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High temperature tensile properties of as-cast Mg–Al–Ca alloys

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ABSTRACT

In order to choose the appropriate process parameters for further thermo-mechanical processes, high temperature tensile tests of as-cast Mg–Al–Ca alloys containing 1, 4 and 7% of total amount of Al and Ca (denoted as AX1, AX4 and AX7, respectively), where Al:Ca = 29:26, were examined. During the tensile test, the Mg_2Ca and $(Mg, Al)_2Ca$ compounds contribute to not only grain refining but also become crack initiation sites and lower the ductility, thus the AX4 alloy exhibits the best deformability. The area reduction above 50% is obtained at $\dot{\varepsilon} = 1.4 \times 10^{-2}\ s^{-1}$ and $T = 623$ K in AX series alloys and at which the dynamic recrystallization has also occurred. Therefore, extrusion deformation was performed at $\dot{\varepsilon} = 4.2 \times 10^{-2}\ s^{-1}$ and $T = 623$ K. The grain size of as-extruded specimen decreases with increasing total amount of Al and Ca contents and the average grain size of AX4 alloy specimen is refined to 1.4 µm.

1. Introduction

Magnesium alloys are very attractive in applications for the automotive, railway and aerospace industries from the ecological point of view because of the lightness of magnesium alloys. One of the technical challenges for the widespread use of the magnesium alloys in the structural applications is on the improvement of creep strength at elevated temperatures. Recently, it has been reported that addition of Ca to magnesium can enhance the high temperature strength and creep resistance [1–7] and can retard fire burning during the melting of the alloy [8]. The ignition proof property of Ca suggests that special safety precaution to avoid explosion and fire is not needed for the melting and casting of Mg–Ca alloys. Furthermore, calcium is the cheap element compared with yttrium and rare earth metals, which are conventionally used to improve the heat resistance. Because of these advantages, Mg alloys containing Ca are expected to be used as several structural components [9,10]. Also, Mg–Al–Ca system alloys are often considered because of their good castability [11].

Up to date, limited data are available for the processing of the ingot of Mg–Al–Ca system alloys by extrusion or rolling. Bai et al. investigated the effect of Al contents on microstructures, tensile and creep properties of Mg–(4–7 wt.%)Al–1 wt.%Sr–1 wt.%Ca alloys and found that both strength and ductility increase with an increase in Al content at ambient and elevated temperatures of 175 °C and 200 °C [12]. Han et al. indicated that the hardness of the as-cast Mg–5.0 wt.%Al–(0.5–2.0 wt.%)Ca alloys increase with increasing Ca content [13]. Watanabe et al. reported that hot extrusion is effective for refining the microstructure of Mg–6 mass%Al–2 mass%Ca alloy [9], respectively. However, effect of the total amount of Al and Ca on microstructure changes and the high temperature deformation properties of Mg–Al–Ca alloys has not been reported in detail.

Therefore, in the present study, three kinds of Mg–Al–Ca system alloys have been prepared and the effect of total amount of Al and Ca contents on microstructures of the as-cast and as-extruded specimens and high temperature tensile deformation properties have been investigated.

2. Experimental methods

The materials used in the present study were three kinds of Mg–Al–Ca alloys containing 1, 4 and 7% of total amount of Al and Ca (denoted as AX1, AX4 and AX7, respectively), where Al:Ca ratio was constant at 29:26 in order to obtain intermetallic compounds of C14 (Mg_2Ca) and C36 $(Mg, Al)_2Ca$ [14,15]. The chemical compositions are listed in Table 1. Pure Mg, Al, Ca and Al–10.0 wt.%Mn master alloy stocks were used to achieve the target compositions. Melting of the alloys was conducted in a mild steel crucible under mixed gas atmosphere of SF_6 and CO_2. After the alloying elements were completely dissolved, the melt was held at 983 K for 10 min then poured into a billet mold with a cavity of 50 mm diameter and 255 mm height. The crystallized compounds observed in as-cast specimens were then analyzed by TEM. TEM discs 0.12 mm in thickness and 3 mm in diameter were cut from near the center of the die-cast specimen and mechanically polished, followed by low angle ion milling. A JEOL 2010 microscope was used in this study under an accelerating voltage of 200 kV.

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Table 1
The chemical composition of alloys used in the present study (wt.%).

	Al	Ca	Mn	Si	Cu	Fe	Mg
AX1	0.50	0.48	0.02	<0.01	<0.001	<0.001	Bal.
AX4	2.00	2.05	0.03	<0.01	<0.001	<0.001	Bal.
AX7	3.68	3.99	0.02	<0.01	<0.001	<0.001	Bal.

High temperature tensile specimens of cylindrical geometry with a diameter of 4 mm and a gauge length of 20 mm were cut from the billets, and tensile tests were performed using a SHIMADZU AG-I 50 kN Electronic Universal Testing Machine at the temperatures of 573 K, 623 K and 673 K under strain rates of $2 \times 10^{-3}\,s^{-1}$, $1.4 \times 10^{-2}\,s^{-1}$ and $1.0 \times 10^{-1}\,s^{-1}$. Then some of the cast billets were extruded at a temperature at 623 K, under a ram speed of $0.1\,mm\,s^{-1}$ and an extrusion ratio of 20, which were chosen based on the high temperature tensile test of the as-cast samples. During all the testing, the specimens were kept at the given temperature within $\pm 2\,^{\circ}C$. The results of tensile tests were based on the average of three or four specimens on each test condition. The specimens for microstructure examination were sectioned and ground using emery papers up to #4000 followed by final polishing on a Struers Rotopol-15 automatic polishing machine using polishing suspension OP-S (0.04 μm sized SiO_2 particles) and then etched using a solution of alcohol (33 ml), picric acid (2 g), acetic acid (5 ml) and water (5 ml). Microstructures were observed by an Olympus BX60M optical microscope and a JEOL FESEM JSM-7000F scanning electron microscopy. The grain sizes were measured using the software of Image-Pro Plus 5.0 with the grain number more than 1000.

3. Results and discussion

3.1. Microstructures of as-cast specimens

The microstructures of the three as-cast samples consist of the α-Mg matrix and intermetallic compounds along grain boundaries. The marked grain refining effect of total amount of Al and Ca contents on microstructures is clearly evident, as shown in Fig. 1. Coverage ratios of grain boundaries with the compounds increase with increasing total amount of Al and Ca contents and all the grain boundaries are covered with the compounds in as-cast AX7 sample. SEM observations reveal that the grain boundary compounds in as-cast samples show two morphologies: the fine lamellar compounds with bright contrast and coarse irregular-shaped compounds with intermediate contrast, as shown in Fig. 2(a), which are identified as Mg_2Ca phase with C14 structure and $(Mg, Al)_2Ca$ phase with C36 structure, respectively, from the selected area diffraction patterns (SADPs) as shown in Fig. 2(b) and (c). Similar results are also reported by Suzuki et al. [14,15].

3.2. Tensile properties at elevated temperature

The variation in elongation-to-failure and area reduction of the necking parts are shown in Fig. 3 as a function of strain rate for the as-cast AX1, AX4 and AX7 alloys tested at different temperature. It is evident that the tensile elongation and area reduction depend on the strain rate and temperature in all the alloys. The elongation and area reduction increase with decreasing strain rate and with increasing testing temperature. Relatively high elongation greater than 100% is obtained at 673 K in as-cast AX1 and as-cast AX4 samples when tensile tested at a low strain rate of $2 \times 10^{-3}\,s^{-1}$. However, the elongation and area reduction seem to have little relationship with increasing total amount of Al and Ca contents. When tested at the same conditions, the AX4 alloy, which contains 4% of total amount of Al and Ca, exhibits the biggest area reduction. For example, when tensile test was carried out at the low temperature of 573 K and strain rate of $1.4 \times 10^{-2}\,s^{-1}$, only the AX4 alloy exhibits the area reduction above 50%. The microstructures near the fracture tips reveal that the cracks easily occur inside the compounds during the tensile tests, as shown in Fig. 4(b). Therefore, the AX7 alloy containing the highest amount of Al and Ca exhibits the worst tensile elongation and area reduction. SEM observations demonstrate the detailed microstructure of cracks as shown in Fig. 4(c). The cracks mainly occur in the coarse $(Mg, Al)_2Ca$ compound, suggesting that coarse $(Mg, Al)_2Ca$ compound is more brittle and easily broken than the fine Mg_2Ca compound. In the case of AX4 alloy, the recrystallization partly occurs at the limited region adjacent to the grain boundaries as shown in Fig. 4(a).

The occurrence of dynamic recrystallization as a function of strain rate and temperature for the as-cast alloy specimens during tensile tests are shown in Fig. 5. It is found that the dynamic recrystallization occurs even at higher strain rate and lower temperature in the AX4 alloy due to the bigger elongation and area reduction of the necking parts obtained during tensile test as compared with AX1 and AX7 alloy specimens. After tensile test at 623 K and $1.4 \times 10^{-2}\,s^{-1}$, the dynamic recrystallization occurs in all the alloys, which is considerable importance for microstructure refinement and is essential for attaining good deformability and superior mechanical properties in Mg alloy [9–13]; simultaneously, under this deformation condition, all the as-cast alloys exhibit the area reduction above 50%, as shown in Fig. 3(e), which is a experiential value and is usually used to determine the hot forging and extrusion deformation condition for steel in factory [16], and above this value, the deformability of materials can satisfy the demands of the factory for further thermo-mechanical processes [16]. Therefore, these test conditions are considered to be the appropriate parameters for the further thermo-mechanical processes of as-cast AX alloys. As shown in Fig. 6(a), SEM observations reveal that, after tensile test, the intermetallic compounds crystallizing along grain boundaries in as-cast state have been crushed into a large number of small blocks and distribute at the grain boundaries newly-formed by dynamic recrystallization. Some nano-scale rod-like compounds

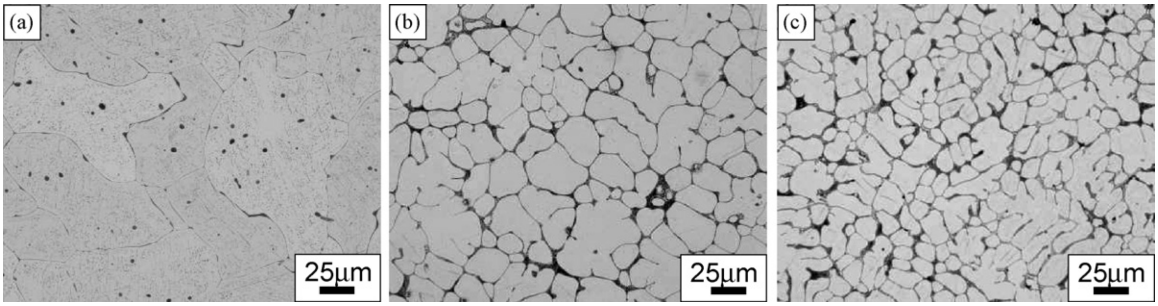

Fig. 1. The typical optical micrographs of as-cast (a) AX1, (b) AX4 and (c) AX7 alloys.