.156 (T-298) \quad (1)$$

where, ρ_0 (kg/m³) is the density at 298 K, T (K) is the absolute temperature.

The specific heat capacity C_p (J/(g $\times$ K)) can be calculated using the Neumann-Kopp rule [22–24] combined with thermodynamic database [25–29].

The thermal conductivity λ (W/(m $\times$ K)) was obtained by Equation (2) [30]:

$$\lambda = \alpha \times \rho \times C_p \quad (2)$$

The error ranges for measurement of the density and thermal diffusivity are both estimated to be $\pm 3\%$. The reliability of the heat capacity calculated based on the thermodynamic database is estimated to be less than $\pm 5\%$. The total uncertainty for the thermal conductivity is believed to be less than $\pm 7.85\%$.

3. Results

The phase diagrams of the Mg-RE (RE = La, Ce, Nd, Sm) systems calculated by Thermo-Calc software and the reported thermodynamic descriptions [25–29] are shown in Figure 1. The prepared alloys are located in two-phase regions at 673 K. The intermetallic compounds can be obviously observed in both as-cast and annealed alloys. More intermetallic compounds are observed to precipitate with RE addition. The morphology of the intergranular phase strongly influences the overall properties of alloys [31].

Figure 1. Phase diagram of: (a) the Mg-La system; (b) the Mg-Ce system; (c) the Mg-Nd system; (d) the Mg-Sm system.

3.1. Mg-La Alloys

Figure 2a–d show the back-scattered electron (BSE) images of Mg-La alloys. It shows that both the as-cast and annealed alloys consisted of α -Mg phase and the intermetallic compound LaMg_{12} . The LaMg_{12} phase appearing light-grey in Figure 2a almost fully surrounds the grains of α -Mg phase appearing dark-grey, and separates them from each other. Such behavior is intimately connected with the complete and incomplete wetting of grain boundaries by the melt or second phase [3,32]. With La addition, more LaMg_{12} is precipitated. Besides this, a distinct dendritic morphology was observed in as-cast Mg-10La alloys with island-shaped α -Mg separated by LaMg_{12} , and the LaMg_{12} in the annealed Mg-10La alloy was evenly distributed. Therefore, the grain size was decreased. Figure 2e,f present the XRD patterns for phase identification. The results are consistent with the analysis by SEM.

Figure 2. BSE micrographs of (a) the as-cast Mg-4La alloy, (b) the as-cast Mg-10La alloy, (c) the annealed Mg-4La alloy, and (d) the annealed Mg-10La alloy; and XRD diffraction patterns of (e) the as-cast Mg-4La and Mg-10La alloys, and (f) the annealed Mg-4La and Mg-10La alloys.

It is observed from Figure 3 that thermal conductivity in two states decreased obviously after La was added. For as-cast alloy, it decreased from 127.699 W/(m $\times$ K) in Mg-4La alloy to 100.295 W/(m $\times$ K) in Mg-10La alloy at 298 K. For the annealed alloy, it decreased from 133.748 W/(m $\times$ K) to 113.189 W/(m $\times$ K). Besides this, Figure 3a,b illustrate that the thermal conductivity in two states increased with temperature rise, which is contrary to the trend in pure magnesium measured by Hol et al. [33]. The thermal conductivities steadily rose in as-cast Mg-4La alloys from 127.699 W/(m $\times$ K) at 298 K to 135.127 W/(m $\times$ K) at 673 K. As for the annealed alloy, it even reached 138.476 W/(m $\times$ K) at 673 K. Consequently, the thermal conductivity increases at the same composition and temperature after homogenization annealing treatment.

Figure 3. Thermal conductivity of (a) as-cast Mg-La alloys; (b) annealed Mg-La alloys.

3.2. Mg-Ce Alloys

As shown in Figure 4a–d, both α -Mg phase and intermetallic compound CeMg_{12} were observed in the as-cast and annealed states in Mg-(4,12)Ce alloy. With the addition of Ce, the amount of CeMg_{12} increased significantly. For as-cast alloys, the CeMg_{12} phase in Mg-4Ce alloy was continuously distributed in the network of α -Mg matrix, while the α -Mg matrix phase was completely separated by CeMg_{12} in the island shape and irregular distribution in Mg-12Ce alloy. For annealed alloys, CeMg_{12} in the Mg-4Ce alloy became a semi-continuous network, which was distributed in the α -Mg. The CeMg_{12} in the annealed Mg-12Ce alloy was evenly distributed and the grain size was decreased. Figure 4e,f show the XRD patterns, which confirm the observations by SEM.

Figure 4. BSE micrographs of (a) the as-cast Mg-4Ce alloy, (b) the as-cast Mg-12Ce alloy, (c) the annealed Mg-4Ce alloy, and (d) the annealed Mg-12Ce alloy; and XRD diffraction patterns of (e) the as-cast Mg-4Ce and Mg-12Ce alloys, and (f) the annealed Mg-4Ce and Mg-12Ce alloys.

Figure 5a,b demonstrate that after Ce was added, the thermal conductivity in two states reduced dramatically. For as-cast alloy, it reached 118.298 W/(m $\times$ K) at 298 K in Mg-4Ce alloy, then it decreased to 91.986 W/(m $\times$ K) in Mg-12Ce alloy. In annealed state, it dropped from 137.031 W/(m $\times$ K) to 111.876 W/(m $\times$ K). Figure 5a,b also illustrate that the thermal conductivity increased significantly with temperature rise at the same Ce content, which is different to the trend in pure magnesium. The value of thermal conductivity of the as-cast Mg-4Ce alloy reached 118.298 W/(m $\times$ K) at 298 K, then it increased to 129.687 W/(m $\times$ K) at 673 K. As for annealed Mg-4Ce alloy, it increased from 137.031 W/(m $\times$ K) to 143.129 W/(m $\times$ K). It also demonstrates that thermal conductivity increased at the same composition and temperature after homogenization annealing treatment.

3.3. Mg-Nd Alloys

The BSE images are shown in Figure 6, where the metastable phase NdMg_{12} and stable phase $\text{Nd}_5\text{Mg}_{41}$ are observed in Figure 6a,b and Figure 6c,d, respectively. Zhai et al. [34] found that rapid cooling of Mg-24 wt.% Nd leads to the appearance of metastable phase. When the cooling rate is 14 $^{\circ}\text{C}/\text{min}$, the metastable phase NdMg_{12} forms. Stable phase $\text{Nd}_5\text{Mg}_{41}$ forms when the cooling rate decreases to 6 $^{\circ}\text{C}/\text{min}$. In this experiment, metastable phase NdMg_{12} appeared in the as-cast alloys because of the excessive cooling rate. NdMg_{12} was distributed as semi-continuously region in the as-cast Mg-4Nd alloy. With Nd addition, more NdMg_{12} was observed to precipitate as shown in the as-cast

Mg-10Nd alloy. The NdMg_{12} phase showed a network structure with inhomogeneous region. After homogenization treatment, $\text{Nd}_5\text{Mg}_{41}$ was distributed semi-continuously and regularly among the grains of α -Mg in Mg-4Nd alloy. $\text{Nd}_5\text{Mg}_{41}$ appeared as a network and the grains gradually became coarser in the annealed Mg-10Nd alloy. Figure 6e,f show the XRD patterns, which confirms the phase constitution by SEM.

Figure 5. Thermal conductivity of (a) as-cast Mg-Ce alloys; (b) annealed Mg-Ce alloys.

Figure 6. BSE micrographs of (a) the as-cast Mg-4Nd alloy, (b) the as-cast Mg-10Nd alloy, (c) the annealed Mg-4Nd alloy, and (d) the annealed Mg-10Nd alloy; and XRD diffraction patterns of (e) the as-cast Mg-4Nd and Mg-10Nd alloys, and (f) the annealed Mg-4Nd and Mg-10Nd alloys.

Figure 7a,b illustrate that the thermal conductivity in the two states decreased almost linearly after Nd addition. The value of thermal conductivity reached 98.951 W/(m × K) in as-cast state for Mg-4Nd alloy at 298 K, then it decreased to 66.291 W/(m × K) in Mg-10Nd alloy. In annealed state, it dropped from 117.084 W/(m × K) to 92.480 W/(m × K). Figure 7 also shows that the thermal conductivity increased significantly with temperature rise at the same Nd content, which is inconsistent with the pure magnesium. The value of thermal conductivity of as-cast Mg-4Nd alloy reached 98.951 W/(m × K) at 298 K, then it increased obviously to 135.981 W/(m × K) at 673K. Meanwhile, in annealed Mg-

4Nd alloy, it increased from 117.084 W/(m × K) to 133.436 W/(m × K). So, the thermal conductivity increased at the same composition and temperature after homogenization annealing treatment.

Figure 7. Thermal conductivity of (a) as-cast Mg-Nd alloys; (b) annealed Mg-Nd alloys.

3.4. Mg-Sm Alloys

Figure 8a–d show the BSE images of Mg-Sm alloys, where the α -Mg phase and intermetallic compound $\text{Sm}_5\text{Mg}_{41}$ are observed. With Sm addition, more $\text{Sm}_5\text{Mg}_{41}$ is observed to precipitate in the matrix phase. For as-cast alloys, $\text{Sm}_5\text{Mg}_{41}$ in a parallel plank shape uniformly separated the α -Mg in the Mg-4Sm alloy, while the morphology of the $\text{Sm}_5\text{Mg}_{41}$ in the Mg-12Sm alloy showed the microstructure of fish-bone shape. After homogenization treatment, a small amount of the $\text{Sm}_5\text{Mg}_{41}$ was discontinuously distributed between the α -Mg phase. Meanwhile, the fish-bone shape structure observed from the BSE images of the as-cast alloy were transformed into a coarse network structure in annealed Mg-12Sm alloy. XRD patterns of Mg-Sm alloys are presented in Figure 8e,f; the second phase of alloys in two states is $\text{Sm}_5\text{Mg}_{41}$, which is consistent with the result by SEM.

The addition of Sm linearly decreased the thermal conductivity of Mg-Sm alloys as illustrated in Figure 9. For as-cast alloys, thermal conductivity reached 83.398 W/(m × K) in Mg-4Sm alloy at 298 K, then it decreased to 58.404 W/(m × K) in Mg-12Sm alloy. For annealed alloys, it dropped from 99.875 W/(m × K) to 81.075 W/(m × K). Additionally, Figure 9 shows that the thermal conductivity increased significantly with temperature rise at the same Sm content, which is inconsistent with the pure magnesium. The value of thermal conductivity increased from 83.398 W/(m × K) at 298 K to 116.464 W/(m × K) at 673 K in as-cast state for Mg-4Sm alloy. For annealed Mg-4Sm alloy, it increased from 99.875 W/(m × K) to 127.367 W/(m × K). It also demonstrates that thermal conductivity increased at the same composition and temperature after the homogenization annealing treatment.

Figure 8. BSE micrographs of (a) the as-cast Mg-4Sm alloy, (b) the as-cast Mg-12Sm alloy, (c) the annealed Mg-4Sm alloy, and (d) the annealed Mg-12Sm alloy; and XRD diffraction patterns of (e) the as-cast Mg-4Sm and Mg-12Sm alloys, and (f) the annealed Mg-4Sm and Mg-12Sm alloys.

Figure 9. Thermal conductivity of (a) as-cast Mg-Sm alloys; (b) annealed Mg-Sm alloys.

4. Discussion

4.1. Temperature Dependence

Ho et al. [33] measured the thermal conductivity of pure magnesium from 1 to 700 K, and extrapolated it at the temperature range between 773.2 and 923.2 K. It was found that thermal conductivity increased sharply from 1 to 9 K, and reached the maximum value of 5700 W/(m × K) at 9 K, then sharply dropped to 169 W/(m × K) at 100 K. When the temperature was higher than 100 K, the thermal conductivity of pure magnesium reduced slowly with temperature rise.

In this study, although the RE atoms in α-Mg acted as impurities after RE elements were added, there was no temperature dependence of the electrical resistance caused by impurities [35]. Besides this, the number and energy of phonons rose greatly with

the increasing temperature, and lattice vibration became stronger, which hindered its free movement, so the thermal conductivity caused by the phonons decreased. The heat of many metals at ambient temperature is conducted by electrons [36]. The thermal conductivity caused by electrons plays a dominant role in the alloy. The Wiedemann-Franz law indicates that the thermal conductivity caused by electrons increases with the increasing temperature. Consequently, the thermal conductivity of Mg-RE alloys increases with increasing temperature. Figure 3b, Figure 5b, Figure 7b, and Figure 9b compare the thermal conductivity of pure magnesium and alloy. In pure magnesium, the thermal conductivity decreased with increasing temperature. Therefore, the lines of the relationship between thermal conductivity and temperature shown in the above figures are crossed.

4.2. Effect of Homogenization Treatment

The electron conduction is limited by the impurities scattering process, and the scattering of impurities is affected by its own thermal resistance and lattice defects [36]. On the one hand, homogenization treatment can reduce the lattice defects produced during the casting process. It also reduces the scattering of impurities and thermal resistance. Then, it increases the conduction of electrons, whereby the thermal conductivity of the Mg-RE alloys rises. On the other hand, for the Mg-Nd system with the metastable phase Nd_{12}Mg , the metastable precipitation phase transformed into the stable phase $\text{Nd}_5\text{Mg}_{41}$ after homogenization treatment. The solute atoms precipitate and reduce the distortion of lattice and intensify the free movement of electrons and phonons, which increase the thermal conductivity. In this experiment, homogenization annealing treatment increased the thermal conductivity.

4.3. Effect of RE Elements

Figure 10a,b show the influence of the thermal conductivity of the RE elements addition in pure magnesium. With RE addition, RE atoms were dissolved in α -Mg matrix phase. RE atoms that are dissolved in α -Mg destroy periodic arrangement of magnesium lattice, which causes lattice distortion. Consequently, the thermal conductivity decreases. The BSE images indicate that the content of intermetallic compounds increases significantly with the increase of RE content. These two factors cause the decrease of thermal conductivity. In addition, the slope of the lines reflects the decrease levels of thermal conductivity. After adding Nd and Sm, which show observable solid solubility in α -Mg, the levels of the reduction of thermal conductivity are significantly greater than adding La and Ce with negligible solid solubility in α -Mg.

Figure 10. Effect of alloying elements addition on thermal conductivity of (a) as-cast magnesium alloys at 298 K; (b) annealed magnesium alloys at 673 K.

The valence also affects the thermal conductivity. Table 2 shows that the valence number of the four RE atoms is 3, and the valence number of magnesium atoms is 2. The

lattice expands after the RE atom is dissolved in α -Mg. This phenomenon is affected by the following two factors: the electronic structure of magnesium and the shape of the Brillouin zone [14,37]. That is to say, the effects of valence of four elements are similar.

Table 2. Atomic number, valence, atomic radius and maximum solid solubility in the Mg solid solution of the four solute atoms.

Element	Atomic Number	Valence	Atomic Radius/nm	Maximum Solid Solubility wt. %/at. % in Mg
Mg	12	+2	0.1602	—
La	57	+3	0.1877	$5.55 \times 10^{-7} \approx 0$
Ce	58	+3	0.1824	0.52/0.09
Nd	60	+3	0.1821	3.63/0.63
Sm	62	+3	0.1804	5.83/0.99

Thermal conductivity is affected by atomic radius. The RE atoms were dissolved in α -Mg to form a substitutional solid solution, leading to the lattice distortion. The larger the difference of atoms between the RE atoms and the magnesium atoms, the more serious the lattice distortion [14]. That reduces the thermal conductivity. It can be seen from Table 2 that the effect of atomic radius is: La > Ce > Nd > Sm.

In this experiment, the solid solution reached saturation after RE elements were added. Eivani et al. [38] show that, compared with the second phase, the solid solubility has more negative effect on thermal conductivity. The solid solubility of solute atoms in α -Mg is affected by the radius difference between RE atoms and magnesium atoms. The radius difference between Sm atoms and magnesium atoms is the smallest, and the solid solubility is the largest. Table 2 shows the maximum solid solubility of four RE atoms. The influence of solid solubility is: Sm > Nd > Ce > La.

Adding variety of RE elements into magnesium alloy, its thermal conductivity is affected by factors such as atomic radius, solid solubility, and valence. This study has found that the solid solubility among the above factors plays a dominant role in the thermal conductivity of Mg-RE binary alloy.

5. Conclusions

- (1) Thermal conductivity of the studied Mg-RE alloys decreases with RE addition, and increases with increasing temperature.
- (2) The annealed Mg-RE alloys featured higher thermal conductivity than the as-cast alloys at the same composition and temperature. Homogenization treatment can increase the thermal conductivity.
- (3) The solid solubility of Sm in α -Mg is the largest, and Mg-Sm alloy has the lowest thermal conductivity under the same conditions. The reduction of thermal conductivity caused by the addition of Nd and Sm, which show observable solid solubility in α -Mg, is significantly greater than the addition of La and Ce with negligible solid solubility in α -Mg.

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