Grain refinement of pure aluminum by direct current pulsed magnetic field and inoculation

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Abstract: The combined effects of direct current pulsed magnetic field (DC-PMF) and inoculation on pure aluminum were investigated, the grain refinement behavior of DC-PMF and inoculation was discussed. The experimental results indicate that the solidification micro structure of pure aluminum can be greatly refined under DC-PMF. Refinement of pure aluminum is attributed to electromagnetic undercooling and forced convection caused by DC-PMF. With single DC-PMF, the grain size in the equiaxed zone is uneven. However, under DC-PMF, by adding 0.05% (mass fraction) Al−5Ti−B, the grain size of the sample is smaller, and the size distribution is more uniform than that of single DC-PMF. Furthermore, under the combination of DC-PMF and inoculation, with the increase of output current, the grain size is further reduced. When the output current increases to 100 A, the average grain size can decrease to 113 μm.

Key words: direct current pulsed magnetic field; pure aluminum; inoculation; grain refinement; solidification micro structure

1 Introduction

Grain refinement is considered to be an important melt treatment during the casting of aluminum alloy. It is well known that properties of the castings are connected with its as-cast structure; fine and equiaxed grain instead of coarse dendritic grain is good for the improvement of mechanical properties. In recent years, many approaches of refining solidification structure of metals have been developed, including chemical methods like inoculation [1], and external physical field methods such as mechanical or electromagnetic stirring [2], ultrasonic treatment [3], electromagnetic vibration [4], electric current pulse [5,6], pulsed magnetic field (PMF) [7−9].

The widely used method of refinement in the foundry industry is inoculation [10] with introduction of specific substances, called inoculants, into the metal bath, which increases the grain density as a result of creation of new particles in consequence of hampering grain growth rate. This leads to an increase of the equiaxed crystal zone, which guarantees the improvement of mechanical properties and the decrease of component

segregation and hot cracks. However, this method of inoculation of the primary structure is limited to pure metals, because inoculants decrease the degree of purity. Without affecting the refinement of solidification structure of pure aluminum, decreasing the amount of inoculants to satisfy the degree of purity is necessary. So a novel method of refinement of pure aluminum may be potential for industry.

PMF processing has several advantages such as simple equipment, high efficiency, low investment, extensive alloy application scope and low pollution, which make PMF process become one of the most promising new techniques to refine the solidified structure [8,9]. BAN et al [7] applied a pulsed magnetic field to 2124 Al alloy melt and found an evident modification occurring in the solidification structures. GAO et al [8] studied the structural transformation in pure Al under external PMF. WANG et al [9] treated AZ31 alloy with PMF and obtained remarkable microstructure refinement of magnesium alloy.

However, to the best of our knowledge, few studies have referred to direct current pulsed magnetic field (DC-PMF) and the combination of DC-PMF and

inoculation in refinement of pure aluminum. In this work, the grain refinement of pure aluminum by DC-PMF process and the combination of DC-PMF and inoculation were studied, and the refinement mechanism was analyzed.

2 Experimental

The experimental setup consists of direct current switch power supply, frame, and coil. The switch power supply provides the on-off time in the range from 0.1 to 9999 s, the voltage ranges from 0 to 100 V and the current changes from 0 to 200 A. The schematic diagram of the experimental solidification apparatus is shown in Fig. 1. Commercial pure aluminum (99.7%) was used as the experimental materials. Two different kinds of processes were adopted in this experiment. The pure aluminum was melted in a graphite crucible by a resistance furnace and heated to 1073 K. After holding at 1073 K for 20 min, when the temperature of the melt decreased to 1023 K, the melt was poured into a graphite mold (40 mm in diameter and 70 mm in height) that was preheated to 873 K, and the pulsed magnetic field of different intensities was applied. K-type thermocouples with a diameter of 3 mm were 20 mm apart from the mold wall, and put into the melt 30 mm to measure the temperature of the melt. The pure aluminum for the second process was also melted in a graphite crucible by a resistance furnace and heated to 1073 K. After holding at 1073 K for 20 min, 0.05% (mass fraction) Al−5Ti−1B was added and the melt was held at 1053 K for 10 min. When the temperature of the melt decreased to 1023 K, the melt was poured into the preheated graphite mold and DC-PMF of different intensities was applied. The detailed experimental parameters are listed in Table 1.
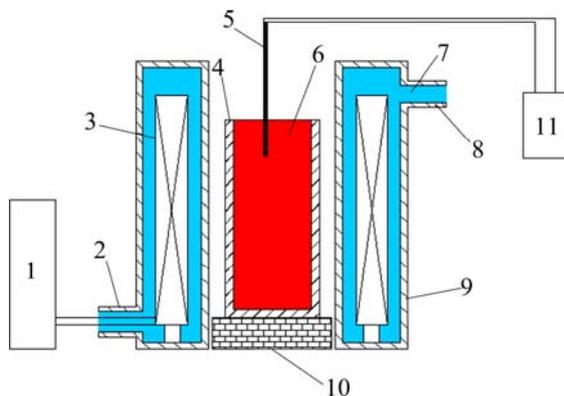
Table 1 Experimental parameters

Specimen	Output voltage/ V	Output current/ A	Frequency of DC-PMF/ Hz	PMF treated temperature/ K	Ti content in melt/ 10^{-6}
1	–	–	–	–	–
2	100	60	5	1023	–
3	100	80	5	1023	–
4	100	100	5	1023	–
5	–	–	–	–	25
6	100	60	5	1023	25
7	100	80	5	1023	25
8	100	100	5	1023	25

The specimens were cut longitudinally along the central plane, and then ground and polished using standard metallographic procedures for macrostructure examination. The etched regent for macrostructure was a mixed acid of 75 mL HCl, 25 mL HNO_3 and 5 mL HF. The grain size measurements were carried out by mean linear interception method using image analyzer.

3 Results

Figure 2 shows the macrostructures of pure

Fig. 1 Schematic diagram of experimental apparatus: 1—Direct current switch power supply; 2—Cooling water inlet; 3—Pulsed magnetic coil; 4 — Graphite mold; 5 — K-type thermocouple; 6—Molten metal; 7—Cooling water; 8—Cooling water outlet; 9—Frame; 10—Firebrick; 11—Paperless recorder
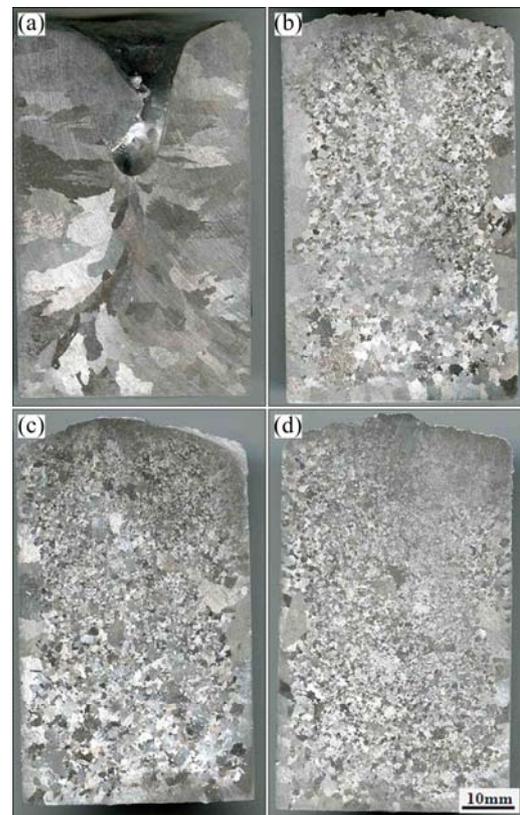

Fig. 2 Macrostructures of pure aluminum under different conditions: (a) Without DC-PMF; (b) With DC-PMF of output current 60 A; (c) With DC-PMF of output current 80 A; (d) With DC-PMF of output current 100 A

aluminum under different conditions. As shown in Fig. 2(a), without DC-PMF treatment, the coarse columnar grains can be seen on the whole section of the ingot. With DC-PMF treatment, fine and equiaxed grains are observed as shown in Figs. 2(b)−(d). Therefore, the solidification structure of pure aluminum can be remarkably refined under the DC-pulsed magnetic field. With the increase of output current, the area of equiaxed crystals increases and grain size is further reduced. However, the distribution of grain size is not homogeneous, there are still some coarse equiaxed grains near the bottom of ingots and inside the sample.

Figure 3 depicts the thermal histories for the two samples with and without DC-PMF measured by K-type thermocouples and recorded by a paperless recorder. A platform is clearly observed on each cooling curve, referred as equilibrium nucleation temperature. Without DC-PMF, the nucleation temperature is about 930 K. However, the nucleation temperature increases to 931 K under DC-PMF. Hence DC-PMF can introduce a very small change in nucleation temperature.
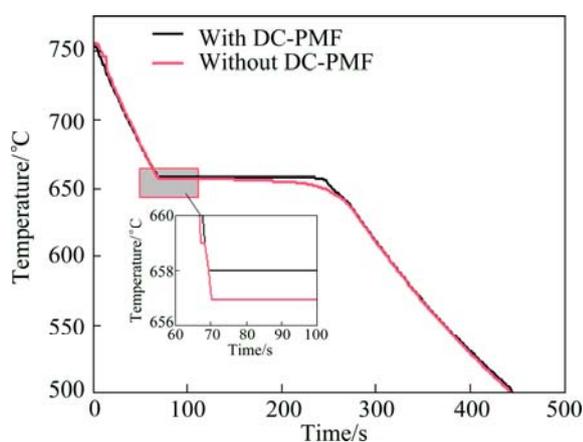

Fig. 3 Measured cooling curves of pure aluminum with and without DC-PMF at output current of 100 A

Figure 4 shows the macrostructures of pure aluminum with the combination of DC-PMF and Al−5Ti−1B. As shown in Fig. 4(a), after adding 25 g/t Ti and 5 g/t B into the melt, it can be seen that there are a few coarse equiaxed grains surrounded by columnar grains in the ingot. However, with the combination of DC-PMF and Al−5Ti−B, more fine and equiaxed grains are observed as shown in Figs. 4(b)−(d). The grain size is relatively uniform in the equiaxed area of the ingots, which is good to the uniformity of mechanical performance of the sample. Therefore, refinement by the combination of DC-PMF and inoculation is better than that by single DC-PMF.

Figure 5 shows the average grain size of ingots with different parameters. Without DC-PMF or inoculation, the average grain size is about 7.3 mm, after adding
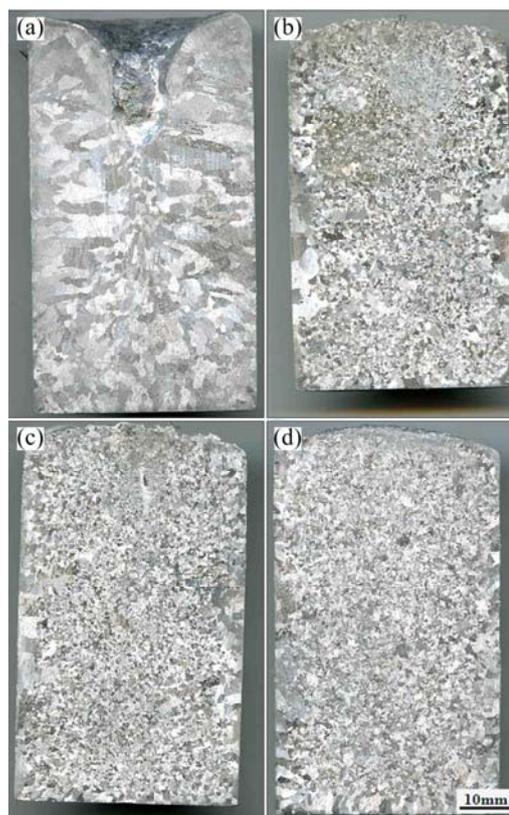

Fig. 4 Macrostructures of pure aluminum ingots with inoculation (a), with combination of inoculation and DC-PMF at output current of 60 A (b), 80 A (c), 100 A (d)
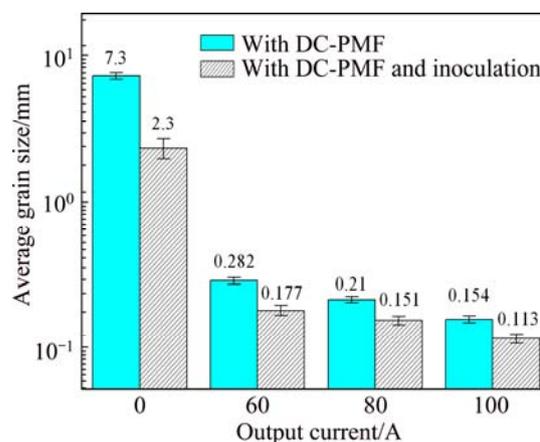

Fig. 5 Average grain size of pure aluminum ingot under different conditions

0.05% Al−5Ti−B, the average grain size decreases to 2.3 mm. However, under DC-PMF, the average grain size reduction is obvious. It is clear that grain size decreases from 282 and 210 μm to 154 μm with the increase of the DC-PMF output current from 60 and 80 A to 100 A, respectively. With the combination of DC-PMF and Al−5Ti−1B, the grain size further reduces from 177 and 151 μm to 113 μm with the variation of DC-PMF output current from 60 and 80 A to 100 A, respectively.

It should be noted that there is an obvious difference in homogeneity of grain sizes between the two processes. Figure 6 depicts the histograms of the measured grain size under DC-PMF output current of 100 A and the combination of DC-PMF and inoculation. With DC-PMF treatment only, the distribution of grain size is not uniform; there is still a certain ratio of coarse grains, as shown in Fig. 6(a). However, with the combination of DC-PMF and inoculation, the grain size distributes much more uniformly. And the grain size is a normal distribution, which is mainly around the average size as shown in Fig. 6(b).
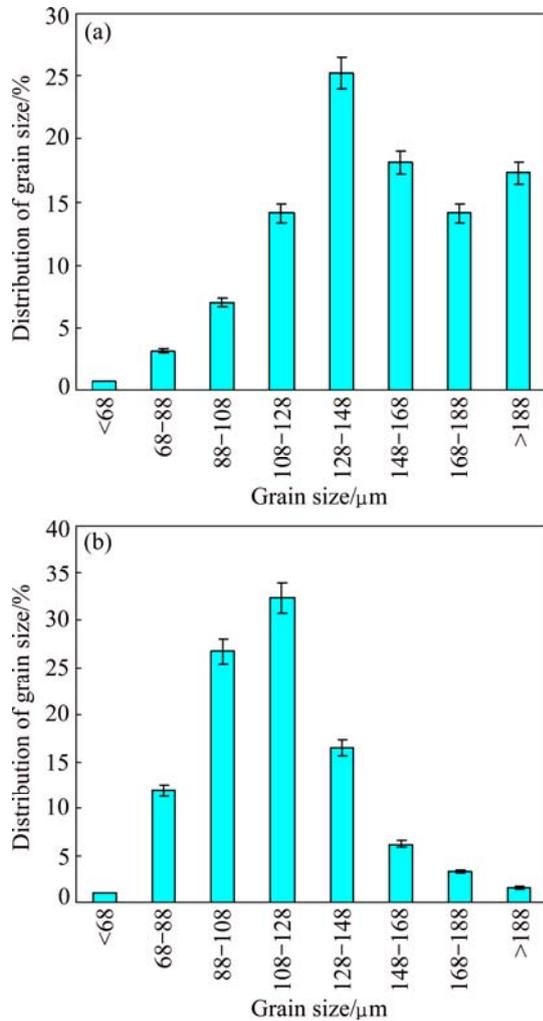

Fig. 6 Histograms of measured grain size with DC-PMF at output current of 100 A (a), with DC-PMF and inoculation at output current of 100 A (b)

4 Discussion

When a time-varying DC-PMF is imposed into the melt, according to Maxwell's law, it will generate an induced current density J in the melt. As a result, a Lorentz force is produced by the interaction between the induced current and the variable magnetic field, i.e.

$F=J\times B$, where F is the Lorentz force; J is the current density generated in the metal; and B is the magnetic flux density.

The Lorentz force can be decomposed into a tangential component and a radial component. The tangential component causes the melt flow along the tangential direction. The radial component produces a pressure p on the melt, which can be expressed as [11]

$$p = \frac{B^2}{2\mu} \tag{1}$$

where μ is the magnetic permeability of the melt.

According to the Clapeyron equation, a variation of temperature will be generated under the influences of the magnetic pressure p. The change of the equilibrium melting temperature ΔT caused by DC-PMF can be expressed as [12]

$$\Delta T = \frac{T_m \Delta V}{\Delta H_m} \int \mathrm{d}P \tag{2}$$

where ΔV is the volume difference between the solid phase and liquid phase; T_m is the melting point; and ΔH_m is the fusion enthalpy.

During the solidification of metal, ΔV and ΔH_m are positive, and so the value of ΔT is positive from Eq. (2). This is the reason why the nucleation temperature increases from 930 K to 931 K with DC-PMF, as shown in Fig. 3. That is to say, an extra electromagnetic undercooling is generated by the increase of melting point. The small undercooling can also provide a very small driving force for nucleation.

Meanwhile, application of DC-PMF to melt will introduce forced convection and vibration. The tangential component of Lorentz force can produce convection, which is well understood. The radial component of Lorentz force can generate vibration, which can also produce convection. Because of the characteristic distribution of magnetic induction lines, the density of magnetic induction line is not uniform along the tangential direction, the radial component of Lorentz force has a gradient, which can generate intensive convection in the melt. The effects of convection intensity level on grain refinement have been well documented in Refs. [13,14]. In summary, three effects contribute to grain refinement: 1) convection tears off crystals from the mold wall, which are then transported into metal bath, where they can be converted into equiaxed crystals; 2) convection causes fragmentation of dendrites by coagulation and partial melting as a result of the temperature fluctuation effect; 3) convection lowers the thermal gradient in the melt so that the undercooled region is extended and a columnar-to-equiaxed transition is favored. This condition increases the survival probability of secondary nuclei, yielding more stable

solid particles.

During solidification under DC-PMF, the heat will transfer along the inner graphite mold wall and the pure aluminum begins to solidify from the wall. In the period of nucleation, the small nuclei are oscillated apart from the mold wall and dissociate into the molten melt under the effect of convection. However, the developed grains which can not be separated by convection will grow to columnar crystals. The schematic illustration of solidification process under DC-PMF is shown in Fig. 7.
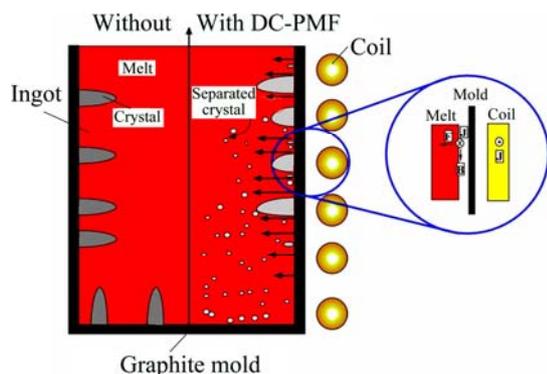

Fig. 7 Schematic diagram of refinement mechanism under DC-PMF

Nevertheless, when the induced current J passes through a mixture of solid and liquid phase, the crowding effect of current occurs due to the difference between the resistivity of two phases. In terms of pure aluminum, the resistivity of liquid melt is about 10 times as large as that of solid phase [15], resulting in more Joule heat at the solid/liquid interface. If the columnar crystals grow into the area where the temperature is higher, the tip of columnar crystal will be remelted.

However, since the intensity of convection in the melt is uneven, the grain size is not uniform under single DC-PMF. When adding small amount of inoculant Al−5Ti−1B into the melt, some particles with high melting point, i.e. TiB_2, TiB, AlB_2 and Al_3Ti, feature an active base for heterogeneous nucleation of aluminum [16]. Under the condition of DC-PMF, by adding inoculant, it is expected that more nucleation events will occur not only at the chill graphite surface but also in the bulk liquid, which will decrease the chance of columnar crystals formation. The forced convection of the melt caused by DC-PMF will make the heterogeneous nucleation sites distribute uniformly and further raise the nucleation rate by effectively extracting the heat released from recalescence. Therefore, when the convection extent is increased in an inoculated melt, more dendrite fragmentation events are expected to occur, which yields more secondary nuclei. Compared to the application of single DC-PMF, the inoculants can distribute more

homogeneously under DC-PMF, resulting in the uniform nucleation and crystal growth. Hence, the grain size under the combination of DC-PMF and inoculation is smaller and distributes more evenly than that using single DC-PMF.

5 Conclusions

1) The solidification micro structure can be greatly refined under the DC-PMF. It is clear that the grain size decreases from 7.3, 282 and 210 μm to 154 μm with the increase of the DC-PMF output current from 0, 60 and 80 A to 100 A, respectively. The refinement of pure aluminum is related to electromagnetic undercooling and forced convection caused by DC-PMF.

2) Under DC-PMF, by adding 0.05% Al−5Ti−B, the grain size is further reduced. The grain size further reduces from 177 and 151 μm to 113 μm with the variation of DC-PMF output current from 60 and 80 A to 100 A, respectively. Meanwhile the distribution of grain size is more homogeneous than that using DC-PMF only.

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直流脉冲磁场和孕育处理共同作用下纯铝的细化

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摘 要：研究直流脉冲磁场和孕育处理共同作用对纯铝凝固组织的影响，探讨直流脉冲磁场和孕育处理的共同作用的细化行为。结果表明：直流脉冲磁场能够显著细化纯铝凝固组织。纯铝的细化主要是由直流脉冲产生的电磁过冷和强制对流引起的。然而只在直流脉冲作用下，等轴晶区域的晶粒尺寸分布不均匀。在施加直流脉冲磁场的基础上加入质量分数为 0.05% 的 Al−5Ti−B，晶粒尺寸更加细小，并且整个等轴晶区域的晶粒尺寸分布比较均匀；随着输出电流的增加，平均晶粒尺寸减小，当脉冲电源的输出电流增加到 100 A 时，平均晶粒尺寸减小到 113 µm。

关键词：直流脉冲磁场；纯铝；孕育处理；晶粒细化；凝固组织

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