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ARTICLE

# Microstructure Evolution of Novel Al-Cu-Li-Ce Alloys during Homogenization

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**Abstract:** The present work is aimed to clarify the formation of the  $\tau_1(Al_8Cu_4Ce)$  phase and its formation mechanism in the high Cu content alloys with Ce addition, i.e high Cu/Li Al-Cu-Li-Ce alloys. The microstructure evolution of the alloy during the two-step homogenization annealing process was investigated. Results show that the coarse Ag+Mg enriched  $T_B$  (Al<sub>7</sub>Cu<sub>4</sub>Li) phase and the primary AlCuCe phase occur under solidification. Two types of the minor  $\tau_1$  phases are formed after homogenization. It is concluded that the  $\tau_1$  phase nucleation mechanism could involve either nucleation on an existing  $T_B$  phase by Ce diffusion from the Al matrix or the transformation of the primary AlCuCe phase into the  $\tau_1$  phase by Ce diffusion through the coarse primary AlCuCe phase shrink. It is deduced that the grain refinement is attributed to the primary AlCuCe, which can promote  $\alpha$  (Al) nucleation and further prohibit the grain growth for the experimental alloy.

**Key words:** novel Al-Cu-Li alloy; cerium element; segregation;  $\tau_1$  phase; homogenization

Al-Li alloys have received considerable interest because of their attractive high strength to weight ratio, large elastic modulus, small anisotropy, excellent resistivity to damage and good weldability. These characteristics make the alloys a very attractive choice in all weight-critical applications<sup>[1,2]</sup>. In past several decades, a number of researchers have paid attention to the microstructure and the mechanical properties of Al-Cu-Li alloys with a trace amount of Ag, Mg and Zr<sup>[3-7]</sup>.

The mechanical properties of the Al-Li-Mg alloys were improved and the negative effect of impurity Fe was controlled by Ce addition. The Ce addition was also reported to affect the ductility and the fracture toughness of 8090 alloy sheets rich in impurities of Fe, Si and alkali metals<sup>[8]</sup>. Once the Cu level is up to 5.8% in the present alloy system, an isothermal section of Al-Cu-Ce ternary system phase diagram<sup>[9]</sup> must be taken into consideration and the Ce containing precipitates observed in such a system might be  $\tau_1(Al_8Cu_4Ce)$  phase. Ce addition in the Al alloys usually has following three effects: (i) hindering the diffusion of the major elements in experimental

alloys and finally retarding the coarsening of primary strengthening phases, e.g. Xiao found that the addition of Ce up to 0.45wt% improved the thermal stability of the  $\Omega$  phase in Al-Cu-Mg-Ag base alloys<sup>[8]</sup>. (ii) forming primary AlCuCe phase which acted as nucleating agents for remainder liquid solidified to  $\alpha(AI)$  and combined with Ce atoms to segregate at the solidification front of the dendrites so as to increase the region of compositional supercooling and finally reduce the arm spacing of secondary dendrites<sup>[10]</sup>, e.g. grain size varied with the Ce content in Al-Cu-Mg-Mn-Ag alloys<sup>[11]</sup> and the dendritic structure could be refined, the morphology of precipitates changed from spherical to needle shape when Ce content varied from 0.1% to 0.4% (mass fraction) in 7055 Al alloy<sup>[12]</sup>. In addition, J. P. Lai et al. [13] also found that addition of Ce could remarkably refine the as cast grains and eutectic microstructure. (iii) forming  $\tau_1$  dispersoids during homogenization and thermal mechanical process. These types of particles played a high antirecrystallized effectiveness in the alloy during following heat treatment<sup>[14]</sup>.

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The aim of the present study is to investigate the feature of the microstructure evolution and the possibility of the formation of the  $\tau_1$  phase in the Ce-added novel Al-Cu-Li complex alloy system during homogenization.

#### 1 Experiment

The alloy with a chemical composition of Al-2.46Cu-5.08Li -0.054Ce-0.1Ag-0.45Mg-0.04Zr (at%) was prepared. Master alloys of Al-Zr, Al-Ce and Al-Cu and pure elements of Ag, Mg, Li and Al were melted in a vacuum induction melting furnace under a controlled atmosphere of argon gas, using high pure graphite crucible. Lithium addition was made by plunging Li wrapped in aluminium foil. Casting was carried out under argon<sup>[15]</sup>. The ingot was homogenized using two-step homogenization cycle at 470 °C, 8 h + 510 °C, 16 h in a salt bath.

Specimens cut from the ingot as-cast and homogenized were observed by optical and electronic microscope. Grain structure of the specimens was determined after being anodized with Barker's reagent (1.8% HBF4 solution with a voltage of 20~30 V and a current of 0.5~1.5 mA) and viewed with cross-polarized light on Leica DMILM optical microscope (OM). Phase analysis was performed with X-ray diffractometer (XRD, Rigaku D/ max 2500). A Quanta-200 environmental scanning electron microscope (SEM) was used for the evaluation of the microstructural features of the alloy. The wave length-dispersive X-ray spectrometer (WDS) microanalysis of the intermediate phases in arbitrarily selected area was performed on JEOL JXA-8230 electron microprobe analysis (EPMA) instrument. In addition, differential scanning calorimeter (DSC) analysis of specimens by NETZSCH STA 449C was carried out under argon atmosphere to analyze the transition of phases during the homogenization treatment.

#### 2 Results

#### 2.1 Microstructure observation

Optical micrographs of the grain structures of the alloy are presented in Fig.1. The alloy in as-cast condition consists of typical large and dendritic grains. The average grain size is 200~300 µm, and the dendrite arm spacing (DAS) is ~30 µm (Fig.1a). Compared to other Al-Cu-Li alloy, e.g. 1469 Al-Cu-Li-Sc (grain size 400~500 µm)<sup>[16]</sup> and Al-Cu-Li-Mn-Zr-Ti alloy (DAS 80~100 µm)<sup>[17]</sup>, the remarkable refinement of the columnar grains containing the dendritic substructure with complex alloying by Ce and Zr is observed; DAS is reduced simultaneously. A great degree of dendritic microsegregation exists within the grains. After homogenization, the dentritic grain structure is eliminated basically, as shown in Fig.1b. The grain boundaries become level and smooth.

Fig.2 shows the SEM images for the solidification and homogenization microstructure of the alloy. Coarse continuous non-equilibrium phases are distributed both within the interiors of dendrite cells and on the dendrite boundaries that

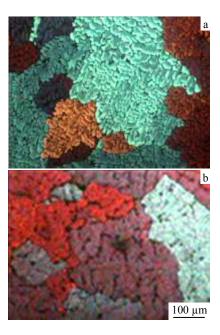


Fig.1 OM images of the experimental alloy with cross polarized light: (a) as-cast and (b) homogenized

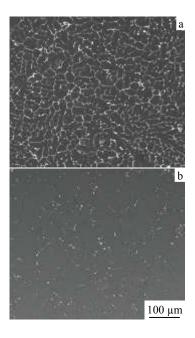


Fig.2 Backscattered SEM images of the experimental alloy: (a) as-cast and (b) homogenized

comprise the shell, as the white net-like parts shown in Fig.2a. However, in the homogenized structure, vast arrays of discontinuous small particles are visible instead of those coarse continuous phases, as shown in Fig.2b. This indicates that most of the continuous non-equilibrium phases are dissolved and some other phases are formed.

## 2.2 Segregation of the alloying elements before and after homogenization

Fig.3 is the SEM images and the elements mappings of the

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