2013
Magnesium Technology
EDITED BY
Norbert Hort
Suveen N. Mathaudhu
Neale R. Neelameggham
Martyn Alderman
Magnesium Technology 2013
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Magnesium Technology 2013

Proceedings of a symposium sponsored by the Magnesium Committee of the Light Metals Division of The Minerals, Metals & Materials Society (TMS)

Held during the TMS 2013 Annual Meeting & Exhibition
San Antonio, Texas, USA
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Edited by
Norbert Hort
Suveen N. Mathaudhu
Neale R. Neclameggham
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Magnesium was produced first in 1808, more than 200 years ago, by Sir Humphry Davy. It took a few decades to develop and establish stable production processes, and in the middle of the 19th century magnesium was available in larger quantities and ready to be used. In fact it met a new technology at these times: photography. Magnesium was utilized in flashlights and this endured until the end of the 20th century. Of course, flashlights are not a structural application. But with time, in the first years of the 20th century again a step in the technical development took place: the Wright brothers started to fly. It was found out early that wooden airplanes would not do their job at all. Light metal alloys had to be used, and magnesium alloys were part of aeronautics from the beginning. In fact an engine for aircrafts made out of a magnesium alloy was shown 1909 in Frankfurt, Germany, at the first „Internationale Luftschiffahrt Ausstellung,” the first international aerospace exhibition. While magnesium was important for aerospace 100 years ago, today it is still looking for acceptance in aerospace industries.

In difference to the slight acceptance in aerospace, magnesium alloys are fairly well accepted in automotive industries as well as for consumer, computer, and communication applications. Due to its excellent specific properties magnesium alloys can compete especially in transportation with aluminium and even with steel leading to tremendous weight savings. This directly leads either to an increase in payload or a fuel reduction (saves money for the end user), or reduces green house gas emissions. Today automotive industries are showing lots of examples for the use of magnesium alloys in various applications, but an average standard car only contains a few kilograms of magnesium alloys. To enhance the use of magnesium even in this field it is still necessary to develop alloys and to optimize processes.

The development of processes for primary production, alloy development and the processing routes started in the early 20th century and continued over the years. Several authors compiled the state of the art. Among them are J. L. Haughton and W. E. Prytherch (1938), A. Beck (1939, reprinted 2001), L. M. Pidgeon, et al. (1946), W. H. Gross (1949), G. V. Raynor (1959), C. S. Roberts (1960), E. F. Emley (1966), R. S. Busk (1986), and in recent years M. A. Avedesian and H. Baker (1999), C. Kammer (2000), K. U. Kainer (2003), and H. E. Friedrich and B. L. Mordike (2004). In all of these books the state of the art is shown and the progress in magnesium alloy and technology development is described. But these books only present a view into the past.

The first Magnesium Technology symposium at the TMS meetings took place at the TMS Annual Meeting in Nashville, Tennessee, 2000. Since this time the symposium is showing the lively research of the active magnesium community in the world and it presents also a look into the future. This year is the 14th edition of this meeting, and it will be held in San Antonio, Texas, from March 3-7, 2013. The symposium is arranged into an opening plenary session and a number of technical sessions which address the full scope of Magnesium research and development. These sessions include primary production, casting and solidification, deformation mechanisms and texture, alloy and microstructural design, corrosion, high temperature behaviour, processing-microstructure-property relationships, and advanced processing and joining.
Last, the organizing of these proceedings would not have been possible without the dedication and assistance of TMS staff. In particular, we would like to recognize Ms. Patricia Warren for her tireless patience and hard work.

Over the past 13 years, the Magnesium Technology proceedings, through its high quality technical content, has become the primary proceedings for the magnesium industry and technical community. We hope that this year's volume will serve you just as well.

Norbert Hort (Helmholtz-Zentrum Geesthacht, Germany)
Suveen N. Mathaudhu (U.S. Army Research Office, USA)
Neale R. Neelameggham (IND LLC, USA)
Martyn Alderman (Magnesium Elektron, UK)
Norbert Hort
MAGNESIUM TECHNOLOGY 2013 LEAD EDITOR

Norbert Hort is the head of the Magnesium Processing Department at the Magnesium Innovation Centre (MagIC) within the Helmholtz-Zentrum Geesthacht Zentrum für Material- und Küstenforschung, Geesthacht, Germany (formerly the GKSS Research Centre). Concurrently he is lecturer at the Leuphana University, Lüneburg, Germany. He studied Materials Sciences at the Clausthal University of Technology (CUT), Germany, where he has been involved in magnesium research since the early 1990s. His diploma thesis (1994) dealt with gas-atomized, hot-extruded magnesium alloys. During 1994-95 he worked as a researcher at the Institute of Materials Sciences (CUT) and he joined the HZG in 2000. He got his Ph.D. degree in Materials Sciences in 2002 from the Clausthal University of Technology. In the MagIC he is responsible for the development of new creep resistant magnesium alloys, grain refinement and the castability of magnesium alloys (viscosity and density of melts, fluidity, and mould filling). A major focus of his research is the development of new magnesium alloys for biodegradable implants in a close collaboration with biologists and clinicians. He is co-author of more than 150 peer reviewed journal papers and contributions to conference proceedings. In recent years he was involved in the organizing committees of the conference series “Magnesium Alloys and their Applications” and of the conference “Light Metal Technologies 2011.” - Since 2009 he also has been the chairman of the technical committee “Magnesium” of the German Society of Materials (DGM).
ABOUT THE EDITORS

Suveen Nigel Mathaudhu serves as the Program Manager for Synthesis and Processing of Materials with the U.S. Army Research Office (ARO), Materials Science Division. He received his B.S.E. in Mechanical Engineering from Walla Walla University in 1998, and Ph.D. in Mechanical Engineering from Texas A&M University in 2006. In his current position, he manages programs which focus on the use of innovative approaches for processing high performance structural materials reliably and at lower costs. He also concurrently serves as an Adjunct Assistant Professor in the Department of Materials Science and Engineering at North Carolina State University, where his research interests include ultrafine-grained and nanostructured materials by severe plastic deformation, consolidation of metastable particulate materials and processing-microstructure-property relationships of refractory metals and lightweight metals, and thermally stable nanocrystalline materials. He has co-authored over 60 technical publications in these areas.

Neale R. Neelameggham is ‘Guru’ at IND LLC, involved in technology marketing and consulting in the field of light metals and associated chemicals, [boron, magnesium, titanium, lithium and alkali metals], rare earth elements, battery and energy technologies, etc. He has over 38 years of expertise in magnesium production technology from the Great Salt Lake brine in Utah. He was involved in Process Development of its startup company, NL Magnesium, through the presently known US Magnesium LLC. and was its Technical Development Scientist from where he retired. Dr. Neelameggham’s expertise includes all aspects of the magnesium process, from solar ponds through the cast house including solvent extraction, spray drying, molten salt chlorination, electrolytic cell and furnace designs, lithium ion battery chemicals and by-product chemical processing. In addition, he has an in-depth and detailed knowledge of alloy development as well as all competing technologies of magnesium production, both electrolytic and thermal processes worldwide. Dr. Neelameggham holds 13 patents and a pending patent on boron production, and has several technical papers to his credit.

As a member of TMS, AIChE, and a former member of American Ceramics Society, he is well-versed in energy engineering, bio-fuels, rare-earth minerals and metal processing, and related processes. Dr. Neelameggham has served in the Magnesium Committee of LMD since its inception in 2000, chaired it in 2005, and has been a co-organizer of the Magnesium symposium since 2004. In 2007 he was made a permanent co-organizer for the Magnesium symposium. He has been a member of the Reactive Metals Committee and Recycling Committee, and a Programming Committee representative of LMD. In 2008, LMD and EPD created the Energy Committee following the symposium on CO$_2$ Reduction Metallurgy symposium initiated by him. Dr. Neelameggham
was selected as the inaugural Chair for the Energy Committee with a two-year term. He received the LMD Distinguished Service Award in 2010 and has been a co-editor of the Energy Technology symposium proceedings volume. Dr. Neelameggham holds a doctorate in extractive metallurgy from the University of Utah.

Martyn Alderman is Divisional Director of Technology for the Magnesium Elektron Group Worldwide. He obtained a Masters Degree in Material Science from the University of Cambridge (United Kingdom) in 1979 and for the next 20 years he worked in aluminium rolling mills producing and developing Al-Cu-Zr, Al-Mn, Al Mg-Mn, Al-Zn-Mg, and Al-Li-Cu alloys for Superplastic forming. He also has significant operational experience direct chill casting and in the extrusion of high strength aluminium alloys.

In 2003 he joined Magnesium Elektron during the acquisition of their large Madison, Illinois rolling operation in the United States. For the next few years he was travelling between the United States and Europe encouraging the wider use of magnesium sheet in transport applications, in particular by use of superplastic forming, and later in managing the post acquisition integration of the Revere North American Graphic Arts business. He chaired the 2010 International Magnesium Association Conference in Hong Kong, and for the past two years had been involved in editing and producing a 270 page Handbook on Designing with Magnesium Alloys.

As a member of TMS, SAE, and AMS, he is a regular attendee at worldwide magnesium conferences keeping abreast of global technology development with the aim of maintaining the Magnesium Elektron’s position as a world leader in magnesium alloys and their end use applications.
MAGNESIUM TECHNOLOGY 2013
Session Chairs

Plenary
Norbert Hort
Helmholtz-Zentrum Geesthacht

Primary Production and Casting
Neale Neelameggham
Ind LLC

Adam Powell
Metal Oxygen Separation Technologies

Mechanical Properties
Alan Luo
General Motors Global Research & Development

Menahem Bamberger
Technion

Corrosion
Liming Peng
Shanghai Jiaotong University

Carlos Caceres
The University of Queensland

Texture and Wrought Materials
Martyn Alderman
Magnesium Elektron

Alok Singh
National Institute for Materials Science

Wrought Materials II
Julian Rosalie
National Institute for Materials Science

Jennifer Hay
Agilent Technologies

Grain Refinement, Twinning, and Composites
Karl Kainer
Helmholtz-Zentrum Geesthacht

Suveen Mathaudhu
U.S. Army Research Office

Phase Formation
Alok Singh
National Institute for Materials Science

Alan Luo
General Motors Global Research & Development

Poster Session
Eric Nyberg
Pacific Northwest National Laboratory
A BRIEF HISTORY OF THE DEVELOPMENT OF GRAIN REFINEMENT TECHNOLOGY FOR CAST MAGNESIUM ALLOYS

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2Centre for Advanced Materials Processing and Manufacturing (AMPAM)
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Keywords: Magnesium, Grain refinement, Solidification

Abstract

Grain size control during casting is important for ensuring the best and most consistent mechanical and corrosion performance is achieved in magnesium alloy components. Also, it is important for the fabrication of wrought Mg products with desired surface quality. Industrially, this means employing grain refinement methods. The important role of grain refinement was realized in the 1930s. However, developing a good understanding of the solidification mechanisms for improved refinement technologies involved in successful refinement has been the subject of ongoing research ever since. This paper provides a brief history of developments both scientifically and industrially, and summarizes current issues related to the development of improved grain refiners.

Introduction

Grain refinement is essentially a nucleation process. The origins of our understanding about nucleation are well summarized by Kelton and Greer in their book ‘Nucleation in Condensed Matter’[1]. As understanding of the deformation behavior of alloys improved, relationships between grain size and properties were developed such as the Hall-Petch relationship developed in the 1950s which predicts the effect of grain size on the yield strength of alloys, including Mg-Al alloys [2]. Thus the casting process itself became a focus for improving the properties of components and this was primarily achieved by stimulating the nucleation process. There are parallels between the developments of grain refining technologies for Al [3, 4] and Mg [5, 6] alloys. Both developments were first reported in the 1930s. The Al-5Ti-1B master alloy developed in the early 1970s works well for most Al alloys [3, 4]. However, for Mg alloys there is no equivalent that provides reliable, repeatable as-cast grain sizes in a cost effective manner. Since then much research effort has been focused on understanding the mechanisms of grain refinement.

Mg alloys are cast by a large variety of casting processes including ingot, sand, gravity die, low pressure die and high pressure die (HPDC) casting methods. Most Mg components produced by HPDC are Mg-Al alloys. However, complex castings for applications such as gearbox housings use precision sand casting as well as HPDC. Alloy ingot and billet are commonly produced by direct chill casting and gravity die casting. Grain refinement technology is not needed for HPDC but is important for other casting processes and can help improve creep resistance for certain Mg alloys used at elevated temperatures.

Given the limited length of a conference paper we will cover highlights rather than attempt to provide a detailed history. However, we hope that there is sufficient information provided so that the reader can gain an appreciation of the pathways followed by founders and researchers in an attempt to develop effective and reliable grain refining technologies. This paper firstly presents a summary of the range of grain refining technologies and associated mechanisms developed to explain refinement. A model that attempts to predict grain size outcomes is then described. Finally, current issues that remain unresolved are presented.

Grain Refinement Technologies

Tables 1 and 2 summarize the key advantages and disadvantages of the developed grain refinement technologies with reference to relevant patents while Table 2 summarizes the grain refinement mechanisms proposed to explain what controls grain refinement. Table 1 is subdivided into Mg-Al alloys, Mg-Zn and RE alloys that do not contain Al, and methods applicable to all alloys.

The development of refinement technologies has more or less followed assumptions derived from observation of casting outcomes. The following puts these developments in the context of four elements, Fe, Mn, C and Zr, that underpin the assumptions used to develop particular refinement methods.

The idea of grain refinement by Fe stems from early work on superheating [7]. Superheating is an important early grain refining method developed for Mg alloys containing Al, and this process was widely used prior to and during World War II in the commercial production of Mg-Al alloys [8]. The process involves heating a Mg alloy to a temperature about 180-300 °C above its liquidus, holding it for a required period, and then cooling quickly to the pouring temperature [7]. It was believed that superheating was caused by Fe picked up from the crucible. The idea that Fe particles acted as nuclei for Mg grains resulted in the invention of an Fe inoculation process called the Elfinal process or FeCl₃ process [9]. The Elfinal process consists of plunging 0.4-1.0% of anhydrous FeCl₃ powder at temperatures between 740 °C and 780 °C into a molten Mg alloy [10]. The first application of the Elfinal process was on Mg-Al-Zn alloys [10] and later Al-free Mg-Zn alloys. Jessup and Petch [11, 12] of Magnesium Elektron Limited (MEL) reported that grain refinement of Mg-Zn-RE-Mn alloys was achievable using a Zn-7%Fe pre-alloy as an Fe additive replacing FeCl₃. Nelson pointed out that the Elfinal process didn’t work for Mg-Al alloys without Mn [8].
In 1945 Tiner [13] studied the superheating of Mg alloys and the influence of Fe and Mn on the response of superheating. He found that Fe favours grain refinement by superheating. Partridge [14], in 1948, observed obvious grain refinement of Mg-Al alloys using a new but slightly rusty mild steel crucible. He concluded that the loosely adherent rusty Fe had produced the observed grain-refining effect. Emley [5], however, suggested that the observed grain refinement was due to the uptake of carbon from the crucible, as the surface layer of a new mild steel crucible contains more carbon than does the surface layer of a repeatedly used crucible. Regarding the Elfinal process, Emley suggested that Fe containing intermetallic particles or Al carbide (Al$_4$C$_3$) particles were possibly the nucleants [5]. Emley apparently preferred the Al$_4$C$_3$ hypothesis, as he believed that the hydrolysis of FeCl$_4$ gave rise to copious HCl fumes, which could attack steel crucibles to liberate carbon from the surface layers [5].

Table 1. A summary of the approaches taken to grain refine Mg alloys with the advantages and disadvantages of each approach.

<table>
<thead>
<tr>
<th>Process</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solute additions</td>
<td>Solute additions can lead to significant grain size reduction even without the addition of a nucleant.</td>
<td>There is a limit to the grain refinement that can be achieved and it is not effective in casting alloys which already have high solute contents. Alloy composition changes and mechanical properties may not be desirable.</td>
</tr>
<tr>
<td>Superheating</td>
<td>Significant grain refinement can be achieved.</td>
<td>Difficult to control. High temperatures (~850°C) must be used that can cause melt handling problems. Not clear what the nucleant particles are.</td>
</tr>
<tr>
<td>Elfinal</td>
<td>Produces a degree of refinement.</td>
<td>Adds Fe which decreases corrosion resistance. Chlorine is produced which has health implications.</td>
</tr>
<tr>
<td>C inoculation</td>
<td>Significant grain refinement can be achieved.</td>
<td>The only approach used commercially at this stage. However, the most effective way of adding C is under debate.</td>
</tr>
<tr>
<td>Native grain refinement</td>
<td>The native particles present in high purity melts are more potent than those present in commercial alloys.</td>
<td>No direct evidence of particles and their composition. Pure alloys are more expensive. Limited to Mg-Al based alloys.</td>
</tr>
<tr>
<td>SiC</td>
<td>Works reasonably well.</td>
<td>Grain refinement is not as effective as desired. Also, SiC is unstable in the melt and appears to be another C-inoculation technique.</td>
</tr>
<tr>
<td>Fe, Mn, Ti additions</td>
<td>Reported that some grain refinement achieved. However, poisoning occurs when Fe and Mn are both present.</td>
<td>Results vary suggesting that the reason for the grain refinement cannot be controlled. It is not understood completely why they work.</td>
</tr>
<tr>
<td>Borides</td>
<td>These are known nucleants in Al-based alloys and are available as master alloys. Reports of good grain refinement.</td>
<td>Borides have been tried in CAST and other work and have been shown to have inconsistent results. Settling of particles is an issue for commercial application.</td>
</tr>
<tr>
<td>AlN, ZnO</td>
<td>Crystallographic edge-to-edge matching (ε2em) predicts AlN and ZnO are possible nucleants.</td>
<td>ZnO is unstable in melts and a previous attempt at using AlN particles did not perform as well as expected.</td>
</tr>
<tr>
<td>High pressure die casting</td>
<td>High cooling rate driven grain refinement without any change of alloy chemistry.</td>
<td>Not suited for the production of basic shapes (billet and slab); having dendritic grains in most cases.</td>
</tr>
</tbody>
</table>

**Mg – Al alloys**

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<tr>
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<td>Grain refinement is not as effective as desired. Also, SiC is unstable in the melt and appears to be another C-inoculation technique.</td>
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<tr>
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<td>High pressure die casting</td>
<td>High cooling rate driven grain refinement without any change of alloy chemistry.</td>
<td>Not suited for the production of basic shapes (billet and slab); having dendritic grains in most cases.</td>
</tr>
</tbody>
</table>

**Mg – Zn, RE alloys that do not contain Al, Mn, Si, Fe**

<table>
<thead>
<tr>
<th>Process</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg-Zr master alloys</td>
<td>Very effective, producing grain sizes down to &lt; 50 microns.</td>
<td>Expensive, high degree of waste Zr in sludge that forms from settling of Zr particles. Reaction with Fe-based crucibles can also consume Zr. Large agglomerates affect mechanical integrity of castings.</td>
</tr>
<tr>
<td>Al additions</td>
<td>Small Al additions appear to grain refine some Mg-RE alloys through the formation of pro-peritectic Al$_4$RE particles that act as nuclei.</td>
<td>More information is required on which alloys this approach can be applied to and the subsequent effect on properties.</td>
</tr>
</tbody>
</table>

Applicable to all Mg alloys

<table>
<thead>
<tr>
<th>Process</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultrasonic Treatment</td>
<td>Depending on the alloy composition Ultrasonic Treatment can significantly reduce the grain size.</td>
<td>Ultrasonic transducers do not function at elevated temperatures and ultrasonic attenuation is a concern. May not be as effective for large castings.</td>
</tr>
<tr>
<td>Electromagnetic stirring</td>
<td>An effective and clean process that can produce fine grain sizes.</td>
<td>Electromagnetic equipment adds cost. The process appears to be more suited to casting basic shapes (billet and slab) rather than components.</td>
</tr>
<tr>
<td>Melt Conditioning</td>
<td>Melt conditioning is able to increase the number of nucleation events, significantly reducing the grain size.</td>
<td>Melt conditioning equipment adds cost to casting process. More data is needed on the performance of castings produced this way.</td>
</tr>
</tbody>
</table>
Tamura et al. [15] found that the presence of Fe increases the grain size of high purity AZ91E alloys. They presented evidence showing that the ternary Al-C-O particles, which are thought to be effective nucleants, would transform to ineffective quaternary Al-C-O-Fe compounds in the presence of Fe [15]. They thus concluded that Fe was an inhibiting element for the grain refinement of Mg-Al alloys, as it poisoned the potency of the Al-C-O nucleants by transforming them to Al-C-O-Fe [15]. Gao et al. [16] then found that the grain size of scrap AZ91 decreased with decreasing Fe content from 0.024% to 0.0009%, and suggested that Fe acts as an inhibiting element for grain refinement. This apparently contradicts the basis of the Elfinal process. On the other hand, it is interesting to note that although the Elfinal process has been known since the early 1940s [8-10, 17, 18], no micrographs or data can be found in the literature (including the original patent on this process [10]) as to the effectiveness of this process. It is likely that some experiments were conducted but for some reason the results were not published.

The influence of Fe on the grain refinement of high purity Mg-Al alloys was investigated using various methods of Fe addition [19, 20]. The addition of Fe in the form of anhydrous FeCl₃ produced obvious grain refinement in high-purity Mg-3%Al and Mg-9%Al alloys. Grain refinement could also be achieved through the uptake of Fe from the crucible surfaces; however, the addition of Fe in the form of ALTAB™ Fe75 compact did not cause grain refinement. The use of anhydrous FeCl₃ also leads to grain refinement in high-purity Mg-Al alloys when these alloys are melted in carbon-free aluminum titanate crucibles, suggesting that the Elfinal process has little to do with the Al₄C₃ hypothesis proposed by Emley. In contrast, Fe- and Al-rich intermetallic particles were observed in many Mg grains when the Elfinal process was used, suggesting that these particles are possibly nucleants for Mg grains.

The above contradictions illustrate the problem in understanding the mechanisms operating during solidification. Although the use of Fe results in good grain refinement via the Elfinal process [10-12], it has been found that Mg-Al alloys with ultra-low levels of Fe have a naturally fine grain size compared to similar alloys that contain normal impurity levels of Fe. Taking another approach, Nelson [8] observed that AZ92 and AZ63 alloys containing <0.001% Fe had a finer grain size than the same alloys containing 0.02% Fe. Tamura et al. [15, 21, 22] confirmed Nelson’s observations. The grain refinement by control of impurity levels is referred to as native grain refinement. They also showed particles containing Al, C and O in the central regions of certain Mg grains in high purity Mg-Al alloys and attributed native grain refinement to the presence of AlC₃ or Al-C-O particles.

Native grain refinement was observed exclusively in high purity Mg-Al alloys. The grain size of Mg-9%Al alloys was found to increase with increasing proportion of commercial purity up to 100% Mg [20]. Little Fe and Mn is present in the high purity Mg-Al alloys and as there was no uptake of any other impurities from the crucible used in this study, it was assumed that the nucleant particles involved in native grain refinement of Mg-Al alloys are AlC₃ or Al-C-O particles as concluded by Tamura et al. [15, 22]. In commercial purity Mg-Al based alloys the presence of Fe or Mn degrades the potency of AlC₃ or Al-C-O by forming quaternary Al-C-O-Fe or Al-C-O-Mn compounds.

Investigations conducted since the 1930s have been unable to provide consensus on the influence of Mn on the grain refinement of Mg-Al alloys. It was believed that superheating is very closely related to the presence of impurity elements such as Fe, Mn and C, in the alloy melts. Tiner [13] found that high-purity Mg-Al alloys with Mn content less than 0.02% showed no appreciable superheating effect, while Mg-Al alloys containing 0.19% or more Mn demonstrated some grain refinement during superheating. Tiner [13] further claimed that binary Mg-Al alloys that can be refined by superheating need to contain either Fe or Mn in excess of their solubilities at the liquidus temperature of the alloy. Nelson [8] subsequently investigated the grain refinement of an AZ92 (Al: 9% and Zn: 2%) alloy with 0.4% or more Mn, and found that alloys with such a high Mn content could not be grain-refined by superheating unless the excessive Mn was settled out by holding at low temperatures (650 – 700°C) and then removed from the melt by sludging out. Nelson’s [8] observations contradict Tiner’s [13], who obtained full grain refinement in Mg-9%Al alloy containing 0.98% Mn.

Tamura et al. [15, 22] re-examined the influence of minor impurity elements on the grain refinement of high purity Mg-9%Al alloys (Fe < 0.001%, Mn < 0.001%). They used an Al-10%Mn master alloy to add Mn. Contrary to Tiner’s observations, Tamura et al. [15] found that the average grain size of a chill bar sample increased progressively from about 40 μm to 160 μm with increasing Mn content from less than 0.001% to 0.47%, where Fe was kept as low as 0.002%. Al-C-O particles were observed in the Mg grains before Mn additions. However, it was found that, in the presence of Mn, these ternary particles transformed to quaternary Al-C-O-Mn. Tamura et al. [15] therefore concluded that Mn was a grain refinement inhibitor, as it poisoned the potency of the Al-C-O nucleants by transforming them into Al-C-O-Mn. Particles that contained Al, Mn, Fe, O, and occasionally Si, were also observed at the centers of Mg grains. To add to the complexity of the effect of Mn, they later published an article about the formation of Mn-containing particles in a commercial AZ91E alloy using a single-roll rapid solidification method [23]. Interestingly, they found that cross-shaped Al-Mn(Fe) particles were frequently observed when the AZ91E melt was rapidly cooled from superheating temperatures (e.g. 900 or 950°C) to 700°C. They then claimed that the cross-shaped Mn-containing particles produced effective nucleation sites for Mg grains. These conclusions differ from those they made before, where they claimed that the nucleant particles are Al-C-O particles and that Mn is a poisoning element for the grain refinement of Mg-Al alloys. Byun et al. [24] observed the existence of Al₅(Mn,Fe)₂ particles within the α-Mg grains in an AZ91 slurry sample containing 0.23% and more Mn, suggesting that Al₅(Mn,Fe)₂ particles act as the heterogeneous nucleation sites. On the basis of Mn solubility calculations, Byun et al. [24] suggested that high Mn concentrations favour grain refinement because supersaturated Mn is likely to precipitate to form Al₅(Mn,Fe)₂ particles where primary Mg grains nucleate. A recent study [25] showed that the addition of 0.1% to 1.0% Mn introduced in the form of an Al-6%Mn master alloy splatter, resulted in obvious grain refinement of high purity Mg-3%Al, Mg-6%Al and Mg-9%Al, as well as commercial AZ31 alloys. The majority of the Mn-containing particles observed in the commercial AZ31 base alloy were of the Al₅Mn type, whereas after addition of extra Mn the predominant group of intermetallic particles changes to the α-AIMn type. The metastable hexagonal α-AlMn phase is likely to be a more effective nucleant for Mg
grains than the stable rhombohedral Al₃Mn₂. It was proposed that the
increase in grain size after a long time of holding at a given
melt temperature is due to the transformation of the metastable ε-
Al₃Mn to the stable Al₃Mn₃. This hypothesis for grain refinement
by Mn can also explain the mechanism of grain refinement by
superheating.

To overcome the shortcomings of the superheating process (e.g.
high operating temperature and applicability to small melts only),
post-war research of grain refinement of Mg alloys shifted to
developing an alternative method applicable to large melts and at
lower operating temperatures. These efforts led to the discovery
of the carbon refining process [26, 27].

In principle, any materials containing carbon can be used as
inoculating additives, and do have a grain-refining effect when
properly inoculated. These additives can be categorized into three
groups: carbonaceous gases, solid carbon additives and organic
carbons. The use of carbon monoxide (CO) or carbon dioxide
(CO₂) was first reported in a US patent [28] to give satisfactory
grain refinement when bubbled through the melt. Subsequently a
number of carbonaceous gases were reported to have a grain-
refining ability to Mg alloys, such as acetylene, methane, and
natural gas [8, 27, 29-31]. Solid carbon additives include carbon
powder, lampblack, magnesite [28], calcium carbide [32, 33],
calcium cyanamide, paraffin wax, silicon carbide [34, 35].

From an operational aspect, the carbonaceous gases and organic
carbons can be more easily introduced into Mg melts than the
solid carbon additives. In addition, bubbling a melt also helps to
degas the melt. The addition of carbon in the form of C₃Cl₄ or
Cl₂-CCl₄ blend was widely used before the 1970s because of the
combined action of grain refining and degassing. The major
problem of using carbonaceous gases, particularly chlorine-based
gases, is the emission of chlorinated hydrocarbons (CHC).
Considerable efforts have been made to produce a commercially
reliable carbon-based grain refiner for Mg-Al alloys. For example,
Renger and Simon [36] reported an effective grain refiner named
Nucleant 5000, which appeared to work satisfactorily on AZ91
alloys. Waxfluorspar-carbon grain refiners were developed early
in England [37] and in Canada [38]. Liu et al. [39] reported an
Al₄C₃-SiC-Al alloy suitable for grain refinement of Mg-Al alloys
such as AZ31 and AZ63. Motegi et al. [40, 41] disclosed a carbon
grain refiner that contains either pure carbon powder or a mixture
of carbon and Nb₂O₅, or V₂O₅ powder. However, to date carbon-
based grain refiners prove unreliable and produce inconsistent
grain refinement in Mg-Al alloys.

There have been many other particles tried in Mg-Al alloys
including borides [42, 43], ZnO [44] and AlN [45]. This is
understandable because the presence of foreign particles will
do with their use, be it instability in the melt, rapid settling or that
the grain refinement efficiency is not sufficient enough.

Table 2 groups the mechanisms of grain refinement in Mg-Al
alloys into four categories, solute additions, impurity poisoning, C
additions and other nucleant particles. A recent review [6] by the
authors has shown that broadly all of the grain refining techniques
that have been proposed so far can be grouped into one of these
categories. This begins to simplify the issues related to grain
refinement of Mg-Al alloys, but more work is required to confirm
these hypotheses.

The only commercially successful technology is Zr grain
refinement by Mg-Zr master alloys while carbon inoculation is
sometimes used for sand and gravity die casting of Rare Earth
(RE) alloys for aerospace and automotive applications [5]. Zr is
almost the perfect grain refiner except that it is not able to grain
refine Al containing alloys due to the affinity of Al for Zr reacting
to form Al₁₃Zr. Some research has been undertaken to understand
why Zr is such a remarkably effective grain refiner, although
further work is required to optimize these alloys. It has been
shown that the performance of Mg-Zr master alloys is related to
the Zr particle number density of particles less than 5 microns in
size as the larger particles settle out during alloying [46, 47].
Recently, it has also been shown that small Al additions are able
to effectively grain refine some Mg-RE containing alloys [48].

There is increasing interest in processes that may assist the
nucleation of grains such as Ultrasonic Treatment (UT) and Melt
Conditioning. The results to date have shown that they are
effective in producing considerable refinement. However, little
industrial application has occurred to date and thus the focus of
future research is on their practical implementation in casting
operations [49].

More details on research into the grain refinement of Mg
alloys undertaken to date can be found in two reviews by the
authors [50].

Theoretical Developments

As highlighted in the previous section and Tables 1 and 2, the
proposed theories of grain refinement of Mg alloys have
essentially been explanations of the observed, and often
contradictory, refinement effects with little definitive evidence of
particle characteristics such as type and size distribution. The
underlying assumptions such as the role of Fe and the presence of
C, were used to develop new refining methods. There has been
much effort focused on identifying potential nucleant particles
[51] or speculation on the type of native or impurity nucleant
particles [20]. It is known that grain size is affected by the Al
content in Mg-Al alloys but alloy composition was not taken into
account when analyzing grain refinement outcomes until about
2005 [50].

In 2001 a theory was proposed that brought together alloy
constitution and particle potency showing that grain size is related
to the growth restriction factor Q [52]. The recent
Interdependence Model, equation 1, is a rigorous predictive
equation that highlights the factors affecting the as-cast grain size
and incorporates elements of the Free Growth Model [53]
developed in the 1990s for the calculation of ΔΤᵣ, the
undercooling required for nucleation on a particle of a particular
size [54].

\[
d_{\text{gr}} = \frac{D \cdot \Delta T_{\text{min}}}{\nu \cdot Q} + \frac{4.6 \cdot D}{\nu} \left( \frac{C^*_q - C_p}{C^*_q (1 - k)} \right) + x_{\text{sd}}
\]  

(1)

where \(D\) is the diffusion coefficient, \(\nu\) the rate of growth of the
solid-liquid (S-L) interface, \(C^*_q\) the composition of the liquid at
the S-L interface at \(x_{\text{grs}}\), \(\Delta T_{\text{min}}\) the undercooling required for
nucleation on the largest available particle, and \(x_{\text{sd}}\) the
incremental amount of undercooling required to activate the next
nucleation event as the temperature gradient moves towards the thermal center of the casting. The constant 4.6 is a cut-off factor for the solute profile in front of the S-L interface where \((C_f(x') - C_{sol})/(C_f(x') - C_{sol}) = 1\%\) [54].

![Graph](image)

**Figure 1:** Representation of a grain size vs \(1/Q\) plot showing the contribution of the nucleation-free zone \(x_{sd}\) and the distance to the next most potent particle \(x_{sd}\) to the final as-cast grain size.

The first two terms in equation 1 are determined from the alloy characteristics and the casting conditions defining the formation of the nucleation-free zone \(x_{sd}\) as insufficient CS is developed within this zone to cause a nucleation event to occur [54]. Figure 1 illustrates the change in \(x_{sd}\) with \(Q\) highlighting that for low solute alloys \(x_{sd}\) significantly restricts refinement by preventing nucleation on suitable particles. The third term \(x_{sd}\) is defined by the distance to the next most potent particle, assumed to be constant in Figure 1. The value of \(x_{sd}\) can be decreased by increasing the particle number density, for example, by adding more Zr master alloy.

Plots of the measured grain size vs \(1/Q\) present a straight line that provides a useful tool for the analysis of the mechanisms of grain refinement and has been successfully applied to Al, Ti and Mg alloys [54, 55]. The application of the grain size vs \(1/Q\) plots has revealed the mechanisms of refinement. For example, superheating of commercial Mg-Al alloys and native refinement of high purity alloys are related [20, 56]. It now appears that superheating removes the effect of impurities allowing the more potent native nucleants whether they are carbides or oxide particles, to be available for nucleation thus producing a finer grain size. Also, a comparison of high and commercial purity Mg-Al alloys has highlighted that a major factor controlling grain size.

### Table 2. A summary of the theories proposed to explain the mechanisms of grain refinement of Mg alloys with the weaknesses of each theory.

<table>
<thead>
<tr>
<th>Process</th>
<th>Related approaches</th>
<th>Future Considerations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solute additions</td>
<td>It is well established that growth restriction by alloying elements contributes to grain size reduction. However, it should be noted that alloys that show a high growth restriction effect may not always be commercially attractive or useful.</td>
<td>Lack of physical data to ensure reliable prediction of grain size using the developed predictive equations. It is also clear that whilst this is a significant effect it is not the most important one, particularly in casting alloys.</td>
</tr>
<tr>
<td>Impurity poisoning</td>
<td>It appears that the superheating effect, the Elfmol process and Native grain refinement are all related to the effect of impurities precipitating on the surface of particles.</td>
<td>Whilst there is circumstantial evidence for this occurring, it is difficult to obtain direct evidence. Also the wide variety of effects means that it is difficult to explain all observations using this approach.</td>
</tr>
<tr>
<td>C inoculation and SiC</td>
<td>Carbon-based inoculants are the most effective nucleants known and the ones that are used commercially. There have been many different approaches proposed. A recent breakthrough has been the understanding that carbon reacts with Al to form Al₃C₃ particles and Al₂MgC which can both act as nuclei. Al₂CO may be a nucleant in some situations.</td>
<td>An effective commercial approach has not as yet been able to be developed and the effectiveness of grain refinement is significant rather than very good.</td>
</tr>
<tr>
<td>Other nucleant particles</td>
<td>No other attractive nucleants have been identified yet, despite many trials.</td>
<td>A potent nucleant such as Zr for Al-free Mg alloys may exist or may not. However, given the commercial importance of Mg-Al based alloys, it is worth further effort to identify a potent grain refiner for these alloys.</td>
</tr>
</tbody>
</table>

**Mg – Al alloys**

| Mg – Al alloys               | Both soluble and insoluble Zr contribute to grain refinement.                        | Prediction capability to take into account simultaneous increases in solute Zr and Zr particle number density needs further refinement. |

**Mg – Zn, RE alloys that do not contain Al, Mn, Si, Fe**

| Mg-Zr master alloys         | Both soluble and insoluble Zr contribute to grain refinement.                        | Prediction capability to take into account simultaneous increases in solute Zr and Zr particle number density needs further refinement. |
| Al additions                | Small Al additions appear to grain refine some Mg-RE alloys through the formation of pro-peritectic Al₁RE particles that act as nuclei. | More information is required on which alloys this approach can be applied to and the subsequent effect on properties. |

**Applicable to all Mg alloys**

| Mechanical approaches       | Includes approaches such as ultrasonic treatment, electromagnetic stirring processes and melt conditioning. | For most of these approaches there is no agreed theory. Depending on the technique, issues such as cavitation and fragmentation of impurity particles and α-Mg grains are proposed as generators of many nucleants. |
is the nucleation-free zone which reflects the effect of alloy chemistry (Q) in producing large grain sizes for AZ31 and finer sizes for AZ91 [54]. It has also shown the improvements in nucleation efficiency on the addition of SiC to Mg-Al alloys [57].

These plots have been applied to analyzing the performance of Mg-Zr master alloys showing that Zr particle number density is the key factor differentiating different types of master alloy [46, 47]. Also, the linear form of \( d_p \) vs \( 1/Q \) has been shown to apply to a wide range of casting conditions and the application of Ultrasonic Treatment highlighting the important role of Q [6].

**Issues Remaining**

Current understanding can be summarized as: (1) high purity Mg-Al alloys exhibit native grain refinement with Al carbide or oxide particles being the nucleant particles and addition of either Fe or Mn individually leads to further refinement; (2) commercial Mg-Al alloys exhibit poor refining performance due to an interaction between Al, Fe and Mn forming an intermetallic layer of lower nucleation potency on the native nucleants; (3) Zr remains the best refiner for Mg-Zn and -RE alloys without Al. However, there is no ideal grain refining technology for Mg alloys. Even the superior Mg-Zr master alloy produces sludge wasting considerable Zr that does not contribute to refinement. In almost every case there are issues with commercial implementation and considerable gaps in our knowledge of the mechanisms of grain refinement.

For Mg-Al alloys poisoning by impurity and minor elements appears to be a major issue preventing the development of a reliable grain refiner and it is probable that a different approach to developing a grain refining system is needed. For all Mg alloys the formation of a nucleation-free zone reduces grain refining performance particularly in lean alloys. Strategies for tackling these issues are discussed in a recent publication [6].

The recent investigations of mechanical methods such as ultrasonic treatment, electromagnetic stirring and melt conditioning show promise. However, proving that these methods will be practical and cost effective for industrial application is an important next step.

Research on the grain refinement of Mg alloys has been very active but that has been largely driven by researchers in universities and institutes over the last two decades. Closer collaboration between academia and industry will help address these issues and more importantly, increase the impact on industrial practice.

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**References**

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