

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

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4/10/2015



Project ID #LM091

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

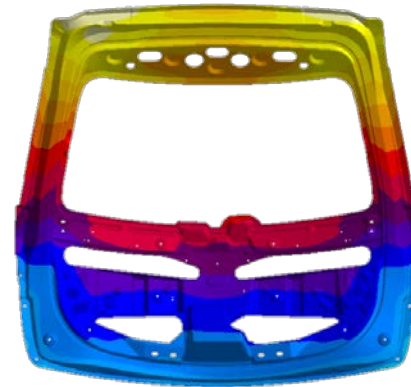
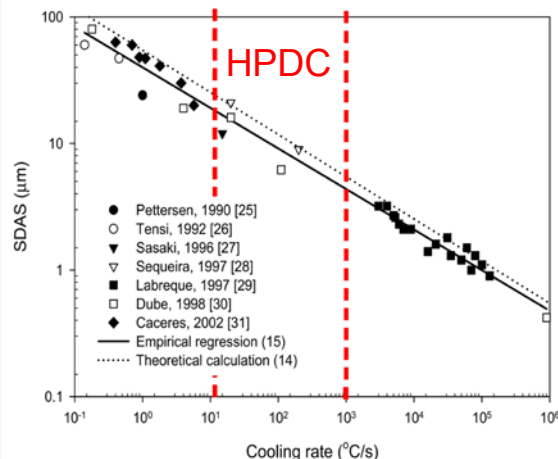
- Lack of understanding and predictive models for HPDC/SVDC Mg processes:
 - Limits ability to quickly optimize Mg components and reduce costs.
 - Limits ability to rapidly develop new alloys & processes for challenging applications and increases risk.

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 1000°C/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

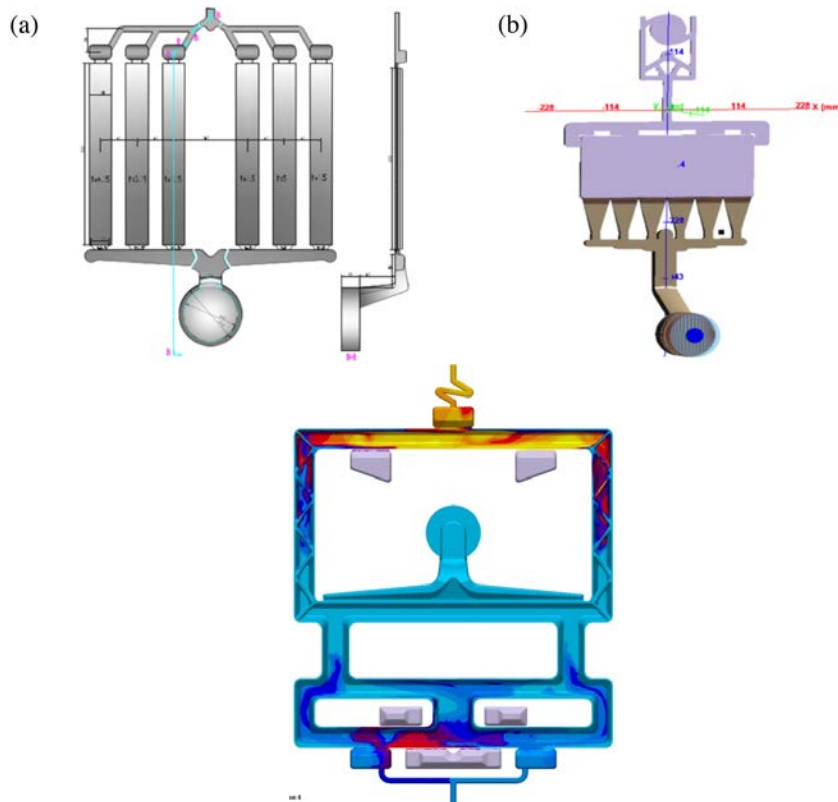
1. Simulate & manufacture high quality HPDC/SVDC plates & complex shapes of binary and ternary alloys
 - Mg-Al & Mg-Al-X; X=Mn, Zr, Ca, Sr (AM, AZ, AX, AK alloys)
 - Precision MagmaSoft simulation
2. Systematic study of phase transformation & microsegregation during HPDC/SVDC
 - Advanced EPMA & analysis
 - 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

Tasks	Year 1				Year 2				Year 3				Year 4			
	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
Task 1: Project Management Plan																
Task 2: Manufacture HPDC/SVDC plates and complex-shaped HPDC/SVDC castings and simulation		★1		★3	A B			C								
Task 3: Quantitative characterization of phase transformation kinetics and microsegregation in HPDC castings								D E				F				
Task 4: Quantitative characterization of phase transformation kinetics and microsegregation during heat treatment of SVDC castings			★2					H				G	I			
Task 5: Develop a physics-based phase transformation kinetics model to capture microstructural evolution and microsegregation during HPDC/SVDC and heat treatment						★4				J						K
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										L						M N

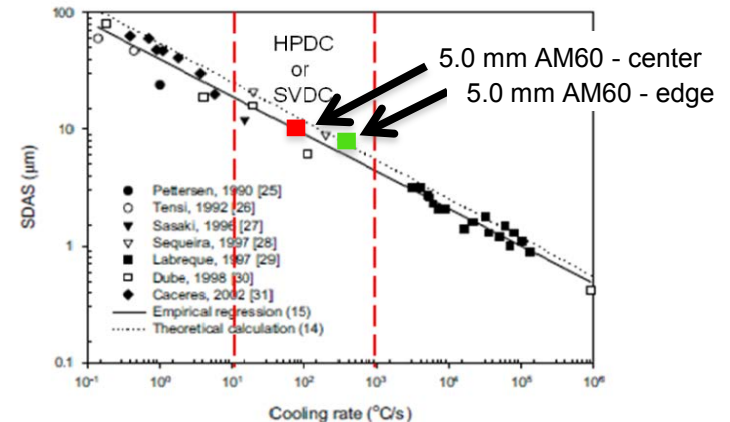
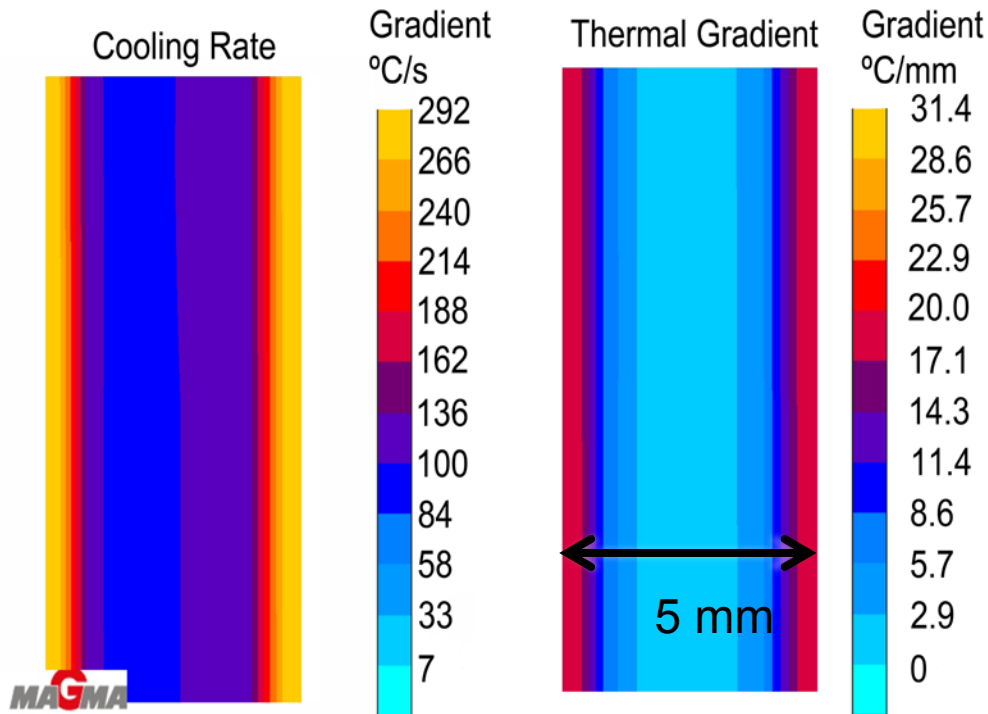
High Pressure Die Casting & Alloys

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



Alloy Compositions (wt%)					
Mg	Al	Zn	Mn	Ca	Sr
Bal	3				
Bal	5				
Bal	9				
Bal	12				
Bal	9	0.5			
Bal	9	1			
Bal	9	2			
Bal	5		0.5		
Bal	5		1		
Bal	5		2		
Bal	5			3	
Bal	5				3

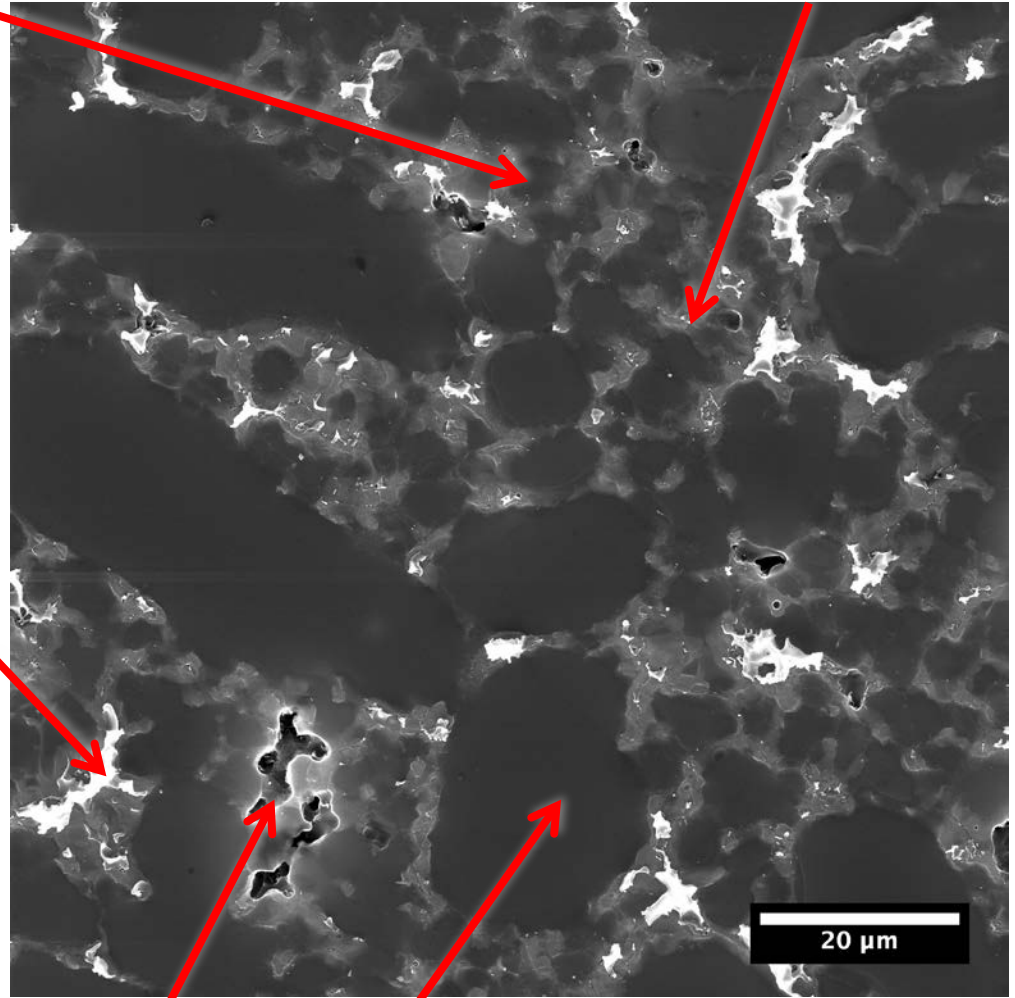
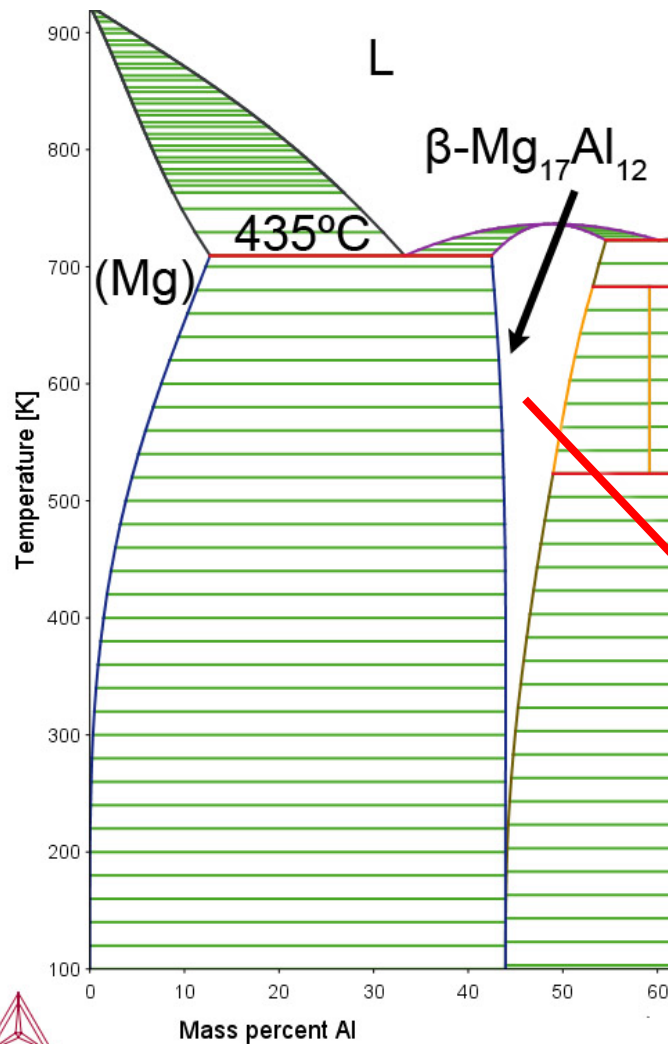
Technical Progress: Precision HPDC Simulation



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

Technical Progress: Phase Quantification

in-mold grains solute-rich regions (SRRs)



porosity

externally
solidified crystals (ESCs)⁹

Technical Progress: Location Dependent Phase Distribution*

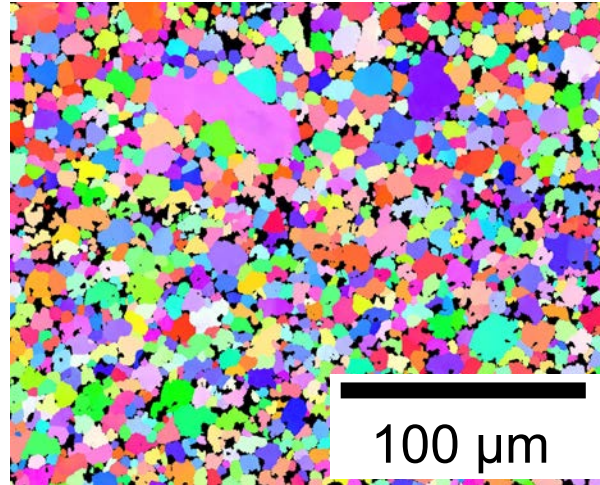
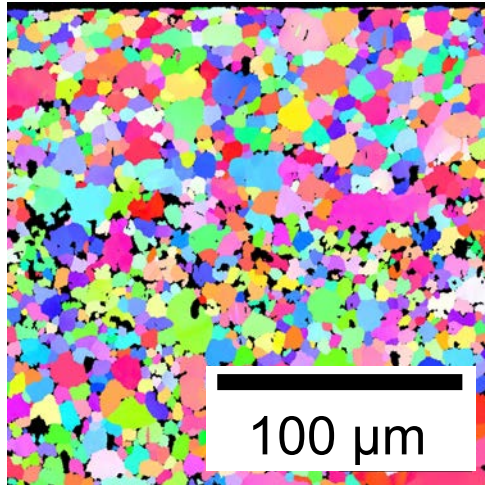
Constituent	Center	Edge
Al- Mn intermetallics	0.1	0.2
pores	1.6	0.3
β -phase	2.0	1.9
solute-rich regions (SRRs)	24.0	38.7

* Example: AM60 2.5mm SVDC Plate

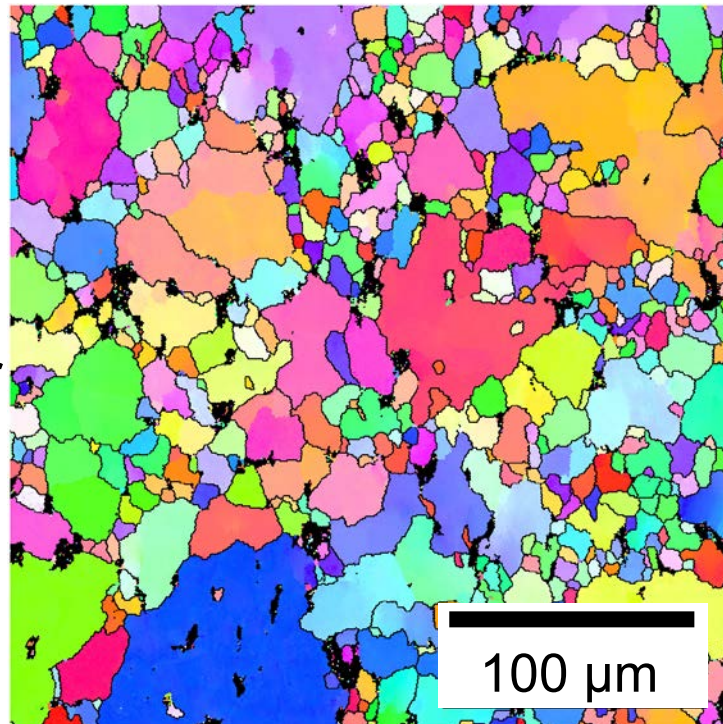
- β -phase volume fraction much lower than predicted by Scheil solidification model (2% vs 9.1%) and not location dependent
- Higher fraction of SSRs observed at edge of castings

Technical Progress: Characterization of Grain Size Distributions*

Edge



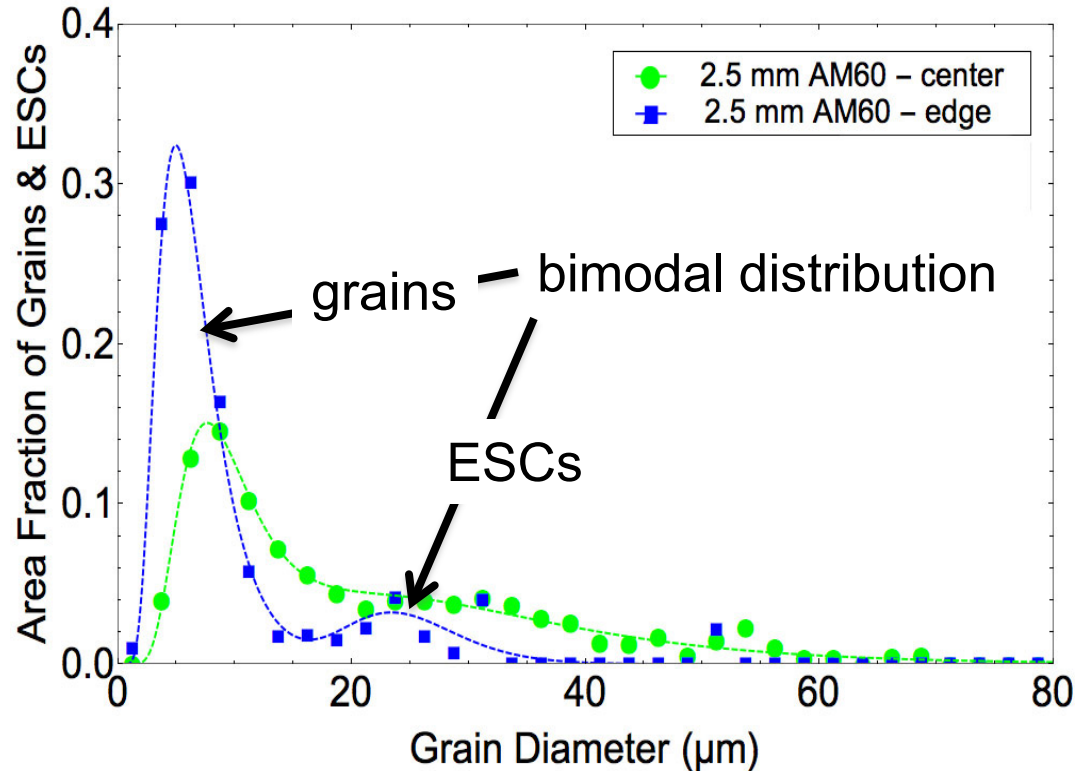
Center



- Effect of:
 - Alloying
 - Plate Thickness
 - Location
- Electron back-scatter diffraction (EBSD)

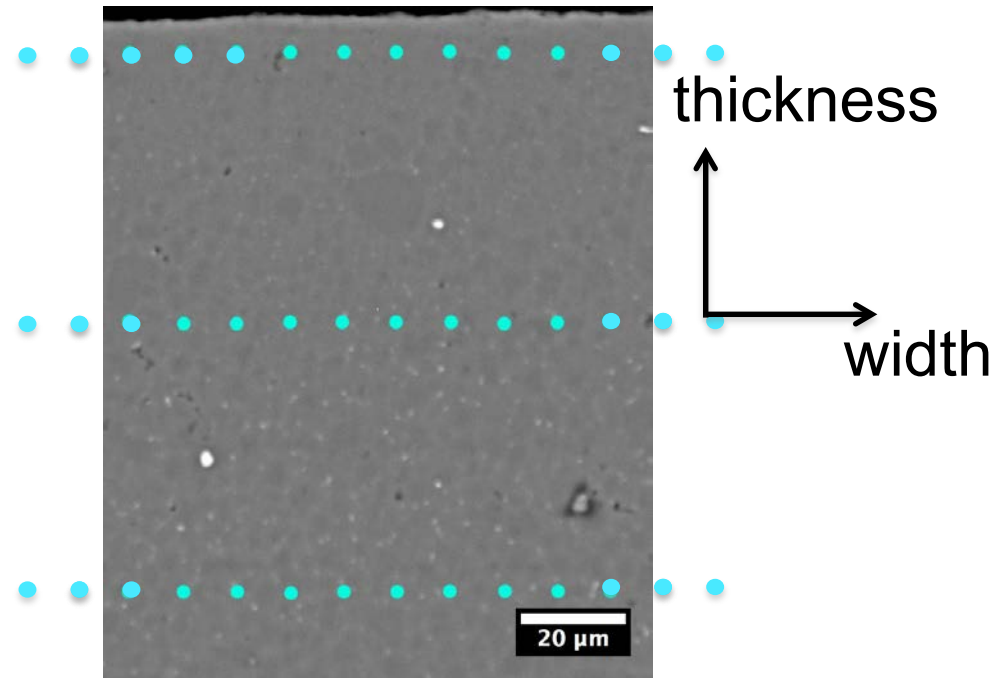
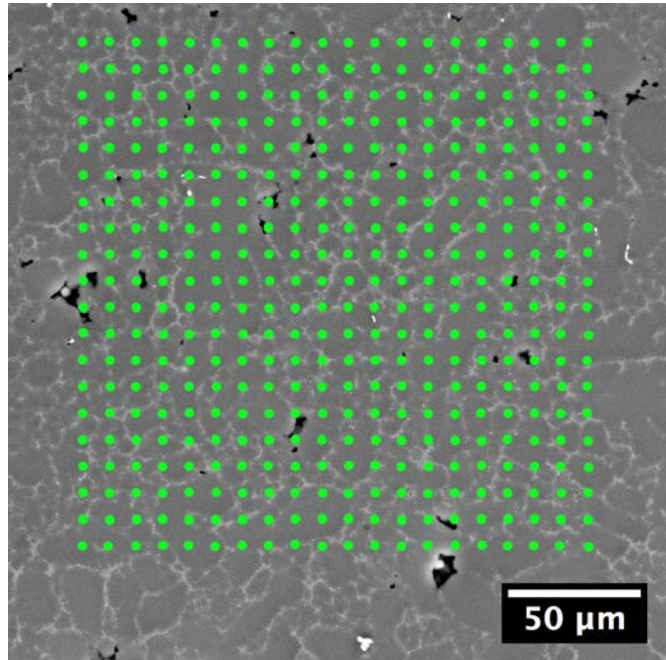
* Example: AM60 5mm thick plate

Technical Progress: Grain Size Distributions



- Bimodal distribution represents in-mold grains and Externally Solidified Crystals (ESC's)
- Lower number of ESC's at edge of castings
- Peak grain size slightly larger at center (8 μm vs. 5 μm)
- Grain size and number of ESC's increase with increasing plate thickness
- Grain size was not influenced by alloy content

Technical Progress: Advanced EPMA Analysis of Microsegregation



To construct segregation profile (WIRS):

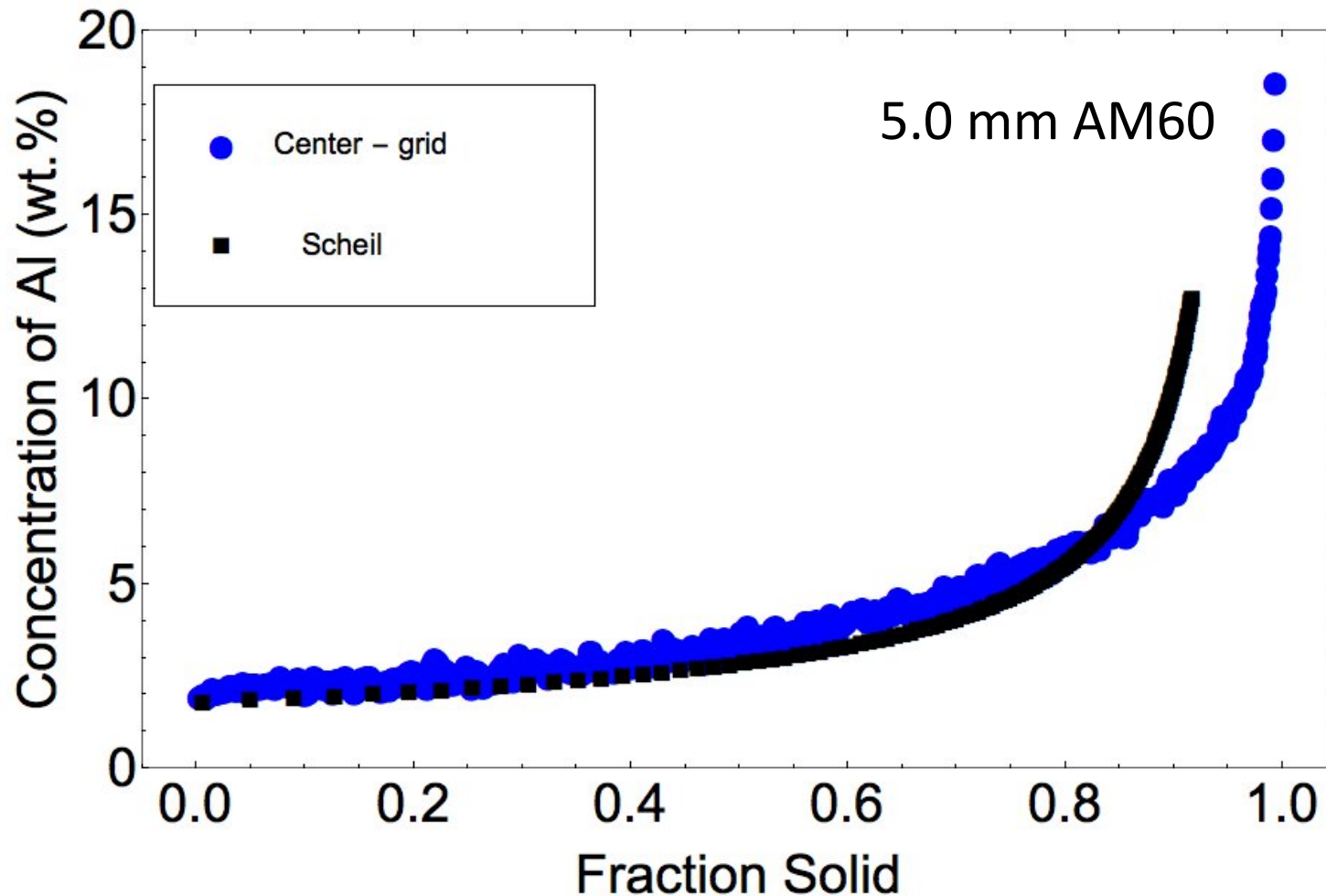
1. determine partitioning direction
2. order and rank data (Al solute)
3. assignment of fraction solid

$$\bar{C}_i^j = \frac{C_i^j - C_{min}^j}{\sigma^j}$$

$$\bar{\bar{C}}_i = \frac{\sum_{j=1}^n \bar{C}_i^j}{n}$$

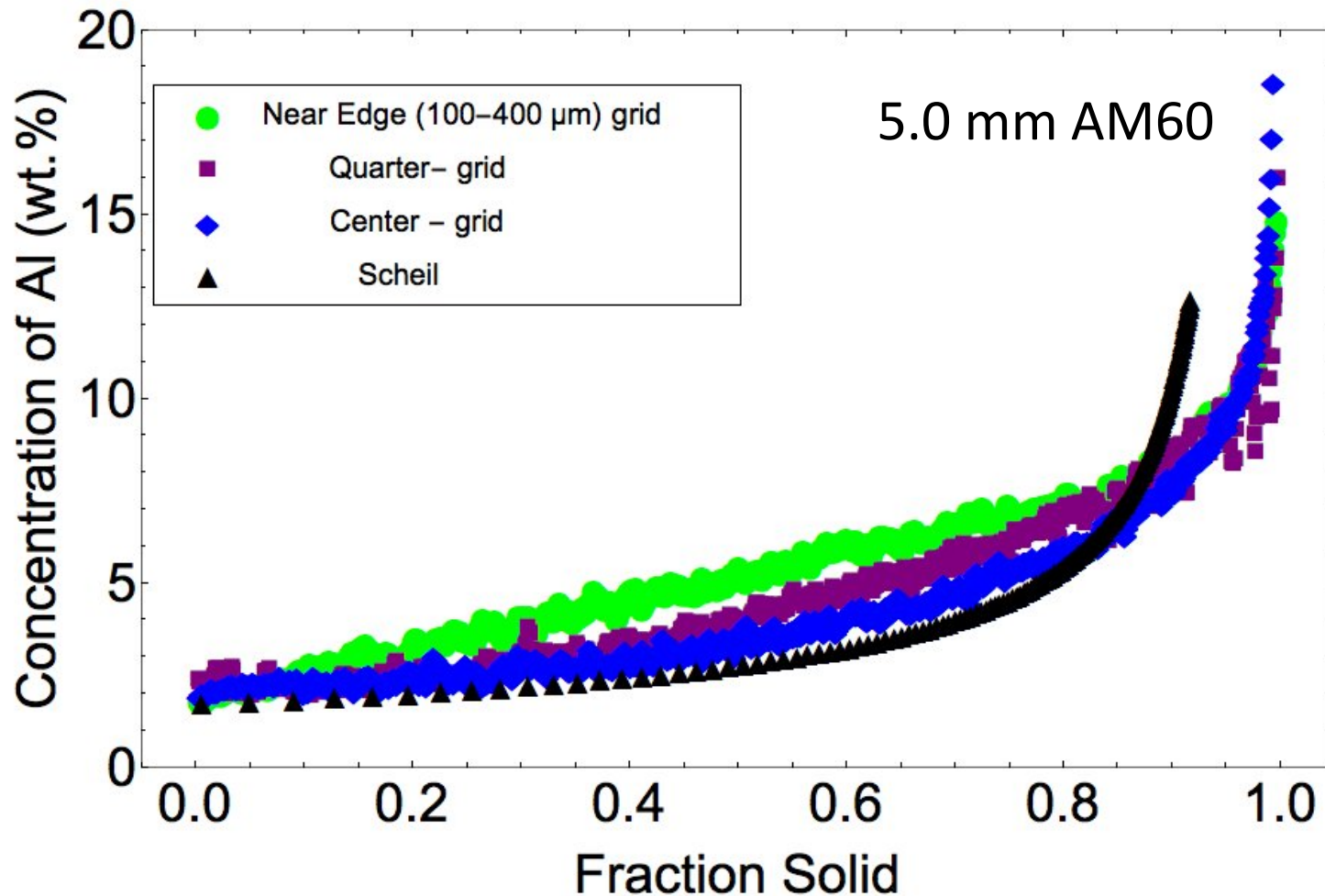
$$f_s = (R_i - 0.5)/N \quad 13$$

Technical Progress: Characterizing the Microsegregation Profile



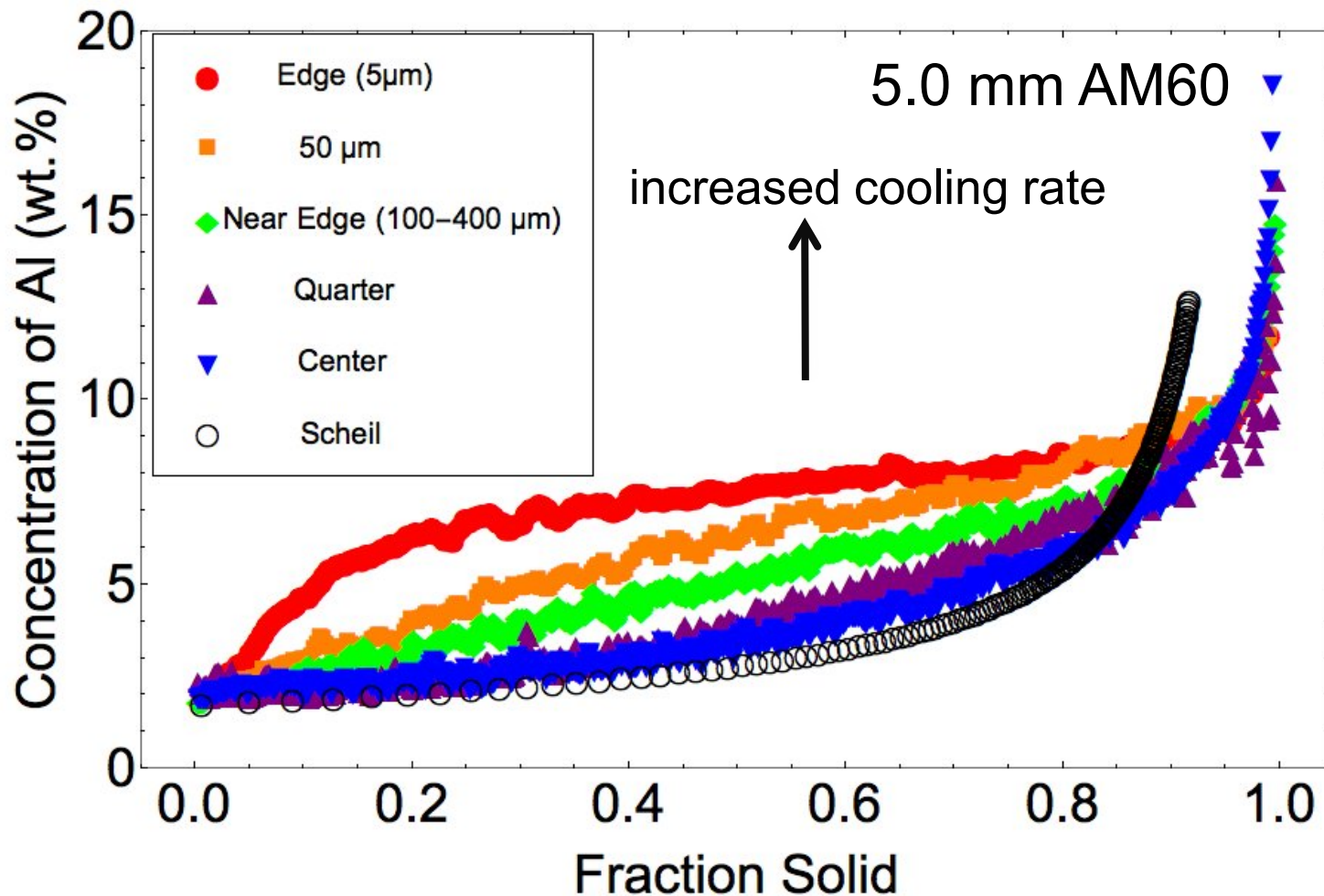
- EPMA microsegregation profile at the center of the castings is close to Scheil up to a high solid fraction

Reconstructing the Microsegregation Profile



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

Technical Progress: Reconstructing the Microsegregation Profile



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

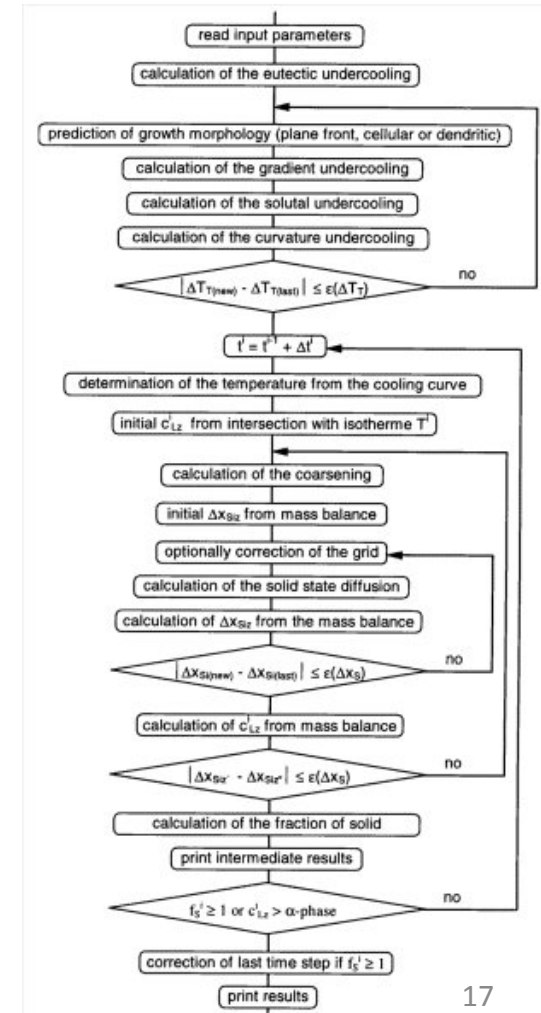
Technical Progress: Micro-Model

As-cast Microstructure Prediction

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

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

- 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



Technical Progress: Materials Commons & NIST D-Space

MaterialsCommons Home ? Help Reviews 0

DOE Mg Kinetics > 0

Processes Files Samples (3) Reviews (0) Notes (3) Sideboard (1)

EBSD HPDC_AM60_5p0_center_scan1... Draft

Mon Feb 09 2015

Inputs

files

sample

-
-
-
-
-
-
-
-

sem-ebsd-data-collection-settings

- Acquisition Time :
- Current(A) :
- Magnification :
- Sample Tilt(°) : 70
- Scan Size (microns) : 500
- Step Size (microns) : 0.7
- Voltage(kV) : 25
- Working Distance (nm) : 20

Outputs

files

- GF_HPDC_AM60_2p5_center_scan1.txt
- IPF_AM60_5p0_center_scan1.tif

Provenance Wizard

SEM EBSD (I) Sample (I) Settings (I) Files (O) Files Done

GF_HPDC_AM60_5p0_center_scan1.txt

130 KB Mon Feb 09 2015

```
# Header: Project1::HPDC_AM60_5mm_center_scan1_Mod::New Partition::Grain Size
2/6/2015
#
# Partition Formula: PCI[&]>0.300 AND GSZ[&;5.000,10,0.300,0,0,8.0,1;]>0.500
# Grain Tolerance Angle: 5.00
# Minimum Grain Size: 2
# Minimum Confidence Index: 0.00
# Multiple Rows Requirement: Off
# Column 1: Integer identifying grain
# Column 2-4: Average orientation (phi1, PHI, phi2) in degrees
# Column 5-6: Average Position (x, y) in microns
# Column 7: Average Confidence Index (CI)
# Column 8: Edge grain (1) or interior grain (0)
# Column 9: Diameter of grain in microns
# Column 10: Aspect ratio of ellipse fit to grain
1 291.704 28.939 76.43 2.460 2.789 0.800 1 6.15 0.73
2 305.412 31.632 64.46 7.133 1.443 0.771 1 4.76 0.57
3 191.174 72.513 157.18 13.320 3.618 0.829 1 9.18 0.52
4 283.970 7.383 84.73 21.600 4.053 0.800 1 6.15 0.46
5 188.411 86.855 168.80 29.521 6.294 0.857 1 11.03 0.57
6 275.728 88.090 87.93 41.346 5.663 0.914 1 14.79 0.59
7 44.748 58.267 310.31 55.339 3.893 0.914 1 11.99 0.62
8 304.818 61.966 54.41 74.925 5.677 0.857 1 19.65 0.30
9 305.864 61.134 53.07 117.567 23.128 0.886 1 51.65 0.40
10 348.488 36.407 37.75 149.890 7.412 0.914 1 14.87 0.70
```

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

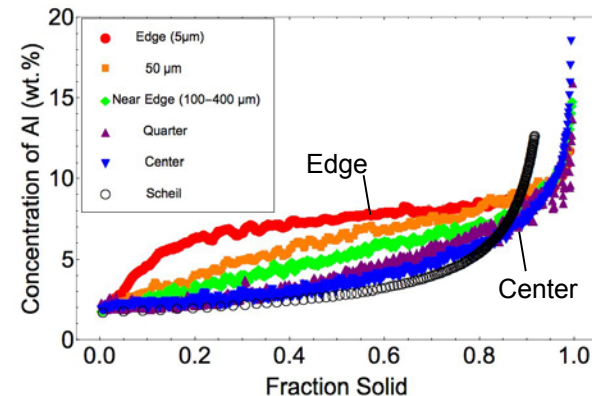
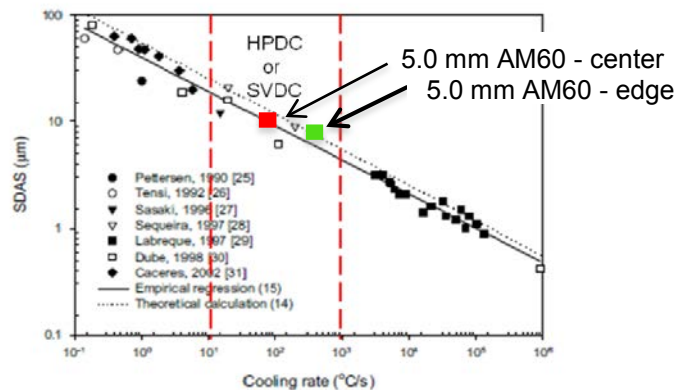
- Continuing systematic characterization of microsegregation & phase transformation kinetics in as-cast AM alloys, Mg-Al binaries and ternaries
- Begin systematic characterization of microsegregation & phase transformation kinetics following solution treatment and aging of selected SVDC alloys
- Continue 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

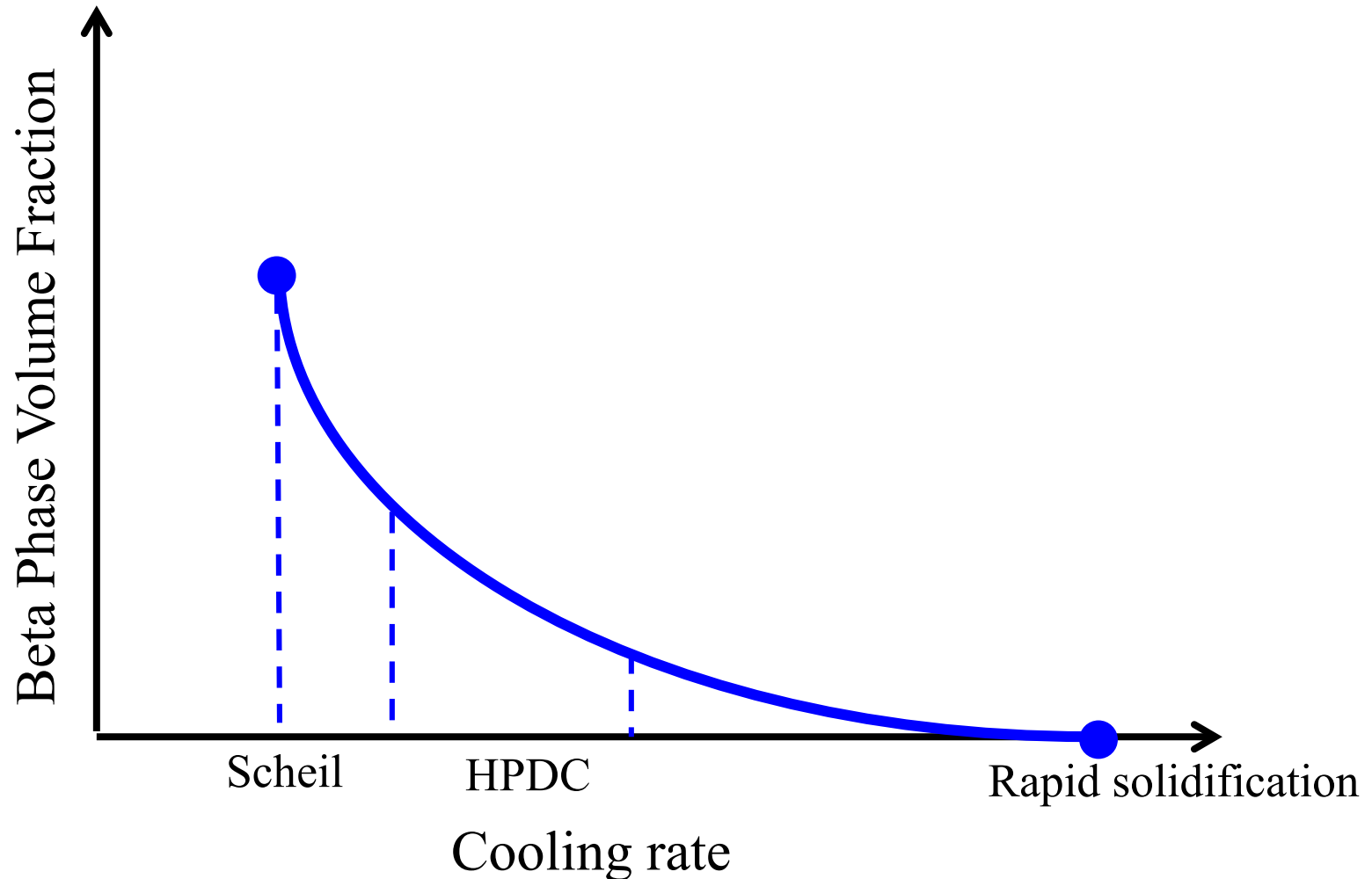
Summary - Findings

- 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



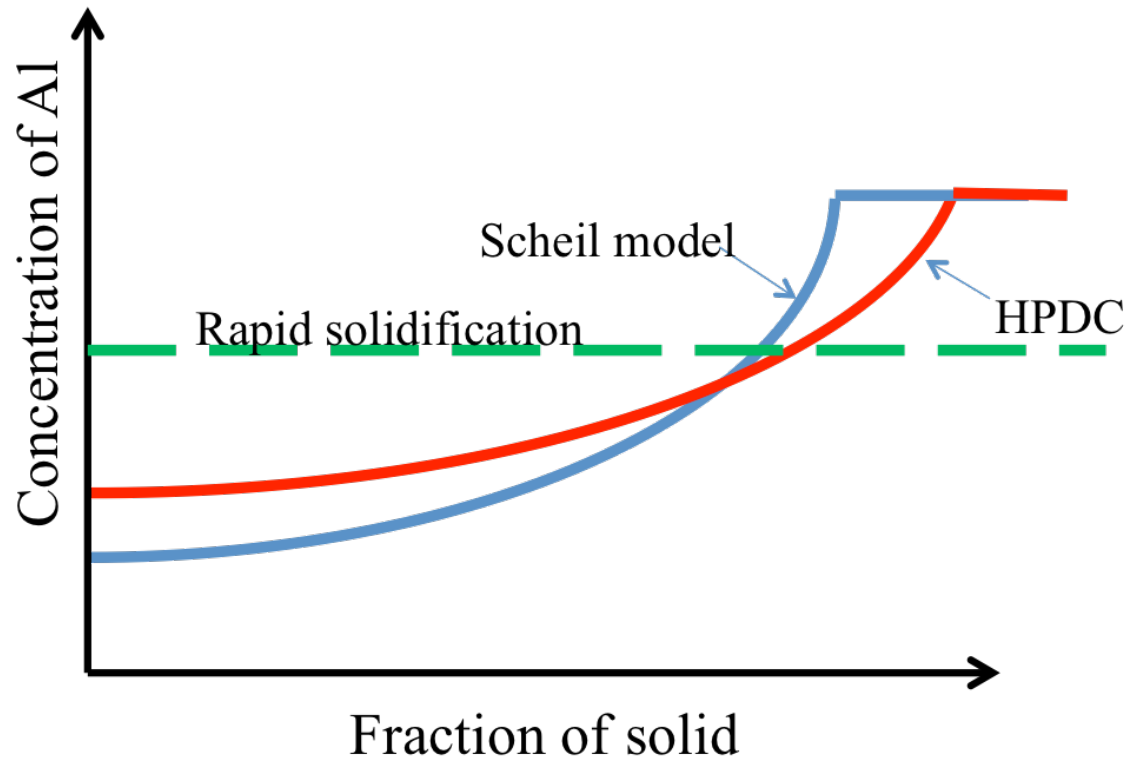
Technical Back-Up Slides

Effect of Cooling Rate on β -phase Fraction



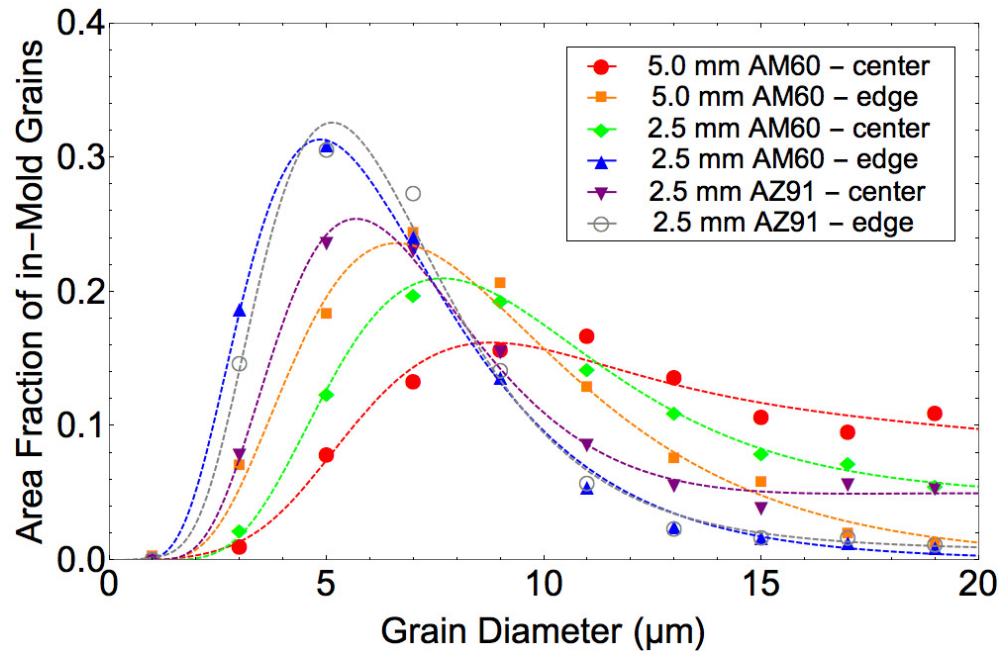
- As cooling rate increases we expect:
 - β -phase fraction to decrease
 - More Al will be trapped in α -Mg (increased fraction of SRRs) ²⁶

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

Technical Progress: Grain Size Distributions - Summary



- 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)

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