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ARTICLE

Formation of Al_2Ca Phase in as-Extruded X20 Magnesium Alloy by Solution Treatment

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Abstract: The as-extruded X20 magnesium alloys containing 2.0wt% Ca were solution-treated at 410 ºC for 20 h. The microstructures before and after solution treatment were observed by optical microscope (OM), scanning electron microscope (SEM) together with an energy dispersive spectroscope (EDS), and transmission electron microscope (TEM). The phase compositions before and after solution treatment were confirmed by X-ray diffraction (XRD). The results show that the amount of β-$Mg_{17}Al_{12}$ phases decreases remarkably after solution treatment, and new Al_2Ca phases are formed. The formation of Al_2Ca phase might be due to high concentration of Ca atoms in residual β-$Mg_{17}Al_{12}$ phases, which leads the place of Mg atoms to be substituted by Ca. On the other hand, Al_2Ca phase could be also described as a completing product between Al-Ca system and Mg-Ca system intermetallics due to its high structural stability.

Key words: as-extruded magnesium alloys; microstructure; Al_2Ca compound; phase identification

Because of low density, excellent cast ability and good machinability, Mg-Al based alloys have become promising materials for automotive, railway and aerospace applications[1-4]. However, the utilization of magnesium alloys at elevated temperature has been postponed due to the creep deformation, caused by the instability of β-$Mg_{17}Al_{12}$ phase[5-9].

In order to solve the creep deformation, the improvement the tensile strength of magnesium alloys at both ambient and elevated temperature has put faith in the addition of alloy elements[10-15]. Element Ca added into Mg-Al alloys has been found to be more promising. The effect of Ca addition on the high-temperature properties of Mg-Al alloys can be explained to both the stability improvement of β-$Mg_{17}Al_{12}$ phase[7] and the formation of new intermetallic with more stability[16-19].

As a novel kind of Mg-Al-Zn system alloy, more research is needed to determine the effect of Ca addition on the microstructure and the mechanical properties. Both the mechanical properties and the oxidation behavior of such a magnesium alloy containing 2.0wt% Ca at the elevated temperature have been investigated in our previous work [20]. In this paper we investigate in detail the effect of solution treatment on the microstructure of as-extruded X20 magnesium alloys and especially focus on the formation of Al_2Ca phases.

1 Experiment

AZ113 (Mg-11Al-3.0Zn-0.4Mn) bulk matrix alloy was prepared by alloying using Mg bulk (99.9% purity), Al bulk (99.7% purity), (Zn bulk 99.9% purity), and Al-10%Mn master bulk (all in mass percent unless otherwise specified). Subsequently, X20 alloy containing 2%Ca was prepared by the addition of Mg-30%Ca master alloys into AZ113 melts, and then extruded at an extrusion rate of 4~15 mm/s and extrusion ratio of 25:1 at 350 ºC. Finally, as-extruded X20 alloys were solution-treated at 410 ºC for 20 h and cooled in air. In the course of solution treatment, all the samples

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were embedded into graphite powders to prevent from oxidizing.

The metallographic specimens were taken along the cross-section of extrusion direction, then polished and etched with 2 g tartaric acid + 100 mL H_2O solution. The microstructures of as-extruded X20 samples before and after solution treatment were observed and characterized by optical microscope (OM), scanning electron microscopy (SEM, JEOL JSM-6700F) together with energy dispersive X-ray spectroscopy (EDS, Oxford INCA) analysis, and transmission electron microscope (TEM, JEOL JEM-3010). The phase composition of X20 samples before and after solution treatment were identified using X-ray diffraction (XRD, Shimadzu Limited XRD-7000), where the scanning speed was 10º/min, the work voltage 40 kV, the current 40 mA, and Cu used as target.

2 Results and Discussion

2.1 Microstructure and phase composition

Fig.1 presents the microstructure and the corresponding XRD patterns of as-extruded X20 samples before and after solution treatment. It can be found that the microstructures before and after solution treatment comprise white phase α-Mg and gray phase β-$Mg_{17}Al_{12}$. XRD results reveal that no characteristic diffraction peaks of new phases are found after solution treatment. The observation shows that before solution treatment, α-Mg grains are surrounded by fine β-$Mg_{17}Al_{12}$ phases (Fig.1a). However, after the solution treatment, the amount of β-$Mg_{17}Al_{12}$ phases decreases remarkably (Fig.1b). It is considered that solution treatment at the temperature 410 ºC for 20 h leads a part of β-$Mg_{17}Al_{12}$ phases to be dissolved. As a result, the Ca content of α-Mg phases is supersaturated because most of Ca atoms are distributed in the β-$Mg_{17}Al_{12}$ phases before solution treatment[20]. Therefore, it is an interesting question how the redundant Ca atoms exist in X20 alloy after solution treatment.

2.2 Identification of Al_2Ca phase

The further observation was carried out by a scanning electron microscope together with energy dispersive X-ray spectroscopy, and the SEM image is presented in Fig.2. It is clear that there are two kinds of granular phases found in the magnesium matrix. The gray phases can be easily identified as $Mg_{17}Al_{12}$, while the white one should be corresponding to a new phase. Unluckily, no distinct peak, as shown in Fig.1, is corresponding to such a new phase. In addition, it is very interesting that most of unknown white phases can be observed to be formed by attaching to β-$Mg_{17}Al_{12}$ phases. Based on the above, it is thought that solution treatment leads unknown white phases to be formed.

Fig.3 shows the EDS result of unknown white phase. The white phase appears to be a stoichiometry of $CaAl_{2.2}Mg_{0.52}$.

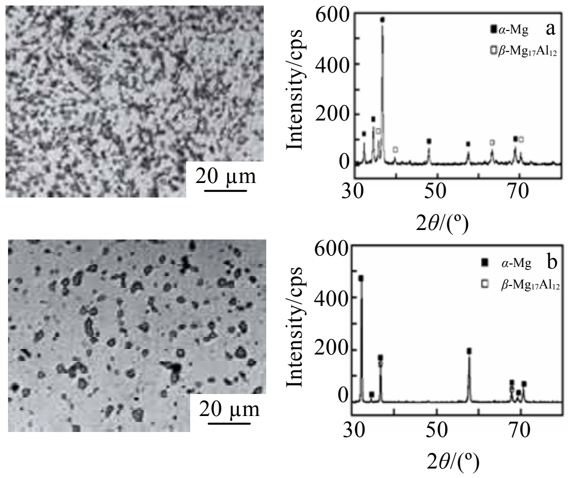

Fig.1 Microstructures and XRD patterns of as-extruded X20 alloys before (a) and after (b) solution treatment at 410 ºC for 20 h

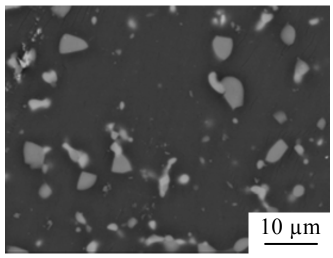

Fig.2 SEM image of as-extruded X20 magnesium alloy after solution treatment at 410 ºC for 20 h

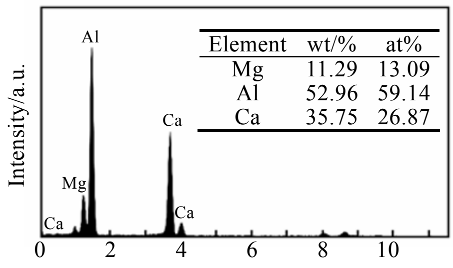

Fig.3 EDS analysis of white phase in as-extruded X20 magnesium alloy after solution treatment

In the past years, $CaAl_{2\text{-}x}Mg_x$ ternary phases were found in the Mg-Al-Ca ternary system[21,22]. $Al_2(Ca,Mg)$ phases with a range from 17.5at% to 22.5at% Mg were also reported by Y. Zhong et al[23]. It is suggested that the existence of $CaAl_{2\text{-}x}Mg_x$ in magnesium alloy seems to be established, but other ternary phases still need to be confirmed[24]. Obviously, the composition of such a white new phase shown in Fig.3 is different from the above results reported. Therefore, the unknown white phase shown in Fig.2 can't be identified as a new ternary phase.