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Discussions on grain refinement of magnesium alloys by carbon inoculation

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Abstract

Carbon inoculation has no effect on magnesium alloys that do not contain aluminium. The hypothesis proposed in a recent article [*Scripta Materialia* 49 (2003) 1129] that segregation of carbon plays a major role in the grain refinement of magnesium alloys by carbon inoculation is inconsistent with many of the observed facts. The Al_4C_3 or Al–C–O hypothesis, which is supported by experimental observations, is still the most reasonable mechanism proposed to date for the grain refinement of magnesium alloys by carbon inoculation.

Keywords: Magnesium alloys; Grain refinement; Carbon inoculation; Grain Refining; Al_4C_3; Al–C–O

1. Introduction

In a recent article [1], an investigation was made into the mechanism of grain refinement of Mg–Al type alloys by carbon inoculation. The authors attributed the resultant grain refinement to the segregation of carbon that occurred during solidification rather than the commonly accepted Al_4C_3 hypothesis. While this is an interesting explanation, we believe that these authors have ignored a number of key issues in the grain refinement of magnesium alloys by carbon inoculation. This article discusses these issues and shows that the evidence presented in Ref. [1] for the segregation of carbon playing a major role is insufficient, and that the hypothesis is inconsistent with many of the observed facts. The Al_4C_3 hypothesis or a modified version of it, i.e. the Al–C–O hypothesis [2–6], is still the most reasonable mechanism proposed for the grain refinement of magnesium alloys by carbon inoculation.

2. Experimental procedure

To better understand the grain refining process of magnesium alloys by carbon inoculation, grain refining tests of pure Mg, Mg–3%Zn and Mg–3%Al alloys were performed on the 1 kg scale of melt using a commercially available carbon grain refiner, Nucleant 5000 (Foseco GmbH, Germany). Sublimed magnesium (99.98%), high purity aluminium (99.99%) and commercial purity zinc (99.95%) were used as raw materials. Melting was conducted in an electrical resistance furnace at 750 °C under a protective cover gas (1.0% SF_6 + 49% dry air + 50% CO_2) using a boron nitride coated mild steel crucible. The same amount of carbon addition (0.6 wt%) used in Ref. [1] was employed in each test. Cone samples were taken from the top of the melt using a boron nitride coated cone ladle (ϕ 20 mm × ϕ 30 mm × 25 mm). The average grain size in the central region of each cone

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sample was measured according to the linear intercept method described in the ASTM standard E112-96.

3. Results and discussion

3.1. Emley's views on the Al_4C_3 hypothesis

In his Principles of Magnesium Technology Emley [7] discussed in detail the various mechanisms proposed for the grain refinement of Mg–Al type alloys by carbon inoculation and made the following concluding remarks on page 208,

Returning to the Al_4C_3 hypothesis, this can explain many of the observed facts ... *Quite recently Schneider and Hilmer[40] have for the first time identified Al_4C_3 nuclei in melts grain-refined with carbon*, and predicted on thermodynamic grounds that this compound can only be formed in melts containing above about 1 pct aluminium—a result in good agreement with grain refining experience. Thus, although perhaps all the relevant facts are not yet satisfactorily explained, *it is clear that Al_4C_3 nuclei play a major role in the grain refinement of Mg–Al type alloys* ... (stress added).

Clearly, the evidence for the Al_4C_3 hypothesis was, on the whole, satisfactory to Emley. The authors of Ref. [1] referred to Emley [7] to support their criticism on the Al_4C_3 hypothesis. It appears that they have missed these concluding remarks made by Emley.

Considering that Emley's *Principles of Magnesium Technology* [7] is a major reference book for magnesium researchers, we would like to take this opportunity to point out that Emley seemed to have used a wrong reference in making the above remarks. Having checked the work of Schneider and Hilmer referred to by Emley, i.e. "Zur thermischen Reduktion der Tonerde", Metall. 1960, vol.14, 186–195, we found that the reference actually has little to do with the grain refinement of magnesium alloys by carbon inoculation. Instead, it is about the formation of Al_4C_3 via thermal reduction of alumina by carbon. So, based on this fact, it is probably still questionable whether Schneider and Hilmer have really identified Al_4C_3 nuclei in magnesium melts grain-refined with carbon or not.

3.2. Dependence of carbon inoculation on aluminium content in magnesium alloys

It has long been recognised that carbon inoculation is effective only to magnesium alloys that contain aluminium [8,9]. In the literature, Mg–Al type alloys that can be effectively grain-refined by carbon inoculation normally contain more than 2% Al [1–6,8,9]. This dependence on aluminium content provides a strong support to the Al_4C_3 hypothesis.

Fig. 1(a) and (b) show the results obtained from the grain refining test of pure magnesium with 0.6% of carbon addition. As expected, no grain refinement was observed. Similar observations were made from the grain refining test of Mg–3%Zn with the same amount of carbon addition (0.6%), as shown in Fig. 2(a) and (b). In contrast, obvious grain refinement was observed when 0.6% of carbon was introduced to the melt of a Mg–3%Al alloy under the same conditions, as shown in Fig. 3(a) and (b). These observations are in good agreement with the current understanding of the grain refinement of magnesium alloys by carbon inoculation.

Table 1 summarises the results obtained by different researchers on grain refinement of magnesium alloys by carbon inoculation. As can be seen, carbon inoculation is effective only to magnesium alloys that contain aluminium. Carbon is not a grain refiner for magnesium alloys that do not contain aluminium. In other words, grain refinement of magnesium alloys by carbon inoculation stems from the interaction between carbon and aluminium. This is a key feature of grain refinement of magnesium alloys by carbon inoculation. The role of segregation of carbon, if the segregation occurs during solidification, seems to be well negligible in the grain refining process. Otherwise grain refinement should have been observed in pure magnesium or Mg–3%Zn and Mg–1.5%Mn alloys when treated with carbon. Further discussion will be made of the segregation of carbon in magnesium alloys subsequently.

3.3. Recent work on Al–C–O nuclei in Mg–9%Al type alloys

Some detailed investigation into the mechanism of grain refinement of Mg–9%Al alloys by carbon inoculation has recently been reported [2–6]. Distilled high purity magnesium (>99.99%), high purity aluminium

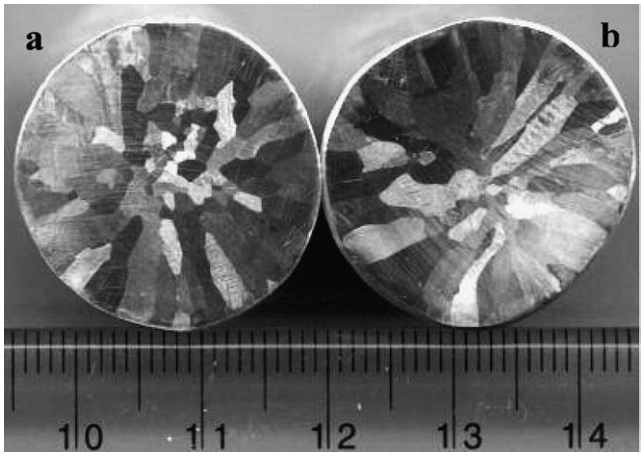

Fig. 1. Grain refinement of pure Mg by carbon inoculation: (a) base metal and (b) after 0.6% of carbon addition in the form of Nucleant 5000 at 750 °C. No grain refinement was observed.