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Enhancing grain refinement efficiency and fading resistance of Al–B master alloys processed by equal channel angular pressing

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An Al–3% B master alloy has been subjected to equal channel angular pressing (ECAP). The grain refining performance and fading resistance of an Al–3% B master alloy on a commercial purity Al (CPA) have been evaluated. The effect of the number of ECAP passes on the size and the distribution of the AlB_2 particles, the grain size of CPA ingots with and without adding the Al–3% B master alloy subjected to ECAP have been investigated. The mean size of AlB_2 particles was significantly reduced from ~34 to ~12 μm after four ECAP passes. Fine blocky AlB_2 particles were uniformly distributed in the Al matrix. It has been revealed that when it was inoculated by the Al–B master alloy subjected to ECAP, the grain size of α-Al was decreased from ~1200 to ~180 μm after four ECAP passes, beyond that, the grain size tends to be saturated. It has been proved that grain refinement efficiency and fading resistance of the Al–3% B master alloy subjected to ECAP in CPA ingots was enhanced.

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I. INTRODUCTION

Pure aluminum has been widely used for manufacturing industrial products due to its low density, high ductility, good electrical, and heat conductivity. However, the coarse grains deteriorate mechanical properties.[1,2] The most effective way to obtain fine grain size in the Al matrix of the castings is to introduce nuclei into the melt using grain refiners, which usually contain $TiAl_3$, TiB_2, and (Al, Ti)B_2 particles.[3–5] $TiAl_3$ is a more powerful nucleating substrate than TiB_2 and AlB_2.[6] TiB_2 does not nucleate grains alone.[7] Its nucleation potential is significantly enhanced by the formation of a monolayer of Al_3Ti compound on the TiB_2 surface.[8] Al–B master alloys have been reported to possess a better grain refining efficiency than that of Al–5% Ti–1% B.[9–11] However, the potential of AlB_2 particles in the grain refinement of purity aluminum is still inconsistent.

The AlB_2(111) surfaces and Al(111)/AlB_2(0001) interfaces were studied by first-principles calculations to clarify the heterogeneous nucleation potential of α-Al grains on AlB_2 particles in purity aluminum, elucidating the poor nucleation potential of α-Al grains on AlB_2 particles.[12,13] The block-like AlB_2 particles have better grain refinement efficiency and resistance against fading on pure aluminum even at an addition concentration below the Al–B eutectic.[4] However, it was observed that AlB_2 particles cannot refine the grains in commercial purity Al (CPA).[14] Therefore it is fundamentally important to investigate the morphology and distribution of boride particles in the master alloy.

Equal channel angular pressing (ECAP) has been proved to be one of the most promising severe plastic deformation techniques for controlling the distribution and size of particles.[15] Al–5% Ti and Al–5% Ti–0.25% C master alloys have been processed by ECAP for enhancing the grain refinement efficiency.[16–18] As a result of ECAP, a transition from platelet shapes to blocky particles has been observed for the Al_3Ti phase. However, the role of AlB_2 particles processed by ECAP to refine grains in CPA ingots is not clear.

The present work is motivated to explore the possibility of enhancing the grain refining efficiency in CPA ingots inoculated by the Al–B master alloy subjected to the ECAP process. The effect of the number of ECAP passes on the size of the AlB_2 particles, the grain size of CPA ingots with and without adding the Al–3% B master alloy have been investigated. The grain refining performance and fading resistance of the Al–3% B master alloy on the CPA ingots have been evaluated.

II. EXPERIMENTAL PROCEDURES

The Al–3 wt% B master alloy ingots with dimensions of 500 × 250 × 40 mm^3 were supplied by Xuzhou Huasheng Aluminum Co. Ltd. (Jiangsu, China) After removing the oxidation layer and surface defects, the billets with a size of 12 × 12 × 80 mm^3 were cut in the longitudinal direction. The CPA (99.79 wt% Al) was used to verify the grain refining performance of the Al–3% B refiner before and after ECAP processing. The composition of the CPA ingot and Al–3% B master alloy is shown in Table I.

ECAP of the Al–B master alloy was conducted at room temperature with a pressing speed of 0.2 mm/s using a die with a channel angle $\phi = 110°$ and the outer arc of curvature angle $\psi = 0°$. All samples were pressed using a molybdenum disulfide lubricant and without rotation between the consecutive passes, which has been designated as route A.[19] The number of ECAP passes was equal to 1, 2, 4, 6, and 8 passes.

The Al–B master alloy in states either before or after the ECAP process was added into the Al melt heated up to 1033 K using the 5 kW electric resistant-heating furnace. After three times of mechanical stirring of the melt, the Al melt was poured into a ring steel mold with an outside diameter of 45 mm and inner diameter of 25 mm. The sample was cut horizontally at 20 mm distance from the bottom. The sectioned surface was polished and etched in a hydrofluoric acid (2.5 mL), nitric acid (12.5 mL), and water (85 mL) reagent, and then the macrostructural evolution was investigated.

The chemical element B (introduced by the Al–B alloy subjected to four passes of ECAP) was added into the Al melt for the grain refinement performances of CPA ingots. The concentration of B in the Al melt was 0.02, 0.04, 0.06, 0.08, 0.10, 0.12, 0.14, 0.16, 0.18, and 0.20 wt%, which was added at 1033 K for 60 min and three times of mechanical stirring.

To study the fading resistance, the Al–B alloy after four ECAP passes was added into the Al melt resulting in an addition of 0.04% B in CPA ingots. The resistance against fading was studied after different holding time.

The mean size of α-Al grains was calculated by using a mean linear intercept method. The pure Al cast samples with additions of Al–B refiner before and after ECAP were studied by the method of optical microscopy. All samples were polished and etched in a hydrofluoric acid (1 mL), nitric acid (5 mL), and hydrochloric acid (5 mL) reagent. X-ray diffraction (XRD) measurements were performed on a D/max-2500 pc X-ray diffractometer (Rigaku, Tokyo, Japan) equipped with a Cu target

TABLE I. Compositions of the commercial pure Al and Al–3% B master alloy (wt%).

Samples	Elements			
	Si (%)	Fe (%)	B (%)	Al
Pure Al	0.05	0.16	...	Bal.
Al–3B	0.11	0.18	2.15	Bal.

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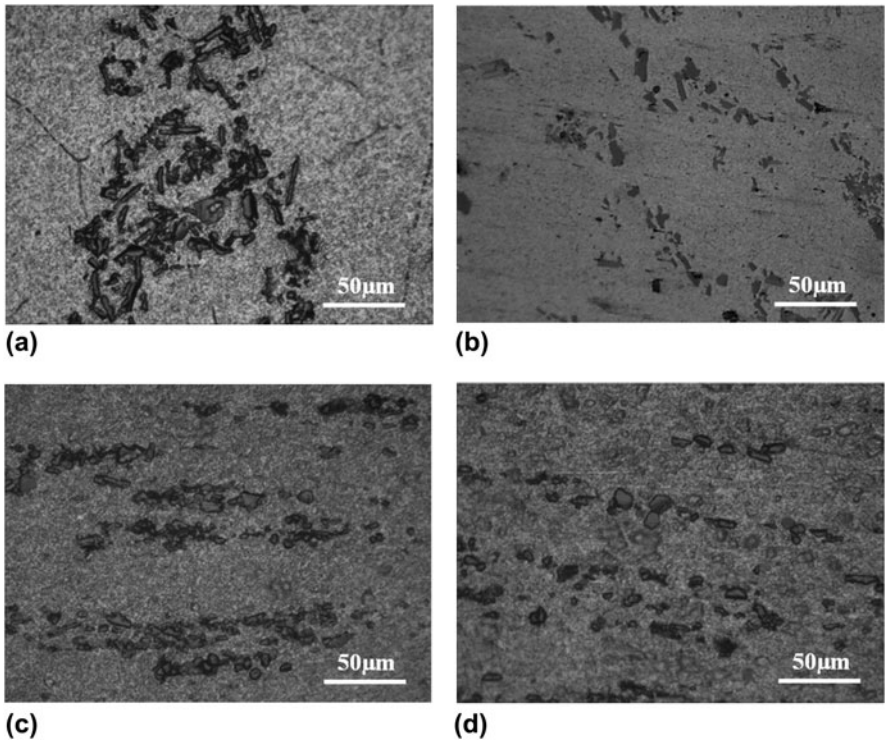


FIG. 1. Optical micrographs of the Al–3% B master alloy before and after ECAP: (a) 0 pass, (b) 1 pass, (c) 4 passes, and (d) 6 passes. The AlB_2 particles were fragmented into the fine and blocky particles and distributed uniformly in the matrix.

operating at 40 kV/100 mA. A series of θ–2θ scans were performed to record the XRD patterns between 30° and 100° at room temperature.

III. RESULTS AND DISCUSSION

A. Effect of ECAP passes on the microstructure of the Al–B master alloy

Optical photographs of the Al–3% B alloy before and after ECAP are shown in Fig. 1. The needle-like AlB_2 particles in the cluster were observed in the as-cast Al–3% B alloy. They are the typical features in the conventional Al–B master alloys.[10,13] The AlB_2 particles were fragmented into fine and blocky particles, and distributed uniformly in the matrix, as represented in Figs. 1(b)–1(d). There are two types of Al-borides in Al–B master alloy, AlB_2 and AlB_{12}. The former is favored thermodynamically[10,20] while the latter is a high temperature phase which is stable only above 975 °C. The existence of the AlB_2 phase was identified by the XRD analysis (Fig. 2). The presence of the AlB_{12} phase was not revealed in the XRD patterns.

As can be seen in Fig. 3, there is a significant decrease in the size of the AlB_2 particles with an increase of the number of the ECAP passes. It reduces from ~34 to ~12 μm after four ECAP passes. Beyond that, there is a limitation for the mean size of AlB_2 particles equal to about 10 μm. Similarly, the mean size of Al_3Ti particles after three or five ECAP

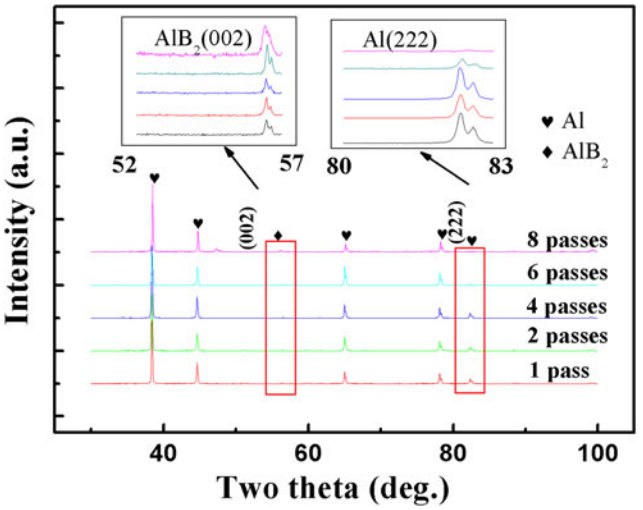


FIG. 2. XRD patterns of the Al–3% B master alloy processed by ECAP.

passes is to be about 10 μm.[17,18] It indicates that it is feasible to refine needle-like AlB_2 particles in the cluster into the blocky particles by the ECAP process.

B. Grain refining performance of CPA ingots inoculated by the Al–B master alloy after ECAP

The macrostructures of CPA ingots inoculated by the Al–B master alloy before and after the different numbers of ECAP passes are shown in Fig. 4. It is shown that

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without the addition of Al–B refiner, the majority of grains are columnar and the volume fraction of the coarse equiaxed α-Al grains is small in the center. When the Al–B refiner was added to the Al melt, the α-Al columnar grains and the equiaxed grains were refined, and they are smaller than that without adding the Al–B alloy. After adding the Al–B refiner by ECAP to the Al melt, there are a large number of fine equiaxed α-Al grains and some fine columnar grains, showing the better grain refinement efficiency than that before ECAP. Furthermore, the proportion of the fine equiaxed α-Al grains tends to expand form the center to the edge with increasing the number of ECAP passes.

A free growth of crystal from the particle model was proposed by Greer et al.[21] The undercooling required for free growth ΔT_{fg} is given by

$$\Delta T_{fg} = \frac{4\sigma}{\Delta S_v d} \quad , \tag{1}$$

where σ is the solid–liquid interfacial energy, ΔS_v is the entropy of fusion per unit volume, d is the nucleant particle diameter. As the undercooling is increased, progressively smaller particles become centers for free growth, which occurs as soon as the required undercooling is reached. However, the smaller particles do not mean the better grain refine efficiency because the onset of growth is controlled by the growth restriction rather than by nucleation. The relation of the mean grain size with the reciprocal of the size of AlB_2 particles is shown in Fig. 5. The grain size decreases rapidly before the size of AlB_2 particles is reduced to ~12.3 μm, after that it slightly increases. It is in agreement with that there appears to be an optimum average particle diameter,[21] in this case of ~12.3 μm.

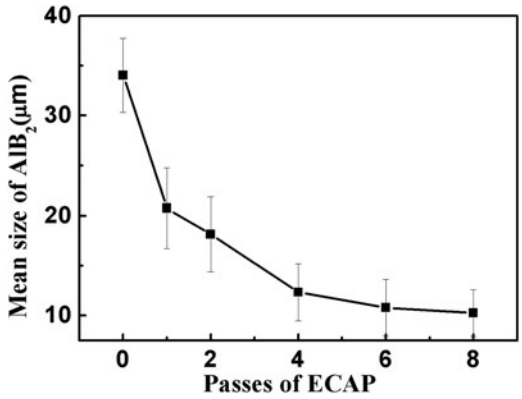


FIG. 3. The mean size of AlB_2 particles in the Al–3% B master alloy against the number of ECAP passes.

C. Effect of B contents on grain refining performance of CPA ingots

The macrostructures of CPA ingots inoculated by the Al–B master alloy before and after four passes of ECAP are shown in Fig. 6. The zone of the equiaxed grains in the center of the CPA ingots inoculated by the Al–B master alloy after ECAP is larger than that of the counterparts before ECAP. The effect of B contents on the grain size of CPA ingots is shown in Fig. 7(a). The grain size of α-Al was reduced from the initial ~1200 to 247 μm (before ECAP) and 219 μm (after ECAP), respectively, when the B content is 0.04%. After that the grain size is slightly decreased.

When inoculating CPA ingots with the Al–B master alloy, not only the AlB_2 particles were needed to be heterogeneous nucleation substrates, but also the impurity elements such as Fe and Si are also required to trigger heterogeneous nucleation.[13] The main impurity elements of Fe and Si in the Al–B master alloy are taken into account in the melt. The final grain size is

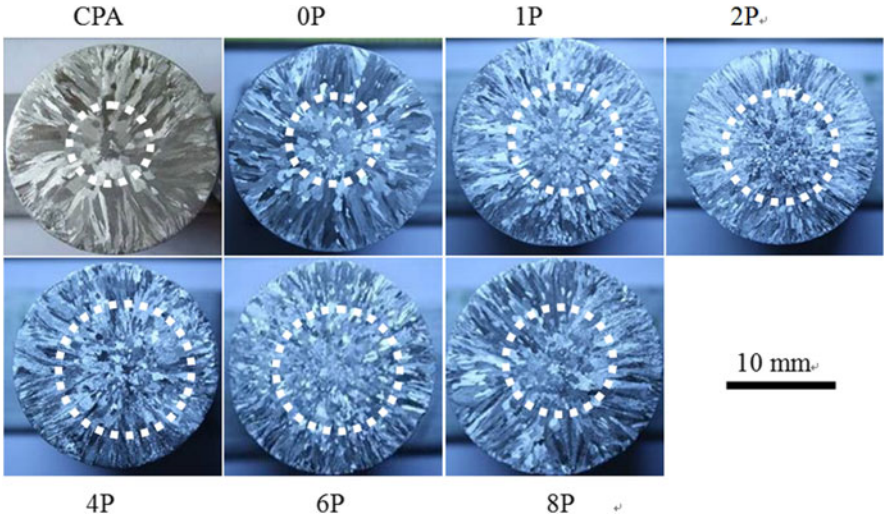


FIG. 4. Macrographs of CPA ingots inoculated by the Al–3% B master alloy against the number of ECAP passes.

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determined by both the nucleant particles and the solute restriction of grain growth.[22] The average grain size can be described as[23–25]

$$d = a + \frac{b}{Q} \quad , \tag{2}$$

where a and b are empirical constants. The intercept, a, corresponds to the maximum number of successfully activated nuclei and the slope, b, is related to the potency of the nucleant particles. Q was originally derived to be inversely proportional to the growth rate of the primary phase, defined as the grain growth restriction (GRF):

$$Q = m(k-1) \cdot C_0 \quad , \tag{3}$$

where m is the slope of the liquidus, C_0 is the solute concentration in the melt, and k is the equilibrium partition coefficient. The values of parameters are $m = -6.6$ and $k = 0.11$ for Si, $m = -3.0$ and $k = 0.02$ for Fe in the Al melt.[23,24] The mean grain size of α-Al, d, varies with $1/Q$ is shown in Fig. 7(b). The equation between d and $1/Q$ for the addition of Al–B refiner without ECAP and after four ECAP passes is fitted, respectively, by the equations:

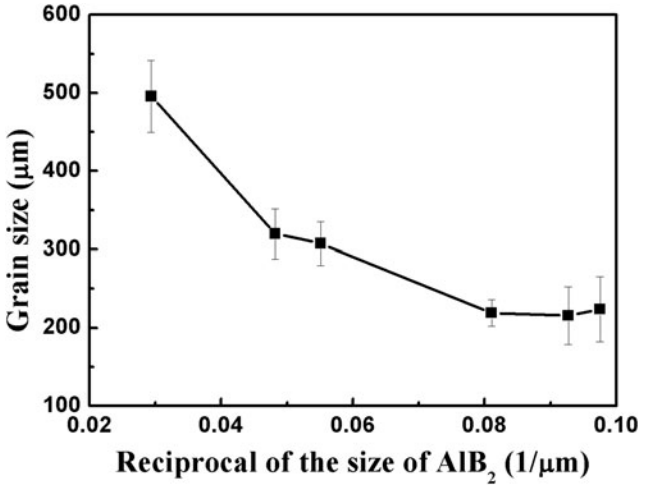


FIG. 5. The mean grain size versus the reciprocal of the size of AlB_2 particles.

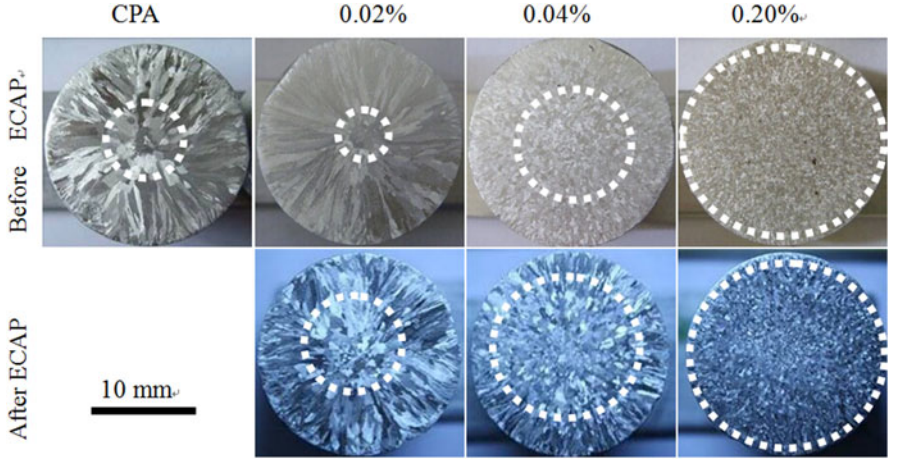


FIG. 6. Macrostructure of CPA ingots inoculated by the Al–B master alloy before and after four passes of ECAP. The addition of B contents into the CPA ingots is 0.02, 0.04, and 0.20%.

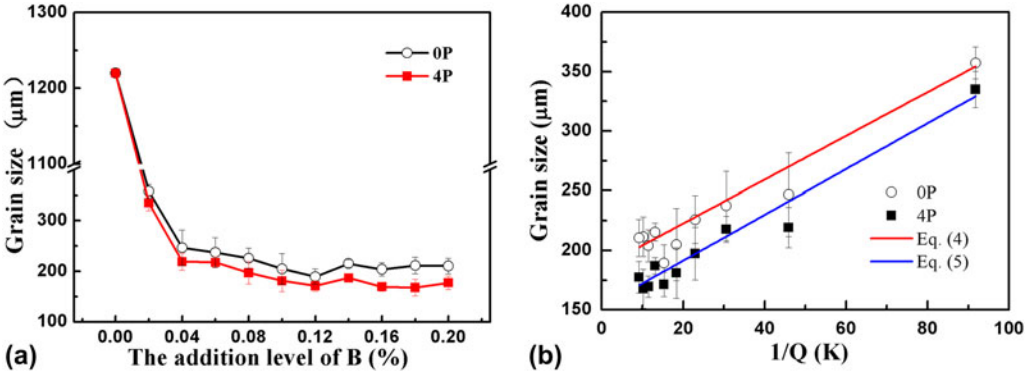


FIG. 7. (a) The effect of B contents on the mean grain size of α-Al and (b) the mean grain size of α-Al versus the $1/Q$.

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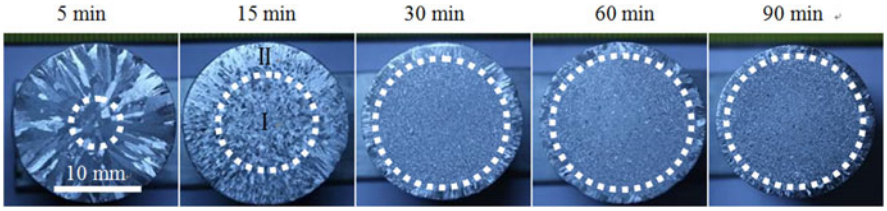


FIG. 8. Macrostructure of CPA ingots inoculated by the Al–B master alloy subjected to 4 ECAP passes with different holding times.

$$d = 186 + \frac{1.84}{Q} \quad , \tag{4}$$

$$d = 153 + \frac{1.92}{Q} \quad . \tag{5}$$

In Eqs. (4) and (5), there is little difference between the slopes. While more nuclei are introduced, the term, a, decreases,[24,25] indicating that ECAP processing could introduce more nuclei particles in one unit volume because the AlB_2 particles were fragmented during ECAP. Therefore, the impurity elements cannot be ignored since they are indispensable to restrict the growth of initial columnar grains.[13]

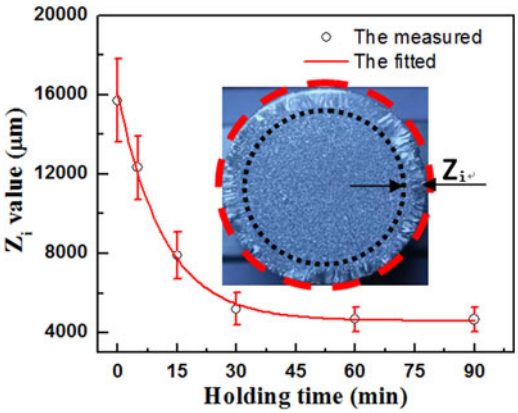


FIG. 9. The length of the initial columnar grains Z_i with the holding time t.

D. Enhancement of fading resistance of the Al–B master alloy by ECAP

To investigate the resistance against fading, the Al–B alloy after four passes of ECAP was added into the Al melt with an addition of 0.04% B. The macrographs of CPA ingots inoculated by Al–B refiner subjected to ECAP with different holding times are shown in Fig. 8. These macrographs are characterized by the equiaxed zone I and columnar zone II, respectively. With an increase of holding time, the equiaxed zone I is expanded. The sample was mainly covered by the equiaxed grains in 30 min. As the holding time increases, a distinct increase of the equiaxed zone is not observed, which means the Al–B refiner subjected to ECAP has an excellent resistance against fading.

Figure 9 represents the length of the initial columnar grain Z_i, which was measured by the width of zone II, with holding time t. The fitted curve is determined by

$$Z_i = 4598 + 11{,}453 \cdot e^{-0.086t} \quad . \tag{6}$$

It is noted that the zone of the initial columnar grain Z_i is decreased with the holding time, after 30 min, the variation of the initial columnar grain Z_i is slight. On the contrary, the length of the initial columnar grain Z_i was linearly proportional to the square root of the holding time t when high purity aluminum (99.85% Al) was inoculated by the Al–3B master alloy.[13] The main reasons are not only originated from the lower cooling rate under an approximately isothermal condition but also due to without the mechanical stirring of the melt. AlB_2 particles with the size below 12.8 μm could survive after settling. As it was above mentioned in Fig. 3, after four passes of ECAP, there are a sufficient number of AlB_2 particles with the diameter of ~12 μm. When the mechanical stirring is applied to the Al melt, it will not lose their grain refine efficiency, resulting in an excellent resistance against fading. The enhanced resistance against fading was also found in Al–5% Ti–0.25% C master alloys subjected to ECAP.[16]

IV. CONCLUSIONS

The effect of the number of ECAP passes on the size of the AlB_2 particles, grain size of CPA ingots with and without adding the Al–3% B master alloy have been investigated. The grain refining performance and fading resistance of the Al–3% B master alloy on the CPA ingots have been evaluated. It is concluded that

(1) The mean size of AlB_2 particles was significantly reduced from ~34 to ~12 μm after four passes of ECAP. With increasing the number of ECAP passes, there is a limitation for the mean size of AlB_2 particles equal to about 10 μm. The fine blocky AlB_2 particles were uniformly distributed in the Al matrix.

(2) Inoculated by the Al–B master alloy subjected to ECAP, the grain size of α-Al was decreased from

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~1200 to ~180 μm after four passes of ECAP. With an increase of the number of ECAP passes, the grain size tends to be saturated. The grain size of α-Al decreases rapidly before the size of AlB_2 particles is ~12.3 μm, after that it slightly increases, indicating that there appears to be an optimum average particle diameter, in this case of ~12.3 μm.

(3) When the B content is 0.04%, the grain size of α-Al was refined from the initial ~1200 μm to 247 μm (before ECAP) and 219 μm (after ECAP), respectively. With increasing the B contents in the Al melt, the grain size is slightly decreased.

(4) For the addition of Al–B refiner without ECAP and with four passes of ECAP, the grain size of α-Al, d, could be well fitted by the GRF: $d = 186 + \frac{1.84}{Q}$ and $d = 153 + \frac{1.92}{Q}$, respectively. It indicates that the ECAP process could introduce more nuclei particles in one unit volume because the coarse AlB_2 particles were fragmented during the ECAP. Meanwhile, the impurity elements cannot be ignored since they are indispensable to restrict the growth of the initial columnar grains.

(5) The Al–B master alloy subjected to ECAP has an enhanced resistance against fading. The length of the initial columnar grain Z_i is decreased with the holding time, $Z_i = 4598 + 11{,}453 \cdot e^{-0.086t}$.

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