# Nucleation and grain formation of pure aluminum under pulsed magneto-oscillation treatment

Dong Liang <sup>a</sup>, Zhuyuan Liang <sup>a</sup>, Qijie Zhai <sup>a,\*</sup>, Gui Wang <sup>b</sup>, David H. StJohn <sup>b</sup> <sup>a</sup> Shanghai Key Laboratory of Modern Metallurgy & Materials Processing, Shanghai University, Shanghai 200072, P R China.

<sup>b</sup> Centre for Advanced Materials Processing and Manufacturing, The University of Queensland, St Lucia, Queensland 4072, Australia

**Abstract:** The grain structure of pure aluminum can be significantly refined by the Pulsed Magneto-Oscillation (PMO) treatment. When PMO was applied to the melt during the solidification, the thermal undercooling increased not only near the mould wall but also in the center of the melt and this greatly stimulated the nucleation of Al grains. PMO treatment promoted more vigorous convection of the melt, resulting in a significant decrease in the temperature gradient from the mould wall to the center of the casting compared to the case without PMO treatment. The resulted lower temperature gradient led to more uniform temperature field of the melt, which in turn favored the survival of the Al grains and allowed them to grow rather than remelt. Consideration of the thermal environment during solidification provides a more complete mechanism of grain refinement of pure metals when PMO is applied.

Keywords: pulsed magneto-oscillation; grain refinement; undercooling; solidification

## 1. Introduction

Since a refined microstructure yields uniform and improved mechanical properties, reduced casting defects and improved downstream processing abilities [1], many techniques have been used to refine the grain structure of metals, such as inoculation by adding grain refiner into the melt [2-5] and inducing localized forced convection during solidification by external forces [6-9]. Modified from pulsed electromagnetic field treatment by narrowing the pulse width and reducing the pulsed current, Gong et al. [10] proposed a promising method referred as Pulsed Magneto-Oscillation (PMO) which only induced a magnetic field in the surface regions of the melt instead of throughout the whole melt. It has been revealed that the electric magnetic force induced by PMO promote the grains nucleated on the mould wall drifting into the bulk of melt resulting in grain refinement of pure Al [10]. As a newly developed method, the interaction between thermal undercooling of the pure metals and convection stimulated by PMO remains ambiguity, and how PMO affects the likelihood of preventing nucleated grains remelting has not been well understood.

The current work aims to investigate the effects of PMO on both nucleation and grain formation of pure Al in particular to address the role of temperature distribution during solidification in promoting grain refinement under PMO by comprehensively studying the thermal undercooling and temperature gradient during the solidification along with characterizing of solidified grain structure.

<sup>\*</sup> Corresponding author. Tel./fax: +86 21 56331218.

*E-mail address*: qjzhai@shu.edu.cn (QJ Zhai).

#### 2. Experimental

530 g of pure Al (>99.7wt%) was melted and preheated inside a clay-graphite crucible with a dimension of  $\Phi$ 65mm (top) ×  $\Phi$ 30mm (bottom) × 85mm (height) by an induction furnace. When the melt reached the designated preheat temperature the induction furnace was turned off, and at the same time PMO generator was switched on so that the melt cooled and solidified under the PMO. Two K-type thermocouples were immersed into the melt 25 mm above the bottom of crucible: one was close to the inside wall of crucible and another was in the center of melt so temperature from these two thermocouples against time can be collected by a data acquisition system.

The solidified samples were sectioned along the vertical axis through the center of the casting and the samples for the grain structures observation were prepared and characterized using standard metallographic procedures. The average grain size of the equiaxed dendrites was determined by the mean linear intercept method and the percentage of the equiaxed grain area relative to the whole section area was determined by Image-Pro Plus software.

### 3. Results and Discussion

Figure 1a shows the cooling curves of the melt cooled from the preheat temperature of 1013 K with and without PMO treatment, where the dashed and solid lines correspond to cooling curves at the center and near the mould wall of the casting, respectively. A magnified region highlighting the thermal undercooling when nucleation occurs is shown in Figure 1b. It can be found that the nucleation process becomes faster as evidenced by a shorter, 4 s, and deeper, 0.5 K, undercooled zone for the PMO treated sample compared to 9 s and 0.4 K undercooling for the untreated sample near the mould wall. The undercooling in center of the melt also increases from 0.1 K to 0.5 K after PMO treatment. A similar tendency was observed when the melt was respectively preheated to 973 K and 993 K. The maximum undercooling under different conditions extracted from these experimental curves relative to the thermal plateau following recalescence is shown in Figure 1c. It is obvious that PMO treatment can increase the undercooling of the melt near the mould wall and in the center of the melt for all the three preheat temperatures. Further, the undercooling increased slightly at the center of the melt, but declined near the mould wall with increasing the preheat temperature with PMO treatment.

Figure 1d shows that the temperature gradient from the mould wall to center of the melt reduces during the melt cooling process. It is revealed that when the melt was treated by PMO the temperature gradient reduced significantly which can lead to a more uniform temperature distribution all over the bulk of melt. The decreasing temperature gradient is because the electromagnetic force generated by PMO act on the melt near the mould wall rapidly creating a high level of convection within the whole melt, as were found by other researches on electromagnetic treatment [11-12]. It also can been found from Figure 1d that with the increasing of preheat temperatures the temperature gradient reduced therefore a more uniform temperature field can be established under PMO treatment, for example, for the melt cooled from 1013 K where the temperature gradient

reduced by more than a half to 0.21 K/cm near the onset of nucleation. It is believed that a higher preheating temperature, longer PMO treatment times in the liquid state can be achieve, consequently the temperature field distributed more uniformly.



**Figure 1**. (a) Cooling curves of melts cooled from preheat temperature of 1013 K with and without PMO treatment. (b) Magnification of the undercooled region of the cooling curves presented in Figure 1a. (c) The values of undercooling of the melt near the mould wall and at the center of the melt with and without PMO treatment, plotted against preheat temperatures of 973 K, 993 K and 1013 K. (d) The temperature gradients of melts cooled from the above three kinds of preheat temperatures with and without PMO treatment versus temperature.

Figures 2a-c show the solidified structures of pure Al cooled from 973 K, 993 K and 1013 K without PMO treatment while Figures 2d-f are the solidified structures with PMO treatment cooled from the same temperatures. The samples without PMO treatment have almost the same grain structure, i.e. typical coarse columnar crystals. On the other hand, the grain structures of the samples cooled from the same preheat temperatures with PMO treatment exhibit a fine equiaxed region at the bottom central. These solidified structures with and without PMO treatment correspond well to the cooling curves shown in Figures 1a-b indicating that PMO treatment stimulates the nucleation process, significantly increasing the rate of nucleation.



**Figure 2**. Solidification structures with and without PMO treatment. (a-c) Cooled from 973 K, 993 K and 1013 K without PMO treatment, (d-f) Cooled from the same temperatures with PMO treatment.

It should be noted that undercooling near the crucible wall decreased with an increase in the preheat temperature under PMO treatment as indicated in Figure 1c, and it would be expected that the nucleation rate should also follow the same trend, i.e. the grain structure should be most refined when cooled from 973 K. However, unexpectedly the grain size decreased and the area percentage of equiaxed grains increased with higher preheat temperature as shown in Figure 3. This is due to the longer time of PMO treatment on the liquid phase when the preheat temperature is higher, which allows more convection of the melt resulting in a lower temperature gradient as discussed above. The lowest temperature gradient of 0.21 K/cm from the mould wall to the center of the casting means that it is more likely for the whole melt to cool to an undercooled condition, promoting the survival and growth of grains. Zhao et al. [12] also observed that when the temperature is most uniform after PMF treatment pure Al had the finest grain structure.



Figure 3. Average grain size and area percentage of equiaxed grains corresponding to

Figures 2d-f plotted against preheat temperature.

The uniformity of the temperature field from the mould wall to the center of the casting is essential as it controls the number of newly nucleated grains that survive from remelting, and the more uniform the temperature distribution of the melt is, the finer and more grains can be achieved under PMO treatment. The success of the PMO treatment, and probably other types of external fields, in grain refining pure metals is a balance between the PMO application time (sufficient convection), preheat temperature (increased cooling rate) and the formation of a low temperature gradient (providing uniform undercooling throughout the melt) which together promote nucleation and the survival and further growth of grains during continued solidification.

### Conclusions

By inducing a pulsed magnetic field in the surface regions of the pure aluminum melt during the PMO treatment, thermal undercooling of the melt can be increased, and the temperature gradient from the mould wall to the center of the casting decreased due to melt convection induced by PMO treatment as well. Increasing thermal undercooling largely promotes nucleation of pure Al grains while decreasing the temperature gradient improves nucleated grains survivability from remelting and allows these grains to grow. Therefore significant fine grain structure can be achieved by PMO treatment.

#### Acknowledgments

The authors gratefully acknowledge financial support from the Foundation of the National Basic Research Program (973 Program, Grant No. 2011CB012902) and the National Natural Science Foundation of China (Grant No. 51320105003). DStJ and GW acknowledge support from the Australian Research Council Centre of Excellence for Design in Light Metals, ARC DP140100702 and the ExoMet Project co-funded by the European Commission's 7th Framework Programme (contract FP7-NMP3-LA-2012-280421), by the European Space Agency and by the individual partner organizations.

#### References

- [1] Mohanty PS, Gruzleski JE. Acta Metall Mater 1995;43:2001-12.
- [2] StJohn DH, Ma Q, Easton MA, Cao P. Acta Materialia 2011;59:4907-21.
- [3] StJohn DH, Frost MT. Scripta Mater 2004; 50:1115-9.
- [4] StJohn DH, Ma Q, Easton MA, Peng C, Zoe H. Metall Mater Trans A 2005;36:1669-79.
- [5] Easton MA, StJohn DH. Metall Mater Trans A 2005;36:1911-20.
- [6] Li GR, Wang HM, Yuan XT, Zhao YT. Mater Lett 2013;99:50-3.
- [7] Zhang L, Li W, Yao JP, Qiu H. Mater Lett 2012;66:190-2.
- [8] Liao XL, Zhai QJ, Luo J, Chen WJ, Gong YY. Acta Mater 2007;55:3103-9.
- [9] Gao YL, Li QS, Gong YY, Zhai QJ. Mater Lett 2007;61:4011-4.
- [10]Gong YY, Luo J, Jing JX. Mater Sci Eng A 2008;497:147-52.
- [11] Wang B, Yang YS, Ma XP. Trans. Nonferrous Met Soc China 2010;20:1685-90.

[12]Zhao ZL, Liu Y, Liu L, Mater Manuf Process 2011;26:1202-06.