



# Thermal stability and crystal structure of high-temperature compound $\text{Al}_{13}\text{CeMg}_6$

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## ABSTRACT

In order to clarify the controversy in the literature reports, we studied the thermal stability of the ternary compound  $\text{Al}_{13}\text{CeMg}_6$  by integrating X-ray powder diffraction, electron probe microanalyzer and differential scanning calorimetry techniques. It was found that the compound  $\text{Al}_{13}\text{CeMg}_6$  decomposes at a certain temperature between 320 and 400 °C with a eutectoid reaction:  $\text{Al}_{13}\text{CeMg}_6 \rightarrow \text{Al}_{11}\text{Ce}_3 + \text{Al}_3\text{Mg}_2$ , and melts at 492 °C via a peritectic reaction:  $\text{Liquid} + \text{Al}_{11}\text{Ce}_3 \rightarrow \text{Al}_{13}\text{CeMg}_6$ . Moreover, the crystal structure of  $\text{Al}_{13}\text{CeMg}_6$  compound was successfully determined by X-ray powder diffraction using the Rietveld method. The compound crystallizes in the hexagonal, space group  $P63/mmc$  (No. 194) with the structure type of  $\text{MgNi}_2$  and lattice parameters of  $a = 0.5525(9)$  and  $b = 1.7866(2)$  nm.

## 1. Introduction

Aluminum alloys with Mg as the major alloying element are one of the most important Al-based wrought alloys because of their high strength-to-weight ratio, excellent weldability, corrosion resistance and mechanical properties [1,2]. The good properties of the Al-Mg alloys are essentially attributed to the final microstructures forming during the preparation process. Moreover, the Al-Mg alloys with addition of rare-earth (RE) elements, like Sc, Sr, and Ce [3–5], may exhibit good creep resistance and strength at elevated temperature due to the precipitates forming as intermetallic phases with REs.

In order for a better understanding of the precipitates behaviors and phase diagrams of the Al-Mg-REs systems, the thermal stabilities and crystal structures of different related compounds are indispensable. Taking the ternary Al-Mg-Ce system for example, four ternary phases (Al,Mg)Ce [6], (Al,Mg)<sub>2</sub>Ce [6,7], (Al,Mg)<sub>12</sub>Ce [7,8] and  $\text{Al}_{13}\text{CeMg}_6$  [6–11] have been reported up to now. However, there exists the controversy on the thermal stability of the stoichiometric compound  $\text{Al}_{13}\text{CeMg}_6$  in the literature reports. According to Odinaev et al. [6], the ternary compound  $\text{Al}_{13}\text{CeMg}_6$  melts congruently at 635 °C. The more recent report by Gröbner et al. [7] indicated that this compound melts incongruently around 400 °C. Moreover, the temperature range for thermal stability of  $\text{Al}_{13}\text{CeMg}_6$  is still ambiguous. Additionally, in the reports by Refs. [6,7], the crystal structure of  $\text{Al}_{13}\text{CeMg}_6$  was reported to be structure type of  $\text{MgZn}_2$ , but it has not been refined up to now. That is because it is difficult to prepare a single stoichiometric compound.

Consequently, the stoichiometric compound  $\text{Al}_{13}\text{CeMg}_6$  is chosen as the target in the present work. A combination of different experimental techniques, including X-ray diffraction (XRD), electron probe microanalyzer (EPMA) and differential scanning calorimetry (DSC), is employed to study the thermal stability of the compound  $\text{Al}_{13}\text{CeMg}_6$ . While the XRD data together with the Rietveld method are utilized to determine its crystal structure.

## 2. Experimental procedure

High-purity elements, i.e., Al (purity: 99.99 wt.%), Mg (purity: 99.99 wt.%) and Ce (purity: 99.9 wt.%) purchased from Alfa Aesar (China) Chemicals Co., Ltd., were used to minimize any potential effect from possible contamination by trace elements. A sample with nominal composition of  $\text{Al}_{13}\text{CeMg}_6$  (i.e.,  $\text{Al}_{65}\text{Ce}_5\text{Mg}_{30}$ , in at.%) was first placed in a graphite crucible, and then melted in an inductive furnace under an argon gas atmosphere. Since Mg is prone to evaporate during melting, an excess of 5 wt.% Mg was thus used. To ensure its homogeneity, the sample was turned and re-melted for three times. The chemical composition of the sample was measured by inductively coupled plasma (ICP) and further confirmed by chemical analysis (CA). The results show that the chemical compositions of the as-cast sample shift to  $\text{Al}_{66.24}\text{Ce}_{4.36}\text{Mg}_{29.40}$  (in at.%). After that, the ingot was divided into two parts. Both of them were enclosed in  $\text{Al}_2\text{O}_3$  crucible, sealed in the stainless steel tube under high pure argon atmosphere, and then annealed in the annealing furnaces at 400 and 320 °C for 40 days, respectively. Finally, all the samples were quenched into liquid nitrogen

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to retain their high-temperature microstructure. Further ICP and CA measurements indicate that the compositions of the alloys annealed at 400 and 320 °C shift to  $\text{Al}_{65.68}\text{Ce}_{3.43}\text{Mg}_{30.89}$  and  $\text{Al}_{64.75}\text{Ce}_{3.30}\text{Mg}_{31.95}$  (in at.%), respectively.

To determine the phase transformation temperatures, DSC (DSC404C, Netzsch, Germany) measurement was performed on the sample annealed at 320 °C between room temperature to 700 °C with the heating rate of 5 K/min under an argon atmosphere. A Pt–Pt/Rh thermocouple was used. The equipment was calibrated by 7 standard pure metals, i.e. In, Sn, Bi, Zn, Al, Ag, Au, and the accuracy for temperature measurement was estimated to be  $\pm 1.5$  °C. All the two annealed samples were then subject to microstructure observation. Each specimen was mechanically ground and further polished on automatic polishing equipment using an oxide polishing suspension (OP-S) at a rotation speed of 250 rpm. After that, the polished samples were examined and analyzed by back-scattered electron (BSE) mode equipped within an EPMA (JXA-8530, JEOL, Japan).

The annealed samples were ground into powder not coarser than 40  $\mu\text{m}$ . The XRD data of samples were collected on a Bruker D8 Advance (D8AA25X) powder diffractometer using Cu K $\alpha$  radiation ( $\lambda = 1.5406$  Å). The diffractometer was operated at 40 kV and 40 mA, the  $2\theta$  scan ranges from 10 to 90° with a step size of 0.02 and a counting time of 3 s per step. The step-scan XRD patterns of the samples were analyzed and indexed on the Material Data Software Jade 6.0 [12]. The quantitative analysis and structural refinement were performed by Topas V5.0 program [13].

### 3. Results and discussion

#### 3.1. Thermal stability of $\text{Al}_{13}\text{CeMg}_6$

Fig. 1(a) and (b) display BSE images of the alloys annealed at 400 and 320 °C for 40 days, respectively. As can be seen in Fig. 1(a), the microstructure dominated by gray plate-like phase. The composition of this phase was determined to be  $\text{Al}_{65.3}\text{Ce}_{6.7}\text{Mg}_{28.0}$ , which is close to the stoichiometry  $\text{Al}_{13}\text{CeMg}_6$ . This observation is consistent with the previous reports [6–11]. According to the ICP and CA measurements, the composition of the sample annealed at 400 °C essentially shifts toward a composition with poor Ce (i.e.,  $\text{Al}_{65.68}\text{Ce}_{3.43}\text{Mg}_{30.89}$ , in at.%), compared to nominal and as-cast alloys. This is also supported by the observation of a certain amount of  $\text{Al}_3\text{Mg}_2$  phase (with composition  $\text{Al}_{61.9}\text{Mg}_{37.3}\text{Ce}_{0.8}$ ). In Fig. 1(b), it was found that the  $\text{Al}_{13}\text{CeMg}_6$  phase totally disappears. Besides the dominating dark-black phase, many needle-like bright phases appear. The composition of dark phase was determined by EPMA to be  $\text{Al}_{78.2}\text{Ce}_{21.3}\text{Mg}_{0.5}$  while the bright phase was to be  $\text{Al}_{62.2}\text{Mg}_{37.7}\text{Ce}_{0.1}$ , which correspond to the stoichiometry phases  $\text{Al}_3\text{Mg}_2$  and  $\text{Al}_{11}\text{Ce}_3$ , respectively. This fact illustrates that the compound  $\text{Al}_{13}\text{CeMg}_6$  is stable at 400 °C, but not stable at 320 °C. Moreover, the thermal stability of  $\text{Al}_{13}\text{CeMg}_6$  phase was further confirmed by XRD

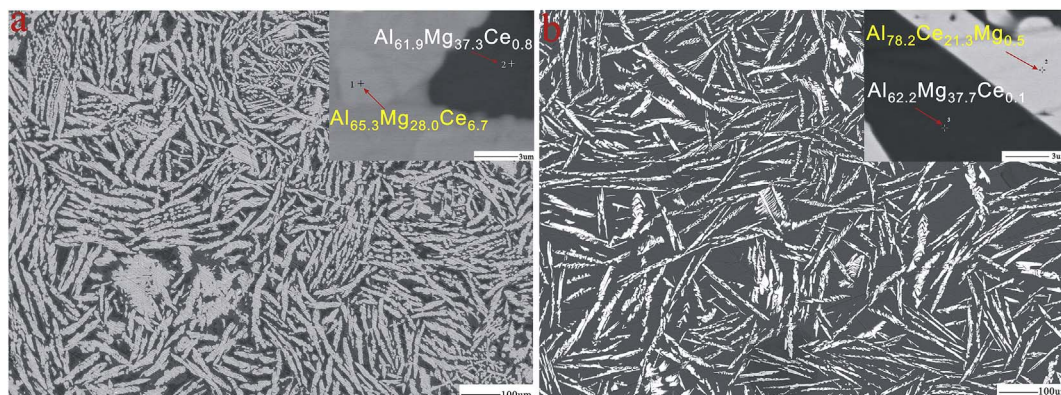


Fig. 1. BSE images of the alloys: (a)  $\text{Al}_{65.68}\text{Ce}_{3.43}\text{Mg}_{30.89}$  (in at.%) annealed at 400 °C for 40 days; (b)  $\text{Al}_{64.75}\text{Ce}_{3.30}\text{Mg}_{31.95}$  (in at.%) annealed at 320 °C for 40 days.

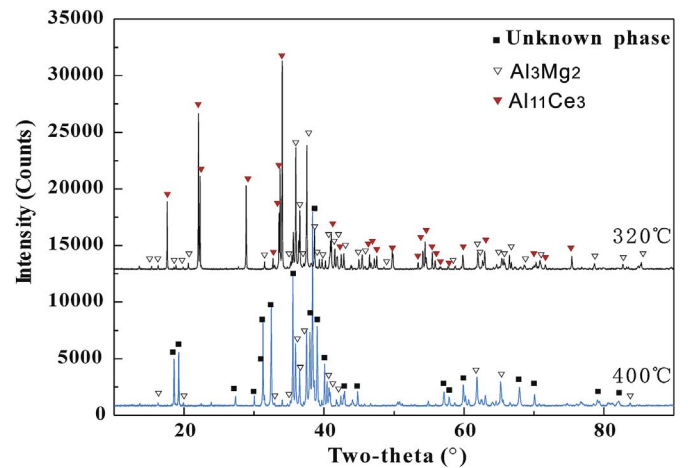


Fig. 2. XRD patterns of the alloys:  $\text{Al}_{64.75}\text{Ce}_{3.30}\text{Mg}_{31.95}$  (in at.%) annealed at 320 and  $\text{Al}_{65.68}\text{Ce}_{3.43}\text{Mg}_{30.89}$  (in at.%) annealed at 400 °C for 40 days.

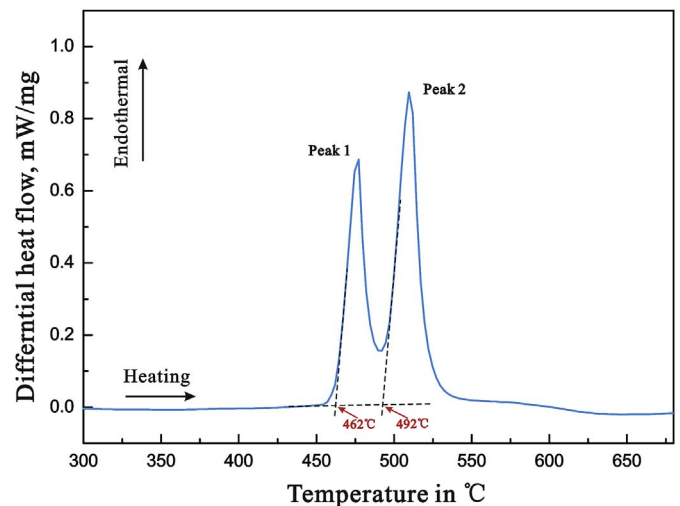


Fig. 3. DSC curve of alloy  $\text{Al}_{64.75}\text{Ce}_{3.30}\text{Mg}_{31.95}$  (in at.%) annealed at 320 °C for 40 days.

results. As shown in Fig. 2, for the sample annealed at 400 °C, the main diffraction peaks of the pattern cannot be explained by the reported phases [6,7]. The existence of a new compound which corresponds to  $\text{Al}_{13}\text{CeMg}_6$  phase is considered. Additionally, the presence of binary compound  $\text{Al}_3\text{Mg}_2$  can be clearly identified. However, for the sample annealed at 320 °C, only two binary compounds  $\text{Al}_{11}\text{Ce}_3$  and  $\text{Al}_3\text{Mg}_2$  could be identified. This is in good agreement with BSE observation and EPMA results.

Fig. 3 shows the heating curve generated from the DSC measure-

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