Phase Transformation Kinetics and Alloy Microsegregation in High Pressure Die Cast Magnesium Alloys

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Project ID #LM091

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Overview

Timeline
• Start: October 2013
• End: October 2017
• 35% Complete

Budget
• Total project funding
  – DOE share: $600K
  – Contractor share: na
• Funding received in FY14: $175K
• Funding for FY15: $148K

Barriers
• Lack of understanding and predictive models for HPDC/SVDC Mg processes:
  – Limits ability to quickly optimize Mg components and reduce costs.
  – Limits ability to rapidly develop new alloys & processes for challenging applications and increases risk.

Partners
• Ford Motor Company (Dr. Mei Li)
• Tsinghua University (Prof. S. Xiong)
Relevance

• Mg components represent a major opportunity for reducing vehicle weight (35-50%), energy consumption and greenhouse gas emissions.

• High Pressure Die Casting (HPDC) is used for over 90% commercial Mg products because it is fast, economical, and yields complex thin-wall Mg components.

• High solidification rates, from 10 to 1000ºC/s – far from equilibrium.

• No systematic, quantitative knowledge of microsegregation or phase transformation kinetics → limits ICME predictive capabilities & thus increase risks, time and cost for use of Mg in new and challenging applications.
Relevance – Project Objectives

• Quantify & understand phase transformation & microsegregation during HPDC/SVDC

• Quantify & understand phase transformation & changes in microsegregation during solution treatment & ageing

• Develop physics-based transformation kinetics micro-models

• Transfer knowledge through NIST D-Space Repository & UM Materials Commons
Approach / Tasks

1. Simulate & manufacture high quality HPDC/SVDC plates & complex shapes of binary and ternary alloys
   - Mg-Al & Mg-Al-X; X=Mn, Zr, Ca, Sr (AM, AZ, AX, AK alloys)
   - Precision MagmaSoft simulation

2. Systematic study of phase transformation & microsegregation during HPDC/SVDC
   - Advanced EPMA & analysis
   - Quantitative Optical and SEM analysis

3. Systematic study of phase transformation & changes in microsegregation during solution treatment & ageing
   - Selected alloys
   - Advanced EPMA & analysis
   - Quantitative Optical, SEM & TEM analysis

4. Develop physics-based transformation kinetics micro-models
   - Analytical model coupled with precision MagmaSoft results
   - Validate ThermoCalc model for precipitate evolution

5. Transfer knowledge through NIST D-Space Repository & UM Materials Commons
# Milestones

<table>
<thead>
<tr>
<th>Tasks</th>
<th>Year 1</th>
<th>Year 2</th>
<th>Year 3</th>
<th>Year 4</th>
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<tbody>
<tr>
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<td>1 2 3 4</td>
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<tr>
<td>Task 1: Project Management Plan</td>
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<td>Task 2: Manufacture HPDC/SVDC plates and complex-shaped HPDC/SVDC casts and simulation</td>
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<td>Task 3: Quantitative characterization of phase transformation kinetics and microsegregation in HPDC castings</td>
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<td>Task 4: Quantitative characterization of phase transformation kinetics and microsegregation during heat treatment of SVDC castings</td>
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<td>H  G</td>
<td>I  K</td>
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<tr>
<td>Task 5: Develop a physics-based phase transformation kinetics model to capture microstructural evolution and microsegregation during HPDC/SVDC and heat treatment</td>
<td></td>
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<td>J  L</td>
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<tr>
<td>Task 6: Transfer the project knowledge to industry and research community through micro-models and data housed in the UM DOE PRISMS Materials Commons and NIST data repositories</td>
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</table>
High Pressure Die Casting & Alloys

• Plate castings being provided by Tsinghua University & Ford (Mag-tec)
• AM60 & AZ91 complex castings provided by Ford

<table>
<thead>
<tr>
<th>Alloy Compositions (wt%)</th>
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<tbody>
<tr>
<td>Mg</td>
</tr>
<tr>
<td>----</td>
</tr>
<tr>
<td>Bal</td>
</tr>
<tr>
<td>Bal</td>
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</table>
Technical Progress: Precision HPDC Simulation

- Precision simulation achieved using advanced IHTC (from Ford/Tsinghua: 3-countries Mg Front End program) and very fine mesh
- Solidification front velocity, cooling rate (°C/s), and thermal gradient (°C/mm) determined as a function of location through plate
- Cooling rates ranging from 100-300°C/s are predicted
Technical Progress: Phase Quantification

![Diagram showing a phase diagram and microstructure image with annotations for porosity, in-mold grains, solute-rich regions (SRRs), externally solidified crystals (ESCs), and 435°C.](image)

- **Porosity**
- **In-mold grains**
- **Solute-rich regions (SRRs)**
- **Externally solidified crystals (ESCs)**
Technical Progress: Location Dependent Phase Distribution*

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Center</th>
<th>Edge</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al- Mn intermetallics</td>
<td>0.1</td>
<td>0.2</td>
</tr>
<tr>
<td>pores</td>
<td>1.6</td>
<td>0.3</td>
</tr>
<tr>
<td>β-phase</td>
<td>2.0</td>
<td>1.9</td>
</tr>
<tr>
<td>solute-rich regions (SRRs)</td>
<td>24.0</td>
<td>38.7</td>
</tr>
</tbody>
</table>

* Example: AM60 2.5mm SVDC Plate

- β-phase volume fraction much lower than predicted by Scheil solidification model (2% vs 9.1%) and not location dependent
- Higher fraction of SSRs observed at edge of castings
Technical Progress: Characterization of Grain Size Distributions*

- Effect of:
  - Alloying
  - Plate Thickness
  - Location

- Electron back-scatter diffraction (EBSD)

* Example: AM60 5mm thick plate
Technical Progress: Grain Size Distributions

- Bimodal distribution represents in-mold grains and Externally Solidified Crystals (ESCs)
- Lower number of ESCs at edge of castings
- Peak grain size slightly larger at center (8 µm vs. 5 µm)
- Grain size and number of ESCs increase with increasing plate thickness
- Grain size was not influenced by alloy content
To construct segregation profile (WIRS):
1. determine partitioning direction
2. order and rank data (Al solute)
3. assignment of fraction solid

\[
\bar{C}^i_j = \frac{C^j_i - C^j_{min}}{\sigma_j} \quad \sum_{j=1}^{n} \bar{C}^j_i = \bar{C}_i = \frac{\sum_{j=1}^{n} \bar{C}^j_i}{n}
\]

\[
f_s = (R_i - 0.5)/N
\]
- EPMA microsegregation profile at the center of the castings is close to Scheil up to a high solid fraction
Reconstructing the Microsegregation Profile

- Microsegregation deviates from Scheil as you move towards the edge of the casting

![Graph showing concentration of Al (wt.%) against fraction solid for different regions in a 5.0 mm AM60 casting.](image-url)
Technical Progress: Reconstructing the Microsegregation Profile

- Solute trapping increases near the edge of the plate where cooling rate is the highest.

![Graph showing concentration of Al vs. fraction solid for different positions in the plate.](image)

- Graph legend:
  - Edge (5µm)
  - 50 µm
  - Near Edge (100–400 µm)
  - Quarter
  - Center
  - Scheil

- Graph title: 5.0 mm AM60

- Arrows indicating increased cooling rate.
Technical Progress: Micro-Model
As-cast Microstructure Prediction

- Solidification rate and composition dependent partition coefficients can be expressed as [Aziz & Kaplan 88’]:

\[ K_v = \frac{a_0 v}{D_i} + K_e + \frac{a_0 v}{D_i} + 1 - (1 - K_e)C_{lv} \]

- \( v \): solidification front velocity (MagmaSoft)
- \( D_i \): interface diffusion coefficient
- \( k_e \): equilibrium partition coefficients (ThermoCalc/PANDAT)
- \( C_{lv} \): the solidification rate dependent liquidus concentration
- \( a_0 \): solute trapping parameter <- experimentally calibrated
Responses Reviewers’ Comments

• NA - This project has not yet been reviewed
Partnerships/Collaborations

- Ford Motor Company: Mei Li, Jake Zindel and Larry Godlewski
  - Provided super vacuum die cast samples and components
  - Provided HPDC MagmaSoft casting simulations
  - Collaborating on development of kinetics micro-model

- Tsinghua University: S. Xiong (under contract to Ford Motor Co.)
  - Provided super vacuum die cast samples

- OSU: J. C. Zhao and Alan Luo
  - Informal collaboration on precipitation evolution and diffusion
Remaining Challenges and Barriers

- Microstructural scale (cell size) and EPMA beam size are similar which makes direct characterization of microsegregation a challenge.
- Our approach is a “forward” model which will be used to extract the actual (true) microsegregation behavior from the experimentally measured EPMA microsegregation profile.
Proposed Future Work

• Continuing systematic characterization of microsegregation & phase transformation kinetics in as-cast AM alloys, Mg-Al binaries and ternaries
• Begin systematic characterization of microsegregation & phase transformation kinetics following solution treatment and aging of selected SVDC alloys
• Continue developing electron probe micro-analyzer “forward model” and use to extract the true microsegregation behavior
• Develop micro-models to predict microstructure & microsegregation evolution during HPDC and after heat treatment
• Transfer knowledge to industry and the research community through micro-models and data housed in UM DOE PRISMS Materials Commons and the NIST DSpace Repository
Summary

• Objective: Combining advanced experimental techniques, analytical models & simulation tools to develop a systematic understanding of phase transformation kinetics in HPDC Mg-Al-X alloys

• Robust characterization & simulation methods are being developed for
  – phase quantification via SEM
  – grain size via EBSD
  – microsegregation via EPMA
  – simulating solidification velocities & phase transformation kinetics

• Templates have been developed for uploading our data & metadata to Material Commons
Summary - Findings

- HPDC alloys have a bimodal distribution with contributions from in-mold grains and externally solidified crystals

- HPDC plates undergo cooling rates from 100 to 300 °C/s

- β-phase volume fraction is much lower than predicted by Scheil solidification models (2% vs ~9% for AM60) and more Al is partitioned to the α-Mg

- Microsegregation profiles are casting location dependent and current models must be modified to account for this
Technical Back-Up Slides
Effect of Cooling Rate on $\beta$-phase Fraction

- As cooling rate increases we expect:
  - $\beta$-phase fraction to decrease
  - More Al will be trapped in $\alpha$-Mg (increased fraction of SRRs)
Developing a HPDC Solidification Model

- Scheil solidification model over predicts the amount of as-cast eutectic phases, and especially will for fast solidification condition (HPDC)
- Is not solidification rate dependent
- Can be improved with solidification rate and composition dependent partition coefficients
• Lower number of ESCs at edge
• Peak grain size slightly larger at center
• As plate thickness increases the grain size increases slightly and a higher fraction of ESCs are observed
• Grain size was not affected by alloy
# Milestones (Details)

<table>
<thead>
<tr>
<th>Milestones</th>
<th>Duration (months)</th>
<th>Tasks</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>0-15</td>
<td>Complete casting SVDC Mg-Al binaries</td>
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<tr>
<td>B</td>
<td>0-15</td>
<td>Complete casting SVDC Mg-Al binaries with a range of processing conditions</td>
</tr>
<tr>
<td>C</td>
<td>12-24</td>
<td>Complete casting SVDC Mg-Al-Mn, Mg-Al-Zn, Mg-Al-Ca, and Mg-Al-Sr ternary alloys</td>
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<tr>
<td>D</td>
<td>2-24</td>
<td>Complete phase transformation kinetic study of binaries and Mn and Zn ternaries</td>
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<tr>
<td>E</td>
<td>2-24</td>
<td>Complete microsegregation characterization of binaries and Mn and Zn ternaries</td>
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<tr>
<td>F</td>
<td>18-36</td>
<td>Complete phase transformation and microsegregation characterization of Ca and Sr ternaries and in complex casting of AM50 and AZ91</td>
</tr>
<tr>
<td>G</td>
<td>12-36</td>
<td>Complete eutectic dissolution phase transformation and precipitate kinetic study of selected Mg-Al binaries and Mg-Al-Zn ternary</td>
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<tr>
<td>H</td>
<td>6-24</td>
<td>Complete microsegregation characterization of dissolution phase transformation in binaries and Mn and Zn ternaries</td>
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<tr>
<td>I</td>
<td>18-42</td>
<td>Complete eutectic phase dissolution, phase transformation, precipitation kinetics, and microsegregation characterization of Ca and Sr ternaries</td>
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<tr>
<td>J</td>
<td>12-30</td>
<td>Complete micro-model of Mg-Al binaries and Mg-Al-Mn and Mg-Al-Zn ternaries for HPDC conditions</td>
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<tr>
<td>K</td>
<td>24-48</td>
<td>Complete micro-model for Mg-Al-Ca and Mg-Al-Sr ternaries for HPDC and heat treatment conditions</td>
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<tr>
<td>L</td>
<td>12-30</td>
<td>Incorporate experimental data on Mg-Al binaries and Mg-Al-Mn and Mg-Al-Zn ternaries into Materials Commons and release to public</td>
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<tr>
<td>M</td>
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<td>Incorporate experimental data on Mg-Al-Ca and Mg-Al-Sr ternaries into Materials Commons and release to public</td>
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<td>N</td>
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<td>Incorporate micro-model into Materials Commons and release to public</td>
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