



# The grain refining mechanism of cast zinc through silver inoculation

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## Abstract

Silver (Ag) inoculation was found to significantly reduce the average grain size of cast zinc (Zn) by up to 90%. The mechanism of such grain refinement in cast Zn was investigated through varying the addition level of this peritectic-forming solute. The reduction in grain size was sensitive to Ag content due to its large growth restriction factor. When the Ag content was over its maximum solid solubility in Zn, the in situ formed nucleation particles, with dendritic morphology (different from the previously reported faceted/spherical shape), were reproducibly observed near the grain centres in the refined alloys. These particles were determined to be pro-peritectic  $\text{AgZn}_3$  phase based on the information from the Zn–Ag phase diagram, results from thermal analysis, phase identification and chemical composition. The high potency of the in situ formed  $\text{AgZn}_3$  particles, as nucleation sites for Zn grains, was further verified by electron backscattered diffraction analysis and crystallographic calculation using the edge-to-edge matching model. A new reproducible hexagonal close packed–hexagonal close packed orientation relationship between  $\text{AgZn}_3$  particles and Zn matrix was experimentally determined for the first time. In addition, the effect of the particle size and size distribution on the microstructural refinement was also investigated. Finally, the grain refinement mechanism was elucidated in terms of the nucleation crystallography and the interdependence theory. © 2014 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

**Keywords:** Grain refinement; Nucleation crystallography; Growth restriction; EBSD; Zn alloys

## 1. Introduction

During casting of metals and alloys, inoculation is generally practised as an important technique to promote microstructural refinement, i.e. reduction in grain size and/or transition of columnar structure to equiaxed grains [1], and to facilitate the downstream mechanical working and shape-forming [2–4]. For more than six decades, the mechanism involved in grain refinement of cast metals through inoculation has been widely studied in light metals, i.e. Al [3,5,6], Mg [7,8] and Ti alloys [9]. Theories/models and reliable grain refiners have been relatively well-developed for Al, Mg and their alloys. However, it is still unknown whether these theories/models developed

in light metals can be directly applied to other metals. Zinc and its alloys are normally utilized as-cast in transportation, electronic and construction industries [10]. They are the favoured engineering materials for many fields due to their typical advantages, i.e. the low melting temperature, precise casting dimensional control, good corrosion resistance and high recyclability [11]. However, cast zinc always exhibits coarse grains, associated with low strength, low ductility and brittle fracture during service [12,13], which limits its applications. It has been predicted that well-refined zinc alloys will have potential to serve as structural and pressure-tight components [12]. Thus, studying the grain refinement of cast Zn is of theoretical and engineering significance. Unfortunately, the progress in grain refinement of cast Zn has been far slower than that for light metals, because there are few efficient grain refiners reported for cast Zn, and the relevant refining mechanism also remains unclear.

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Peritectic solidification plays a significant role in the manufacture of many cast products [14], steel tools [15], superconductors [16] and magnetic materials [17,18]. It has long been recognized that peritectic-forming solute elements contribute to grain refinement during casting or welding [4,8,19]. In situ formed pro-peritectic intermetallic compounds can act as nucleants for the peritectic phase, and these enhanced nucleation events can further promote grain refinement. However, the peritectic theory of grain refinement has been considered as invalid, because the addition of Ti into cast Al at the levels below maximum solubility, at which it was supposed that no peritectic reaction occurs, can also lead to grain refinement. Recently, Wang et al. [4,20] revisited the role of peritectic in grain refinement of Al alloys, which verified the significance of the addition of peritectic-forming solutes in refining the as-cast grains. In our early work [10], two peritectic-forming elements, Ag and Cu, were added to pure Zn and remarkable grain refining efficiency was observed, although Ag is even better. But there are still a number of questions to be answered regarding the role of Ag: (1) What is the predominant grain refinement mechanism at different addition levels, i.e. below or over the maximum solubility ( $C_m$ )? (2) Does any heterogeneous nucleation occur? (3) If so, is it the pro-peritectic particle/phase that acts as the nucleation site? (4) How do the nucleation crystallography and particle features contribute to the grain refinement? To answer these questions, it is essential to comprehensively investigate the grain refinement behaviour/mechanism resulting from the addition of peritectic-forming elements. In this paper, the grain refinement of Ag-inoculated cast Zn is studied. The role of Cu will be addressed in a subsequent paper.

Nucleation crystallography, involved with the interfacial structure, atomic matching and interface orientation between nucleant and nuclei, affects grain refinement efficiency through changing the potency of nucleants (or particles) [21,22]. However, the actual factors that govern the grain refining potency of the nucleants are yet to be understood. Commonly, the nucleating potency of particles can be evaluated using the crystallographic atomic matching (CAM) across the particle/matrix interface [21,23–26]. High CAM reduces the critical undercooling for heterogeneous nucleation and further increase the nucleation rate. A couple of crystallographic geometric models [25,27–29] were developed to quantify the CAM. One such model, the edge-to-edge matching (E2EM) model is used in the current work to analyse the nucleation crystallography between the nucleant and the matrix. Over the past decade, the E2EM model [28–31] has already been validated to be powerful in explaining grain-refining efficacy and predicting new grain refiners for Al, Mg and their alloys [20,21,23,32–38].

The work of Wang and co-workers on binary Al–Zr/Nb alloys [4,20] showed that the peritectic-forming Zr (or Nb) element only generates marginal grain refinement in cast Al when the addition level is below  $C_m$ . This is different from

the experimental results reported in the binary Zn–Ag/Cu peritectic alloys [10], where significant grain refinement was achieved even at the addition level below  $C_m$ . Actually, extensive studies on other Al, Mg, Ti and Fe cast alloys [39–42] also showed that some specific solutes also make a great contribution to the grain refinement. Thus, two questions arise: (1) What properties should a specific solute have to effectively grain refine cast metals? (2) What is the role of the specific solute in grain refinement at different addition levels? Currently, the effects of solute on grain refinement are mainly characterized using a phase-diagram-based parameter, the growth restriction factor ( $Q$ ) [10,43–45]. A solute with a higher  $Q$  value partitions strongly ahead of the advancing solid/liquid (S/L) interface, which induces a rapid build-up of constitutional supercooling (CS). The formed CS zone can provide an additional driving force to facilitate nucleation and/or the rejected solute at the S/L interface will restrict growth of the “previously formed” solid by affecting the S/L interfacial behaviour [41,46,47]. The present work focuses on investigating the predominant roles of peritectic-forming Ag in the grain refinement of cast Zn at different addition levels from three perspectives, including thermal analysis, the nucleation crystallography and the interdependent effects from specific solute and potent particle.

## 2. Experimental

### 2.1. Casting trials

In order to investigate the effect of Ag on the grain refinement of cast pure Zn, various amounts of Ag were added into pure Zn in the form of master alloys. The master alloy (Zn–10 wt.% Ag) was made using super-high-purity Zn ingots (99.995% pure), which was melted in a clay-bonded graphite crucible using an induction furnace, followed by adding compacted Zn–Ag pellets. The pellets consisted of Ag powder (99.9% pure) and Zn chips. The whole melting process of the Zn–Ag master alloy was carried out in a protective atmosphere of argon. In addition, the inner surface of all crucibles was completely coated with boron nitride in order to prevent foreign impurities. Eight binary alloys nominally containing 0.5%, 1.0%, 1.5%, 2.0%, 3.0%, 3.5%, 4.5% and 6 wt.% Ag were prepared, respectively. All solute contents in this paper are expressed in wt.% unless specified otherwise. The melts were isothermally held for 20 min at 600 °C in the furnace. After skimming off the dross and stirring, the melts were then cast into a 30 mm in diameter and 40 mm long cylindrical graphite mould that was preheated to the same temperature as the melts. Additionally, the cooling rates of ingots were measured using N-type thermocouples. To determine the difference between nominal and actual Ag concentrations, quantitative chemical analysis was carried out using inductively coupled plasma atomic emission spectroscopy (ICP-AES). The determined chemical compositions are listed in Table 1.

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