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

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University of Michigan
4/28/2017

Project ID #LM111

Overview

Timeline

- Start: October 2013
- End: October 2018
- 80% Complete

Budget

- Total project funding
  - DOE share: $600K
  - Contractor share: na
- Funding FY14: $175K
- Funding FY15: $148K
- Funding FY16: $145K
- Funding FY17: $132K

Barriers

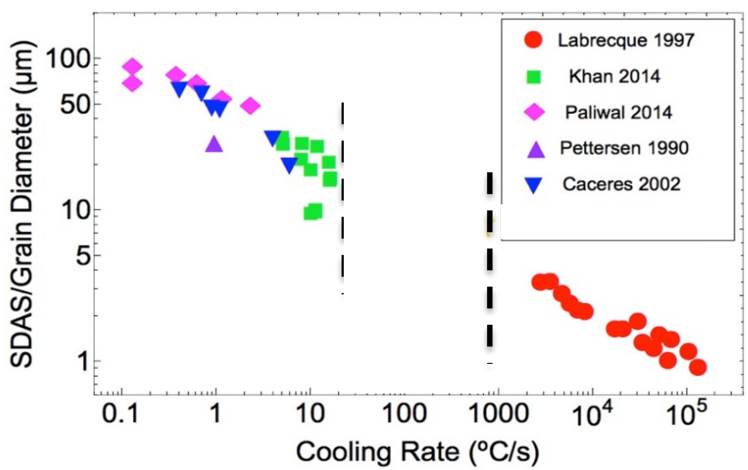
- Lack of understanding and predictive models for HPDC/SVDC Mg processes:
  - Limits ability to quickly optimize Mg components and reduce costs.
  - Limits ability to rapidly develop new alloys & processes for challenging applications and increases risk.

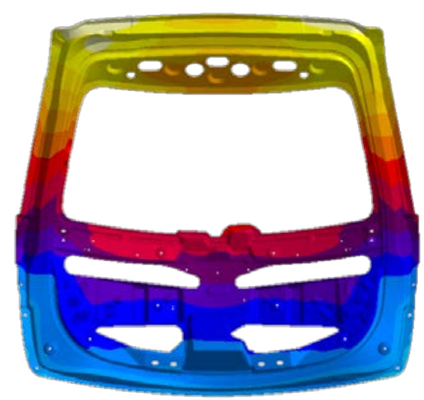
Partners

- Ford Motor Company (Dr. Mei Li)
- Lead – UM (J. Allison)

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, Presentations and Publications

Approach / Tasks

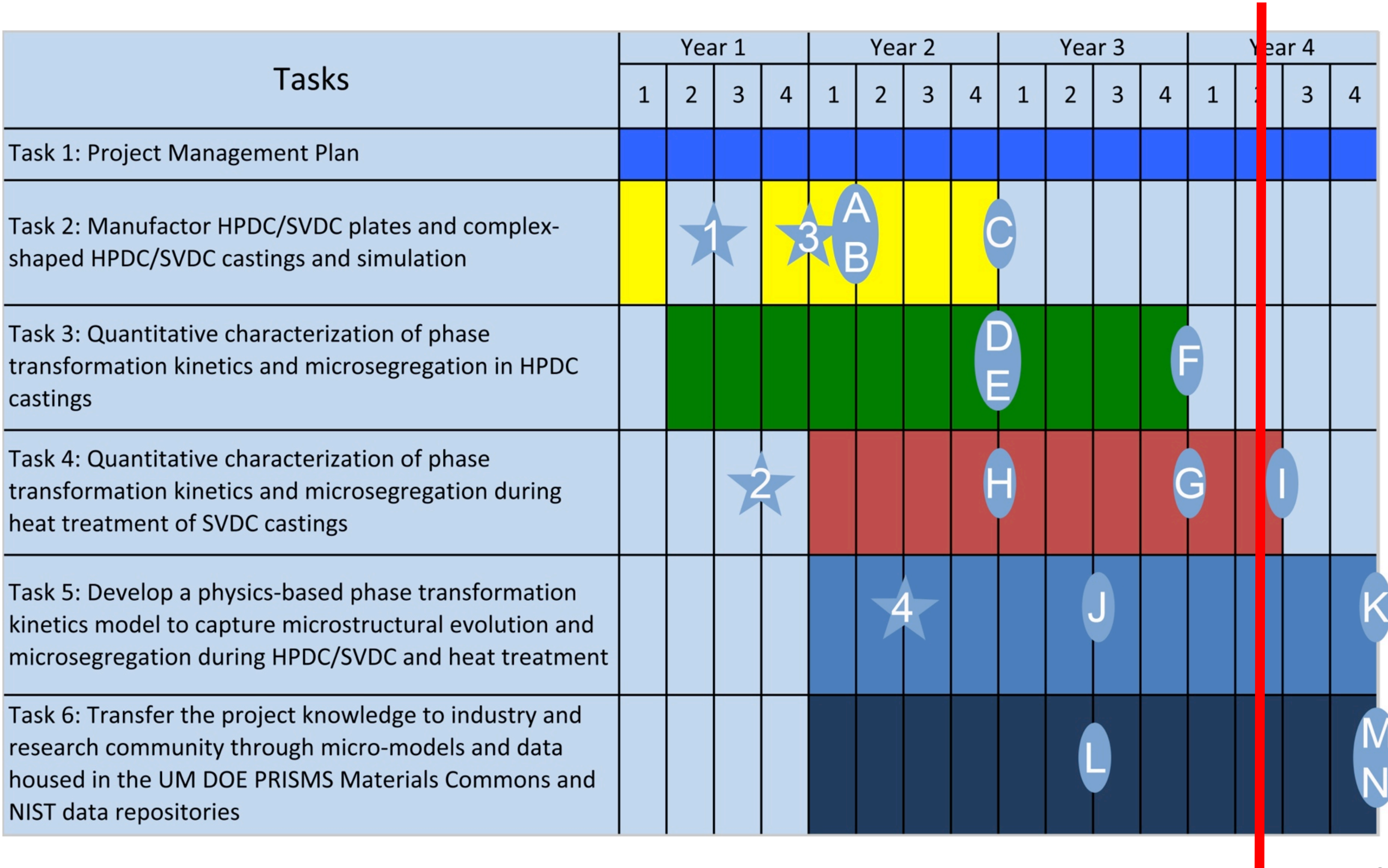
1. Simulate & manufacture high quality HPDC/SVDC plates & complex shapes of binary and ternary alloys
   – Mg-Al & Mg-Al-X; X=Mn, Zr (AM, AZ alloys)
   – Precision MagmaSoft simulation

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

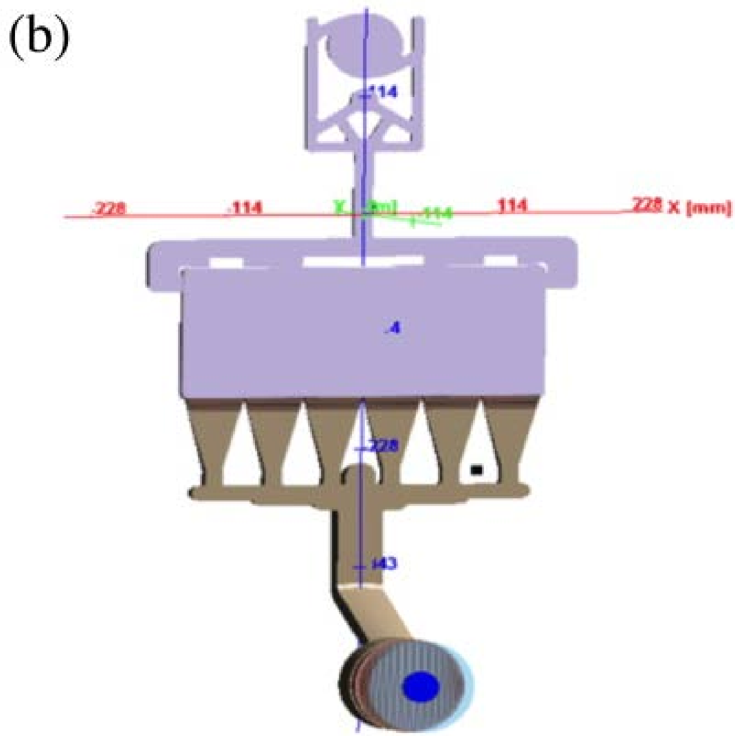
4. Develop physics-based transformation kinetics micro-models
   – Analytical model coupled with precision MagmaSoft results
   – Validate ThermoCalc model for precipitate evolution
5. Transfer knowledge through NIST D-Space Repository & UM Materials Commons

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: Manufactor 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

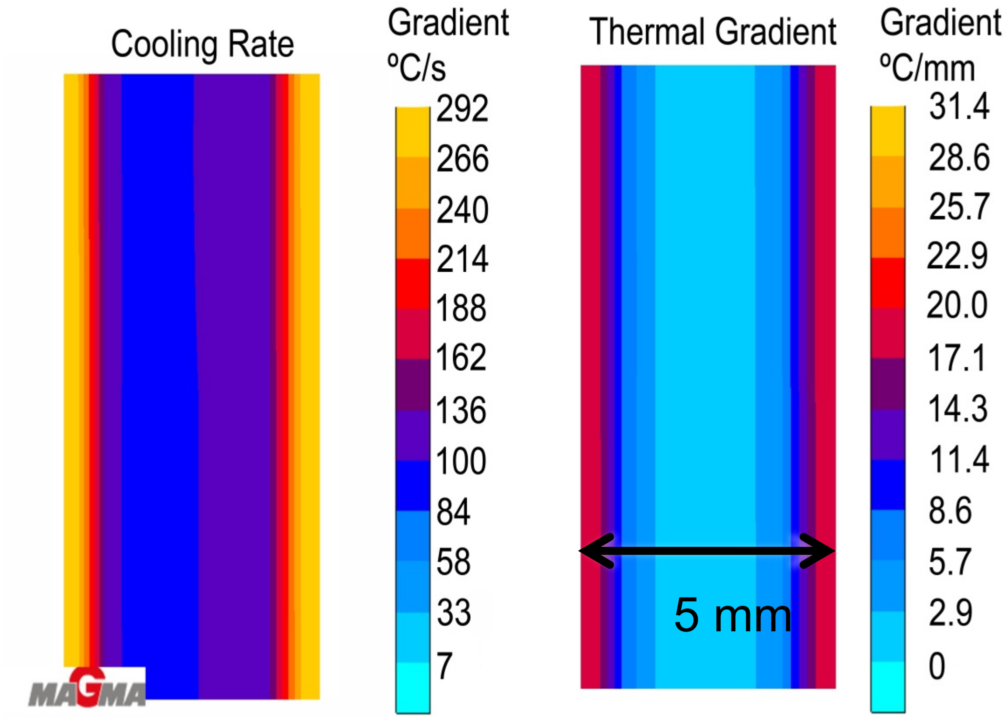
High Pressure Die Casting & Alloys

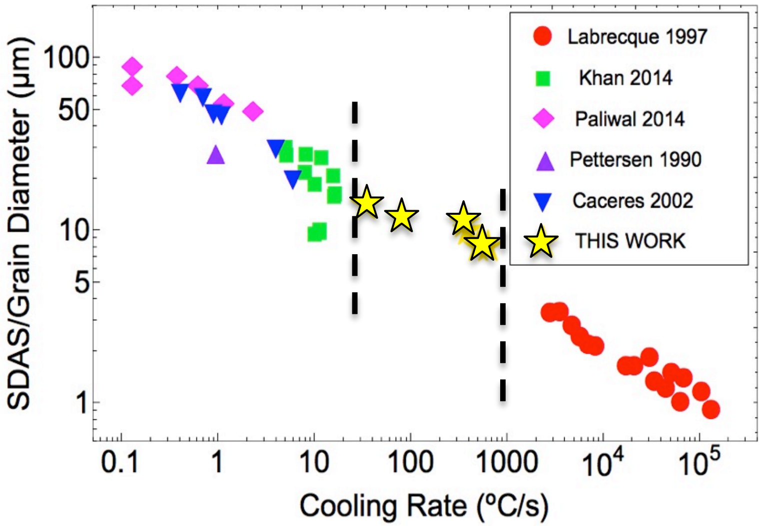
- SVDC Plate castings provided by Ford Motor Company (Mag-tec)



Alloy Compositions			
Mg	Al	Zn	Mn
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

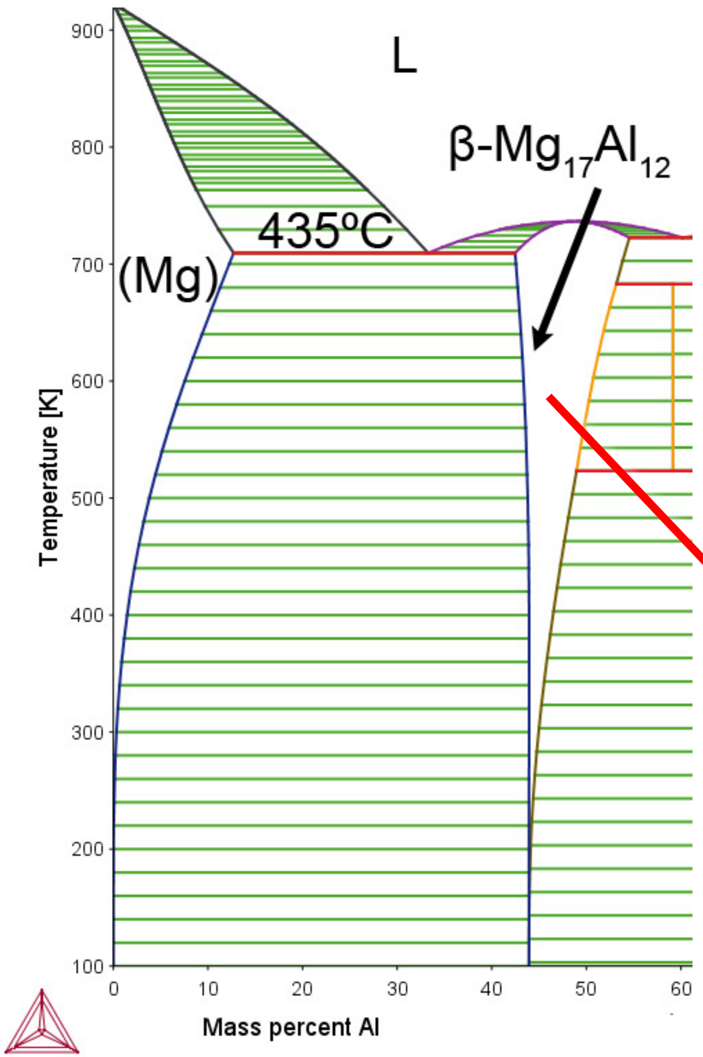
Technical Progress: Precision HPDC Simulation

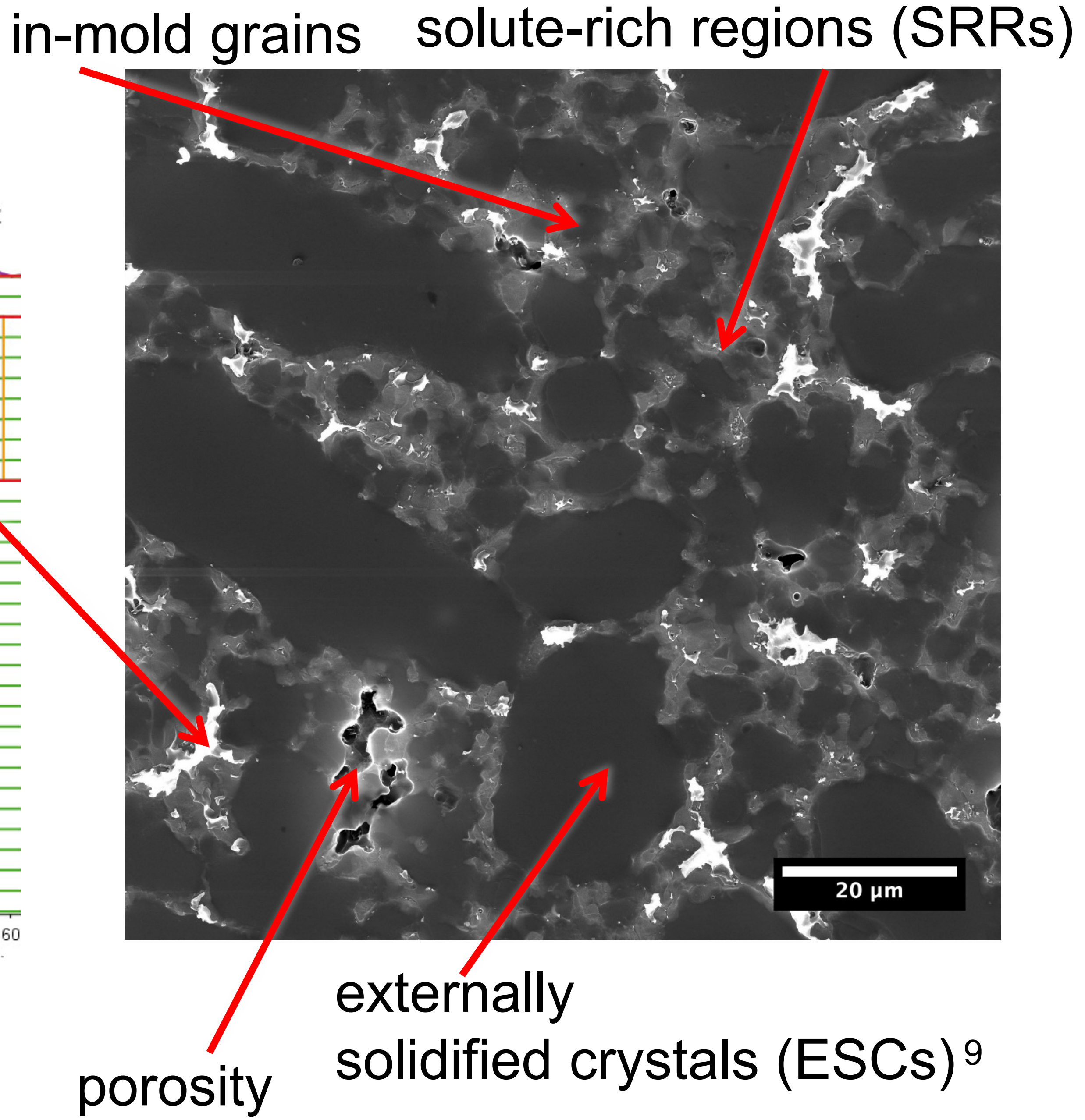




- Precision simulation achieved using advanced IHTC (from Ford/Tsinghua: 3-countries Mg Front End program) and very fine mesh
- Solidification front velocity, cooling rate (ºC/s), and thermal gradient (ºC/mm) determined as a function of location through plate
- Cooling rates ranging from 100-300ºC/s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

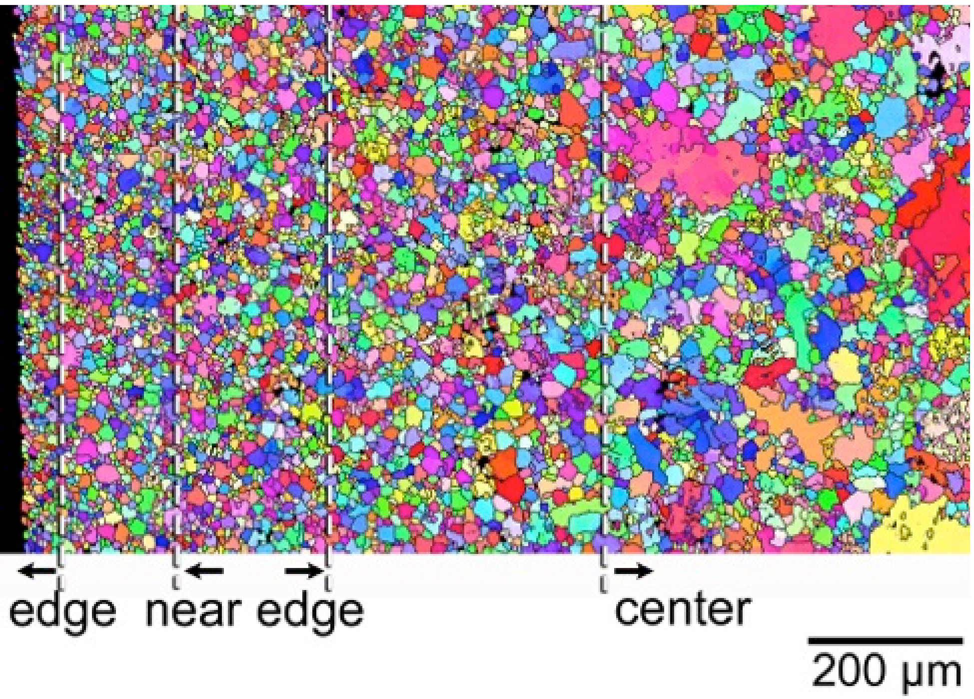
Technical Progress: Phase Quantification

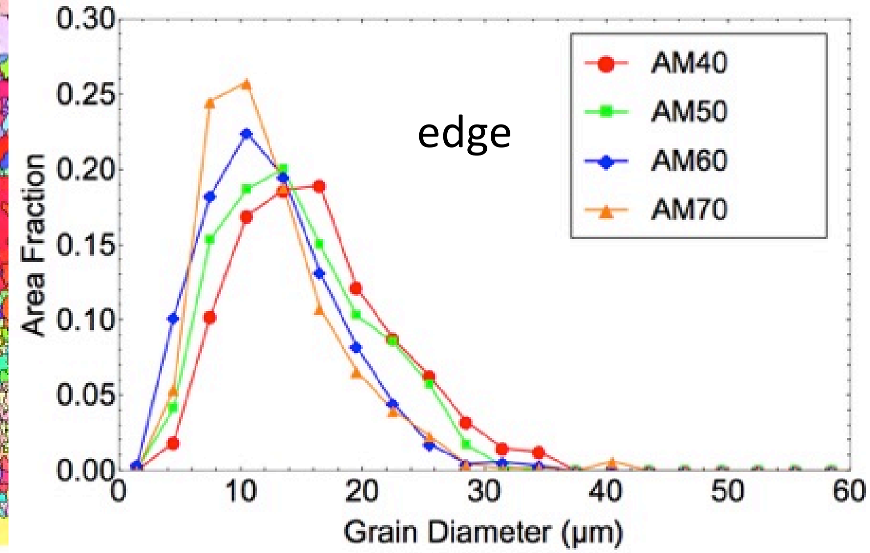


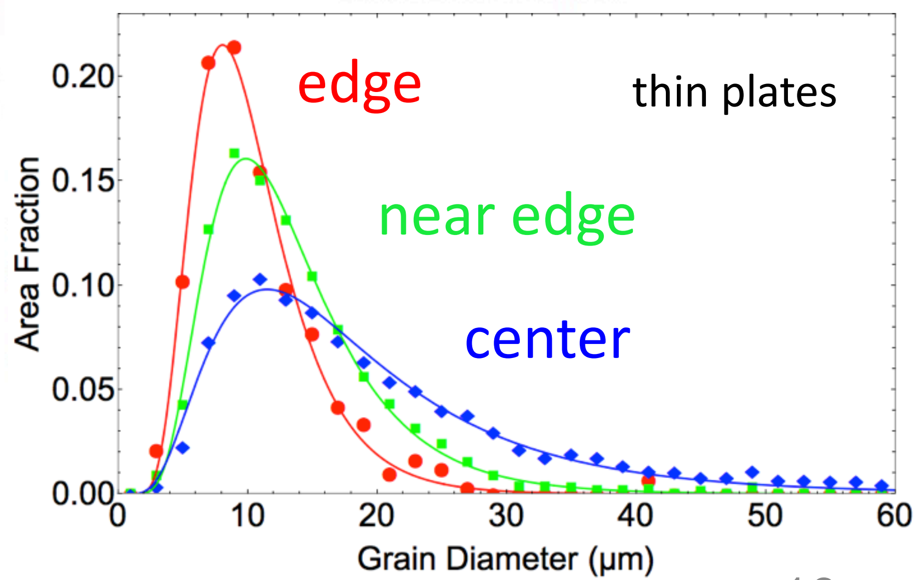


Technical Progress: Grain Size Distributions Are Location Dependent

AM50 – 2.5 mm

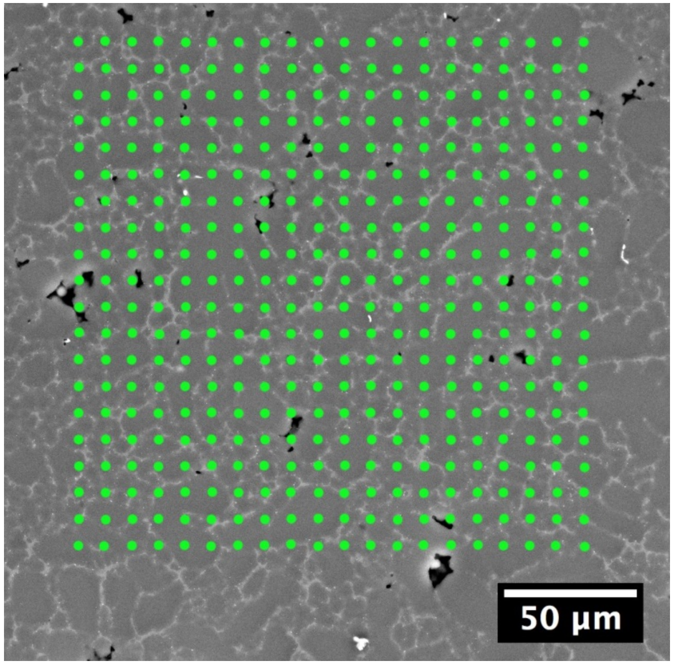


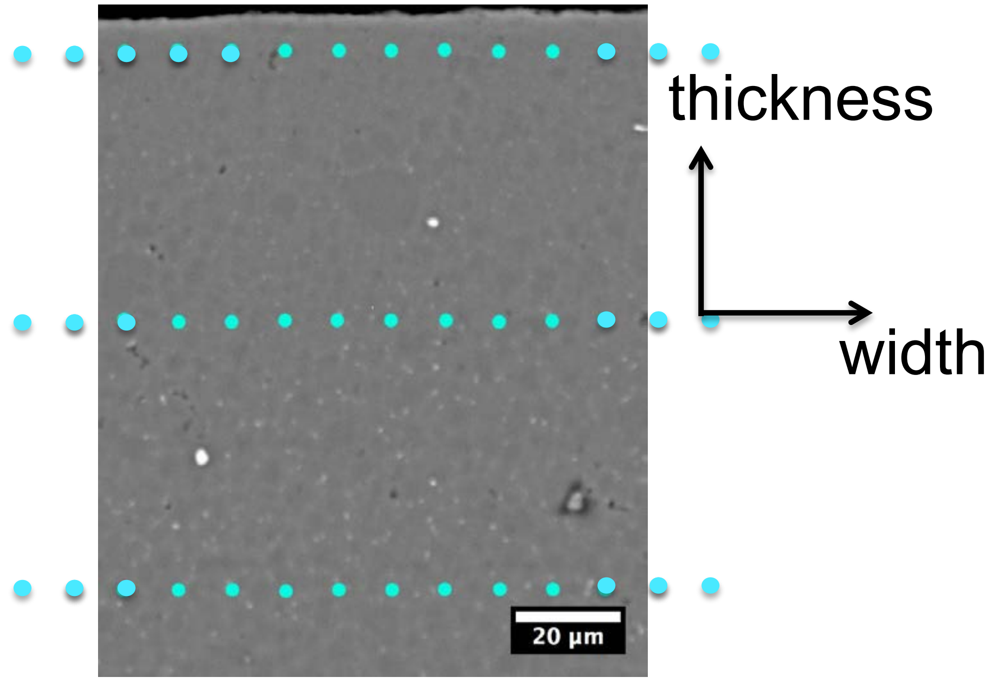




Regions of interest:
- edge (~10 µm)
- near edge (200-400 µm)
- center (>750µm) – Bimodal with ESCs.

Technical Progress: Advanced EPMA Analysis of Microsegregation





To construct segregation profile (WIRS):

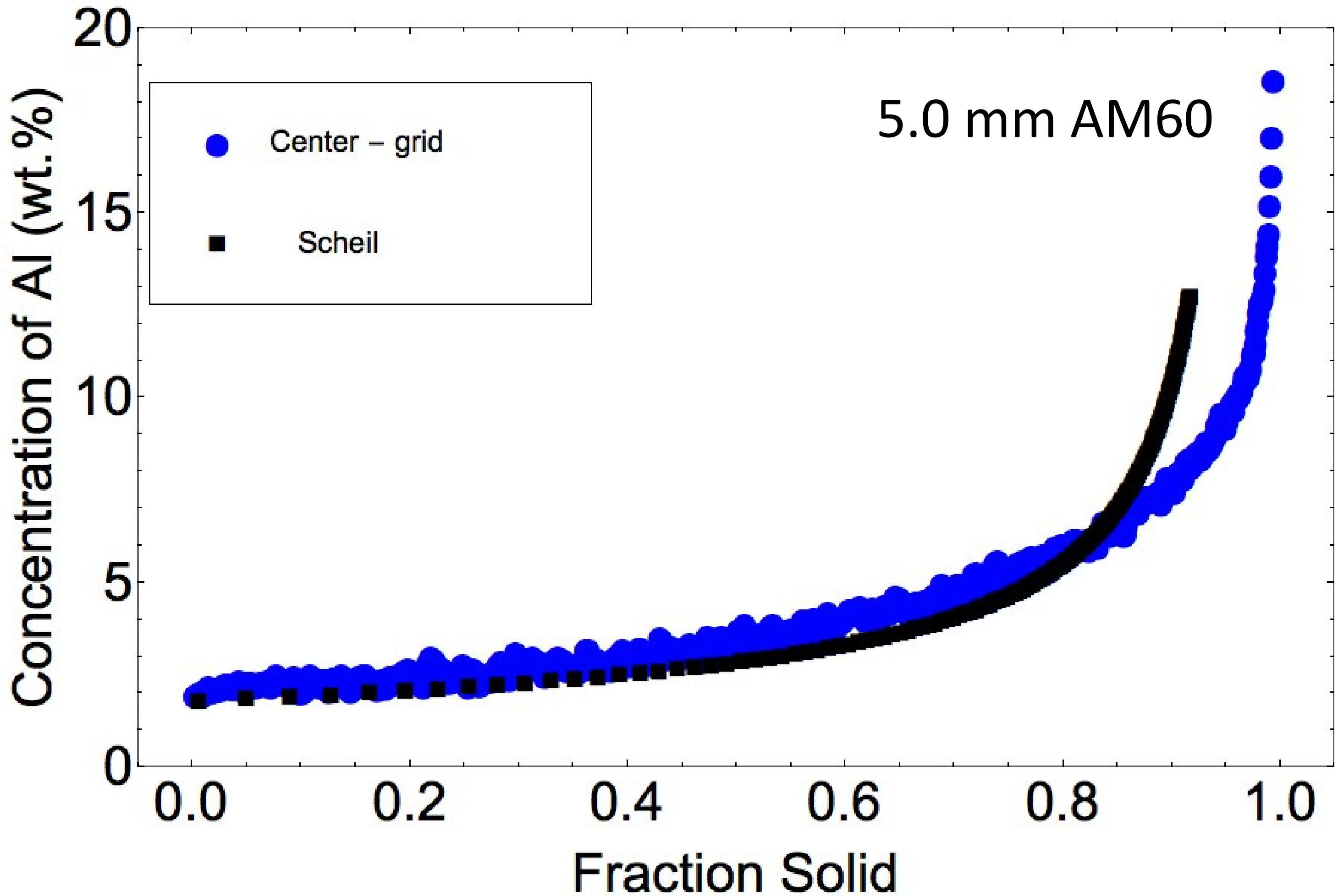
1. determine partitioning direction
2. order and rank data (Al solute)
3. assignment of fraction solid

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

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

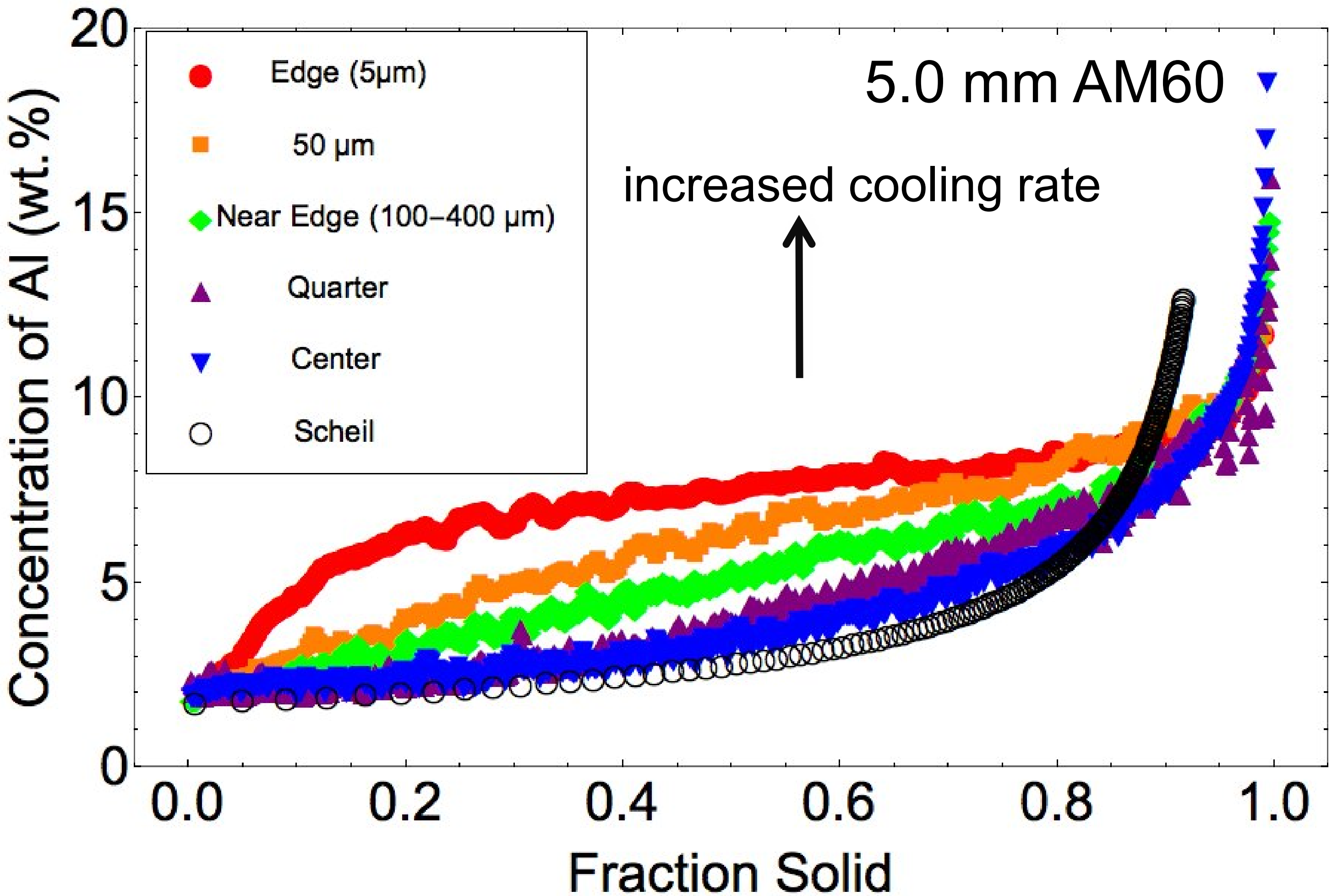
$$f_s = (R_i - 0.5)/N$$

Technical Progress: Characterizing the Microsegregation Profile



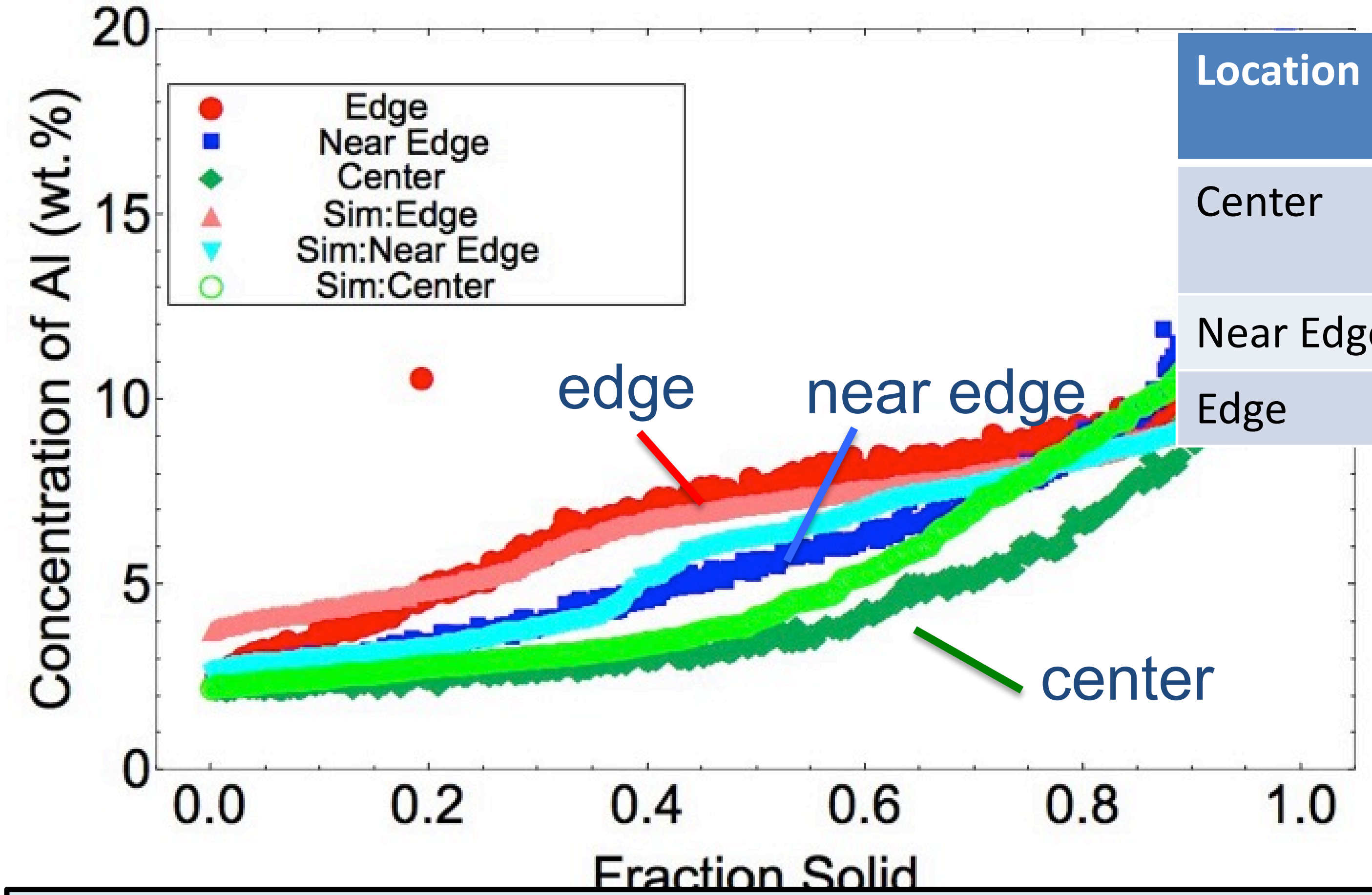
- EPMA microsegregation profile at the center of the castings is close to Scheil up to a high solid fraction

Technical Progress: Reconstructing the Microsegregation Profile



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

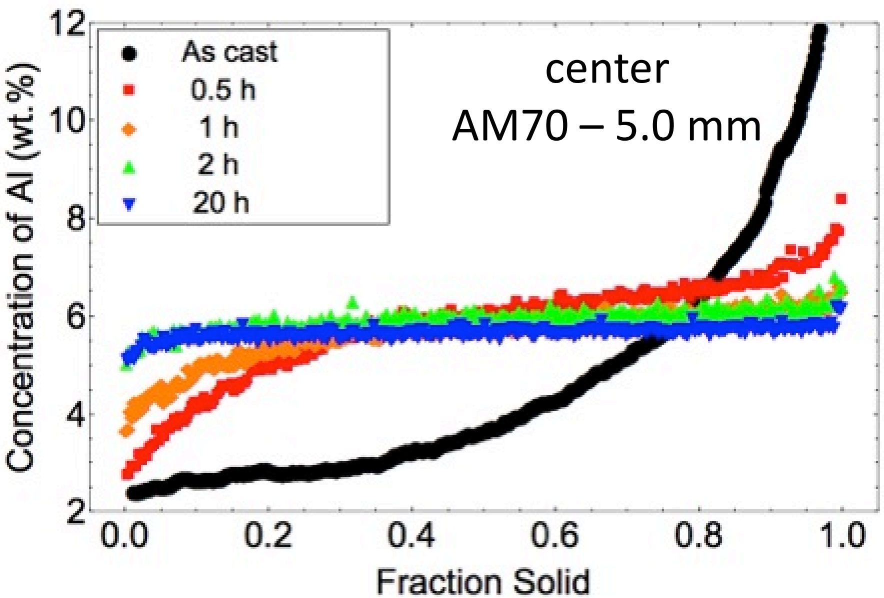
Technical Progress: Solute Trapping Behavior is Dependent on Distance from Casting Surface

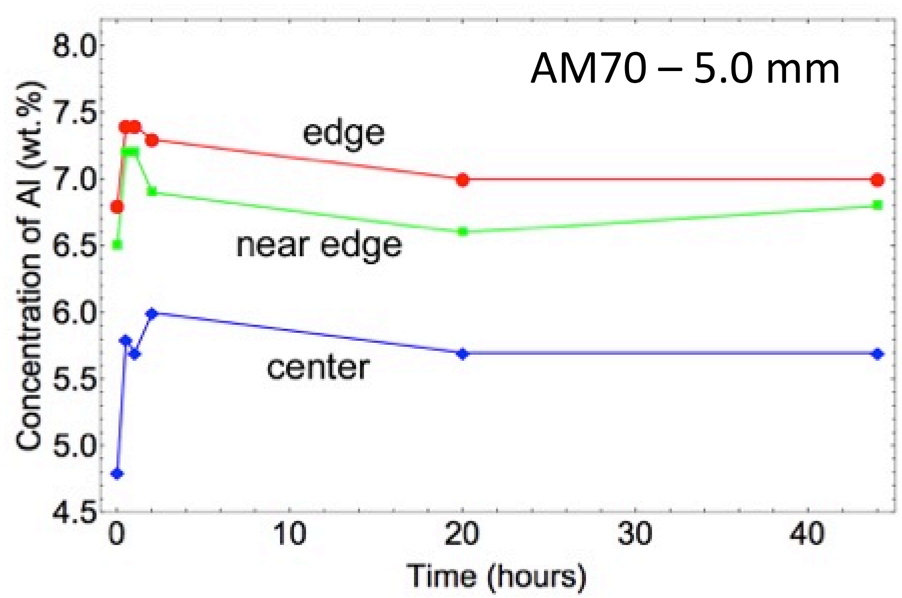


Location	Inverse modeled kv
Center	0.3 (equilibrium)
Near Edge	0.3 to 0.35
Edge	0.5

- Used forward model to estimate kv via inverse modeling
- Estimated k_v increases towards the casting surface
- Increased solute trapping leads to higher Al concentrations at the surface of the castings: macrosegregation

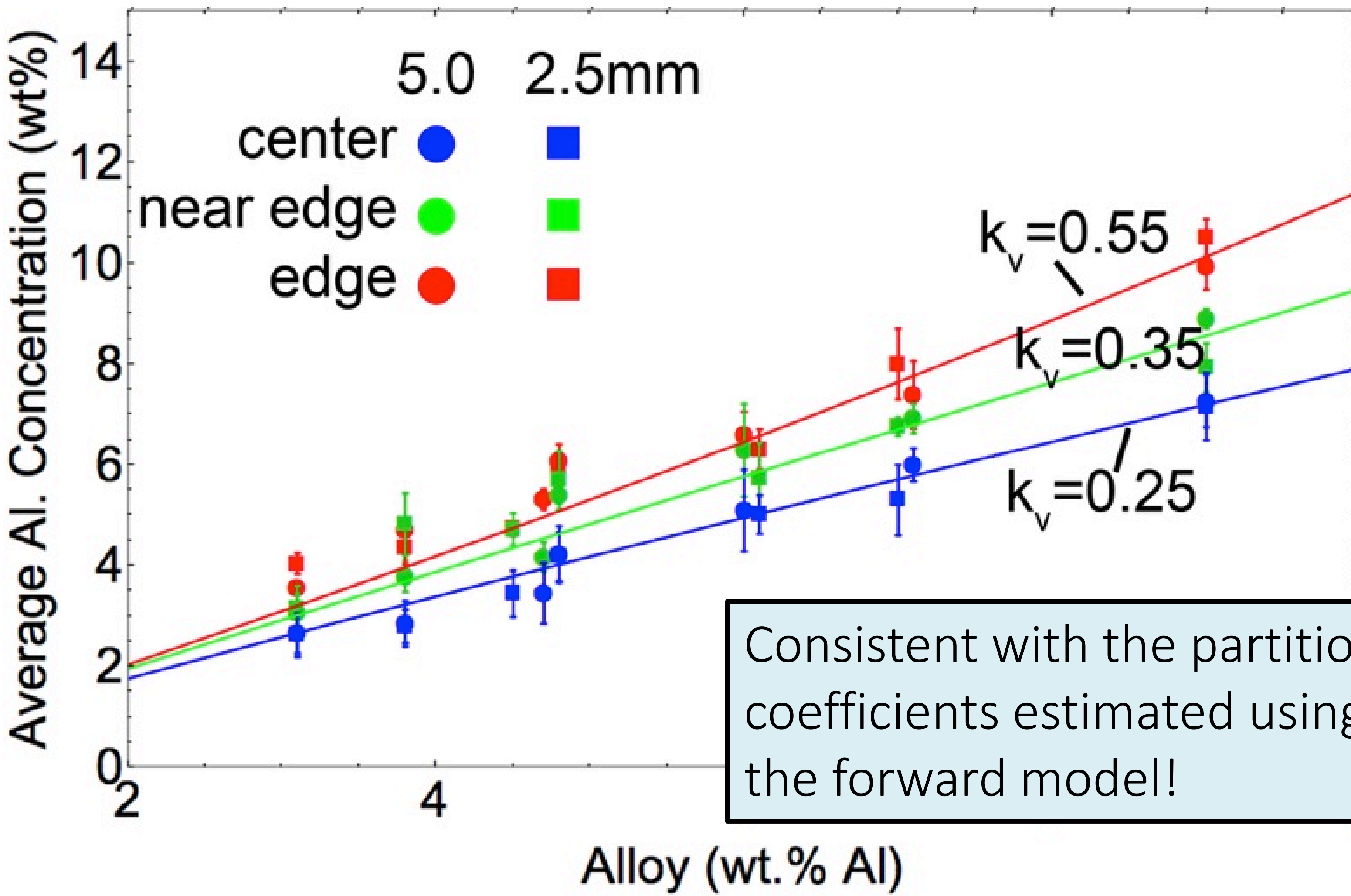
Technical Progress: Macrosegregation Persists Through Solution Treatment (420ºC)





- Microsegregation eliminated within a few hours at 420ºC
- **Macrosegregation** persists after 44 hours
  - less Al available for age hardening in the core

Technical Progress: Partition Coefficient (k_v) Can Be Determined From Macrosegregation



Consistent with the partition coefficients estimated using the forward model!

Technical Progress: Model - Solidification Rate Dependent Partition Coefficients

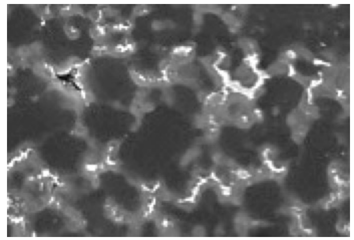
$$k_v = \frac{Bv + \kappa_e}{Bv + 1 - (1 - k_e) c_{Lv}}$$

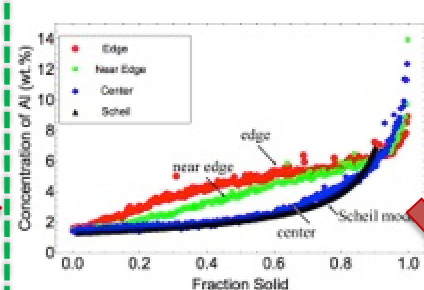
- *B:* calibration parameter
- v: solidification front velocity (MagmaSoft)
- k_e : equilibrium partition coefficient
- κ_e : partitioning parameter (dependent on k_e, C_{Se}, C_{Le})
- C_{Lv}: the solidification rate dependent liquidus concentration

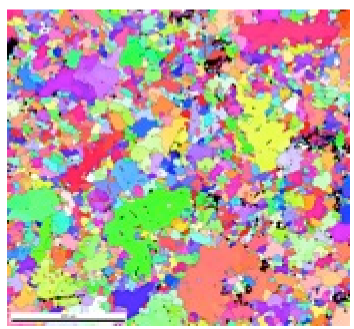
[Baker1970, Aziz1988, Carrard1992, Kraft1996]

Integrated Computational Materials Engineering (ICME) approach for HPDC Mg

Cast and quality mapping (Ford)







Quantitative characterization of microstructure and solidification segregation (UM)

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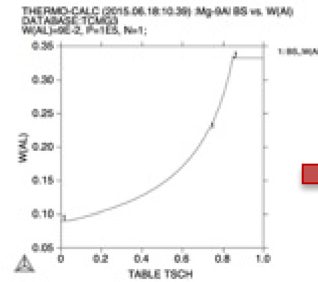
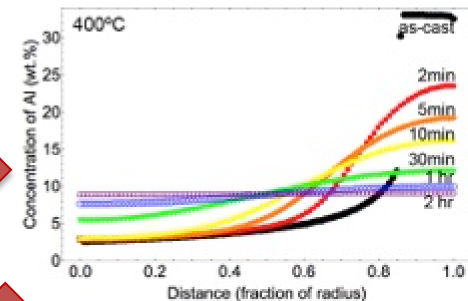
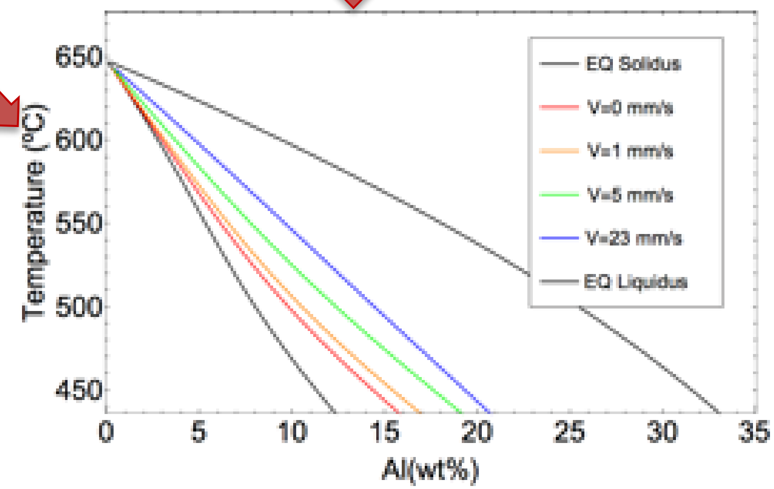
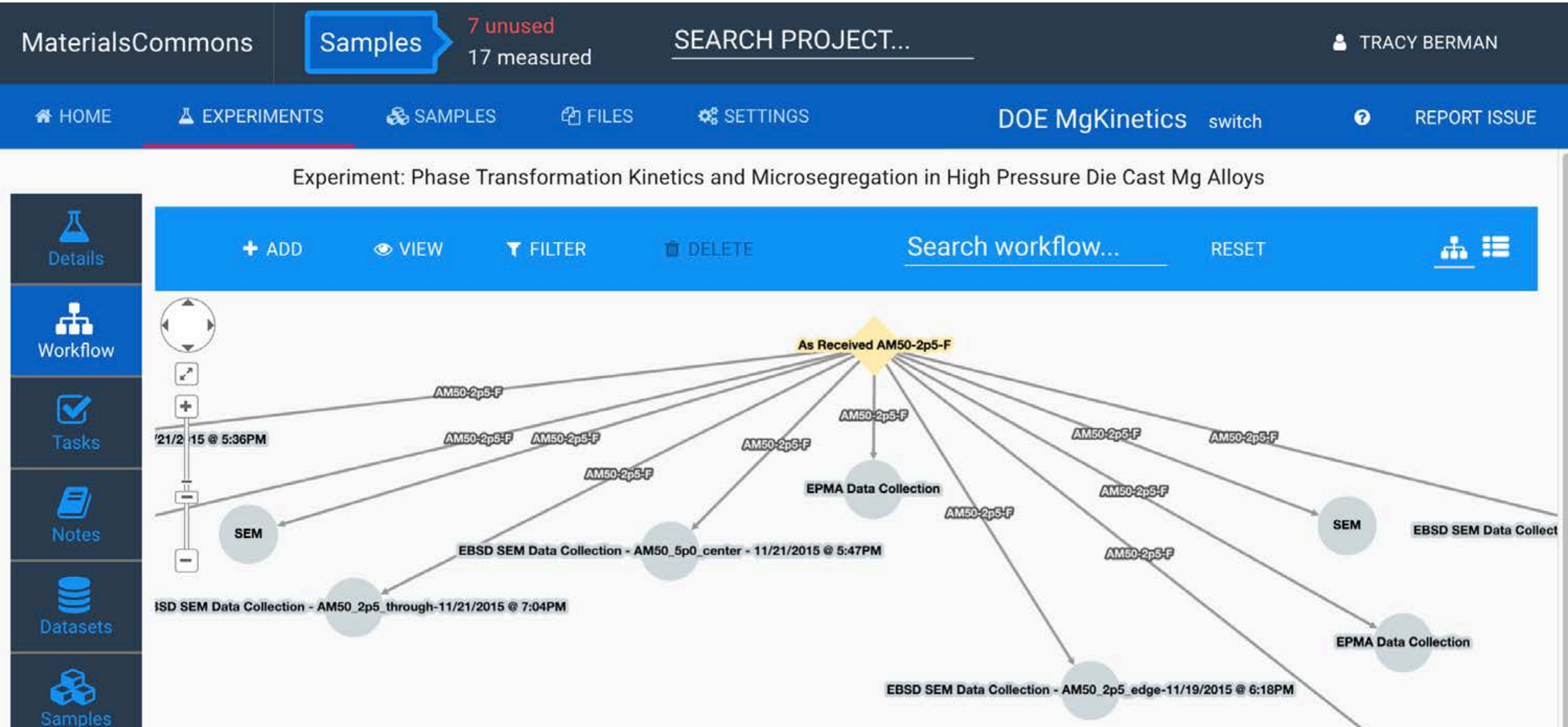






Solidification and Segregation models

Technical Progress: Materials Commons & NIST D-Space



- One dataset has been made public with a published paper
- Major dataset to be released later this summer with major publication.
- Collaboration Site: https://materialscommons.org
- Public Data Site: https://materialscommons.org/mcpub

Responses Reviewers' Comments

- NA – Not reviewed in 2016

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

- Technology Transfer: Concept and methods being used on:
  - Ford DOE-EERE Cast Al Alloy Development ICME Project (New Ford Alloys)
  - LIFT – HPDC Al ICME Project (LIFT 380 Alloy)

Remaining Challenges and Barriers

– None

Proposed Future Work

- Complete and validate development of solidification micro-model including analytical solution for thermal gradient in surface regions.

- Complete systematic characterization of microsegregation & phase transformation kinetics following solution treatment and aging of Mg-Al alloys

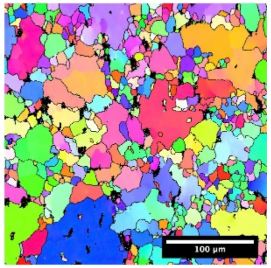
- Develop micro-models to predict microstructure & microsegregation evolution during heat treatment

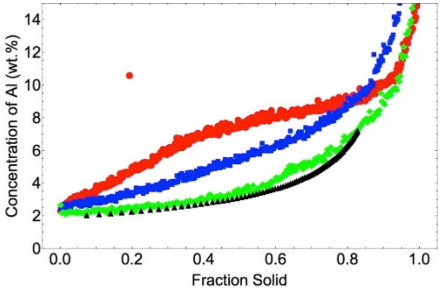
- Transfer knowledge to industry and the research community through micro-models and information housed in UM DOE PRISMS Materials Commons and the NIST DSpace Repository

Any proposed future work is subject to change based on funding levels

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 have been developed.
- Model developed for predicting microsegregation using solidification velocity dependent partition coefficients.
- Data and meta-data is being made available on Materials Commons and D-Space

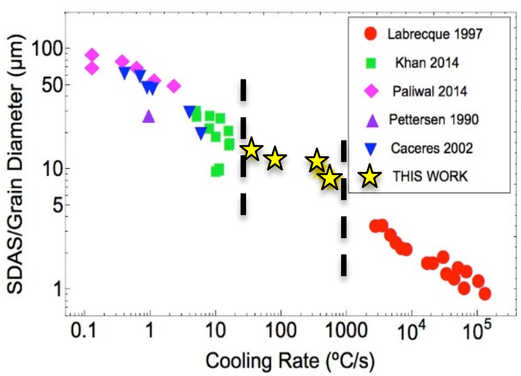


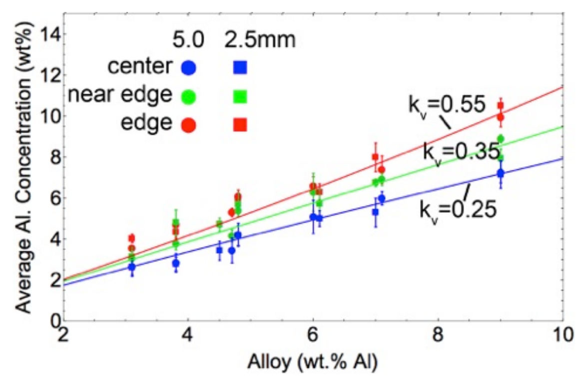


Summary - Findings

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

- Microsegregation in HPDC:
  - Discovered pronounced location dependent segregation (Skin vs Core)
  - Macrosegregation persists after solution treatment
  - Solidification segregation model captures the location dependent k_v in HPDC Mg-Al





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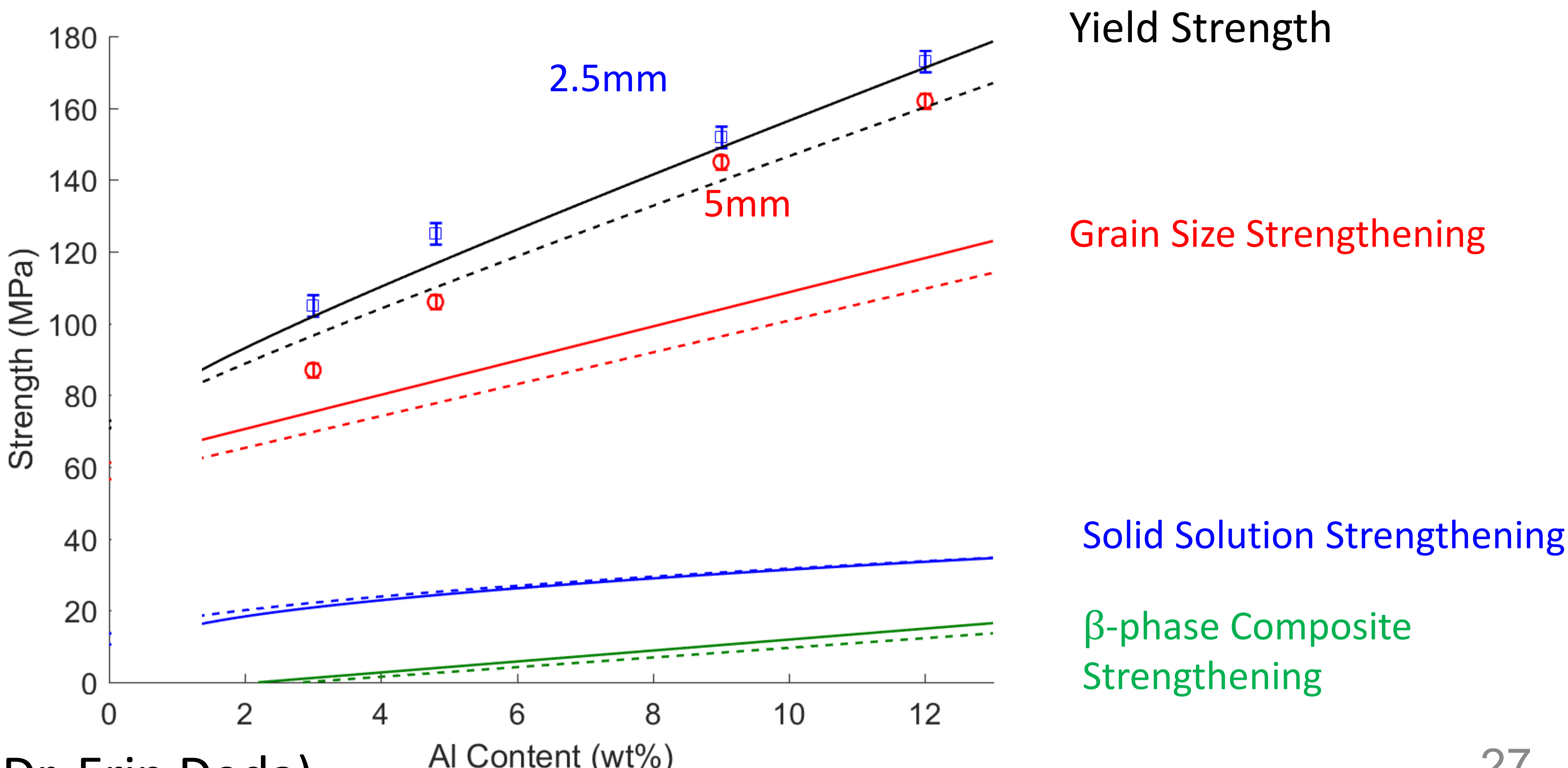
Technical Back-Up Slides

We Need Quicker Methods to Determine Material Properties!

$$YS = \sigma_0 + \sigma_{GB} + \sigma_{SS} + \sigma_{Composite}$$

$$YS_{total} = YS_{skin} * f_{skin} + YS_{core}(1 - f_{skin})$$

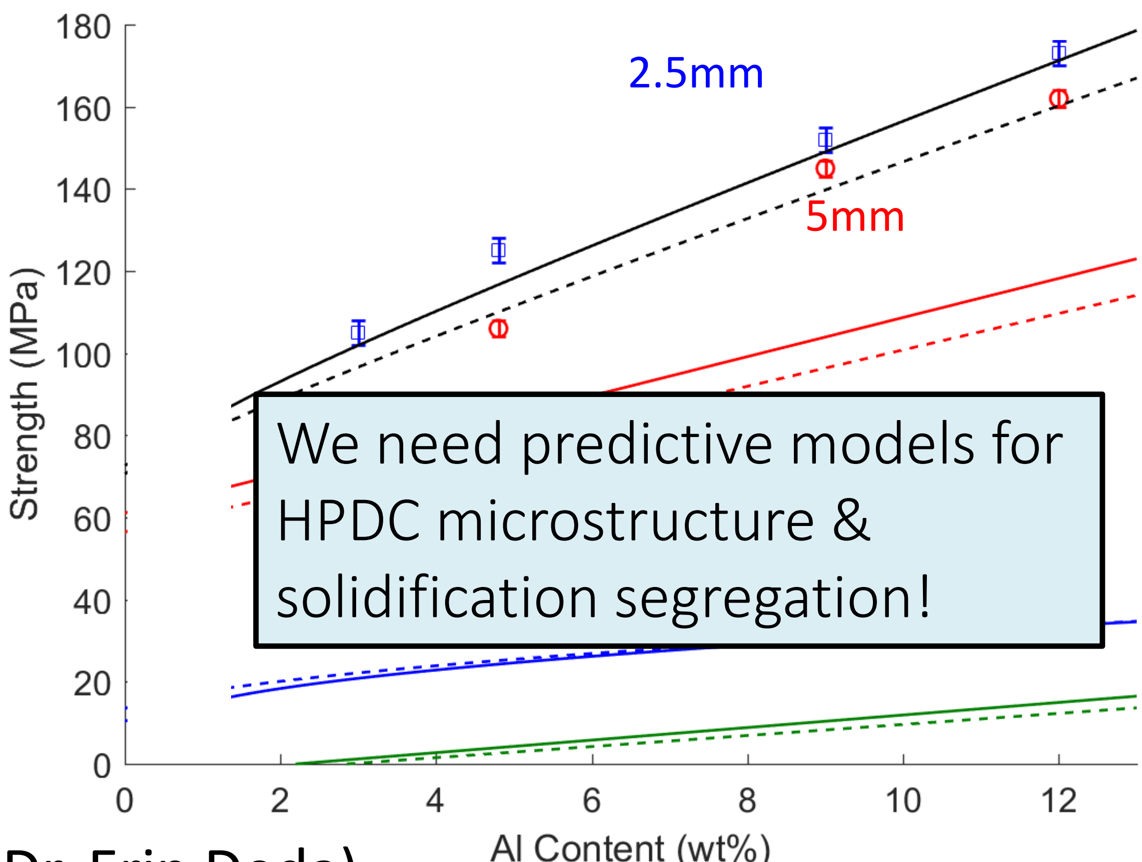


(Dr. Erin Deda)

We Need Quicker Methods to Determine Material Properties!

$$YS = \sigma_0 + \sigma_{GB} + \sigma_{SS} + \sigma_{Composite}$$

$$YS_{total} = YS_{skin} * f_{skin} + YS_{core}(1 - f_{skin})$$



We need predictive models for HPDC microstructure & solidification segregation!

For Each:
Alloy
Casting Thickness
Location (skin/core)

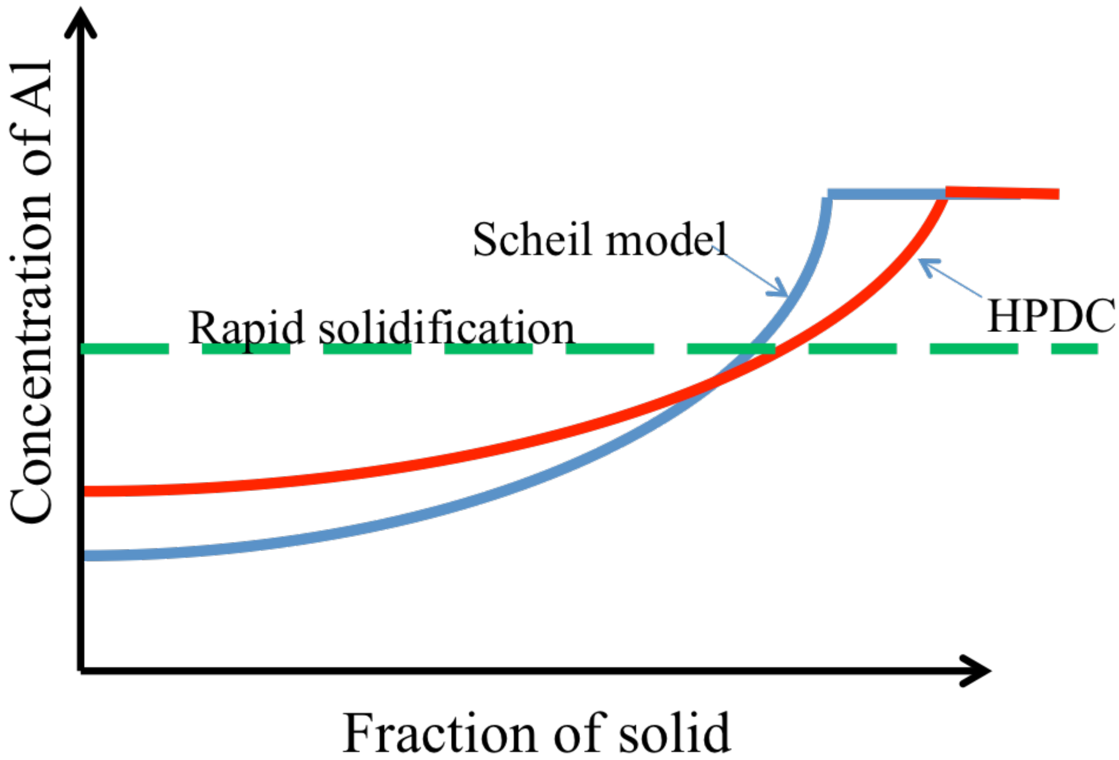
Grain Size:
Electron backscatter diffraction (EBSD)

Solute Content:
Electron Probe MicroAnalysis (EPMA)

β-phase:
Quantitative SEM

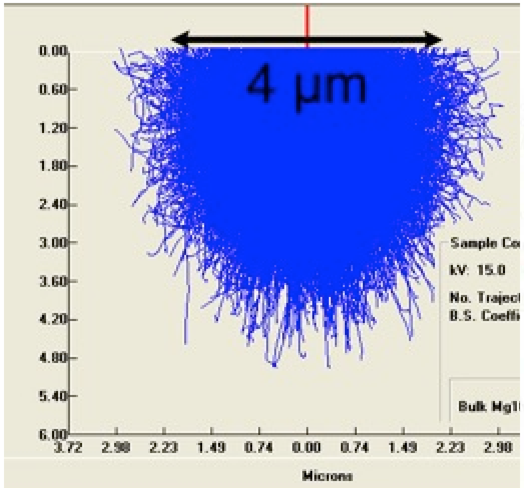
(Dr. Erin Deda)

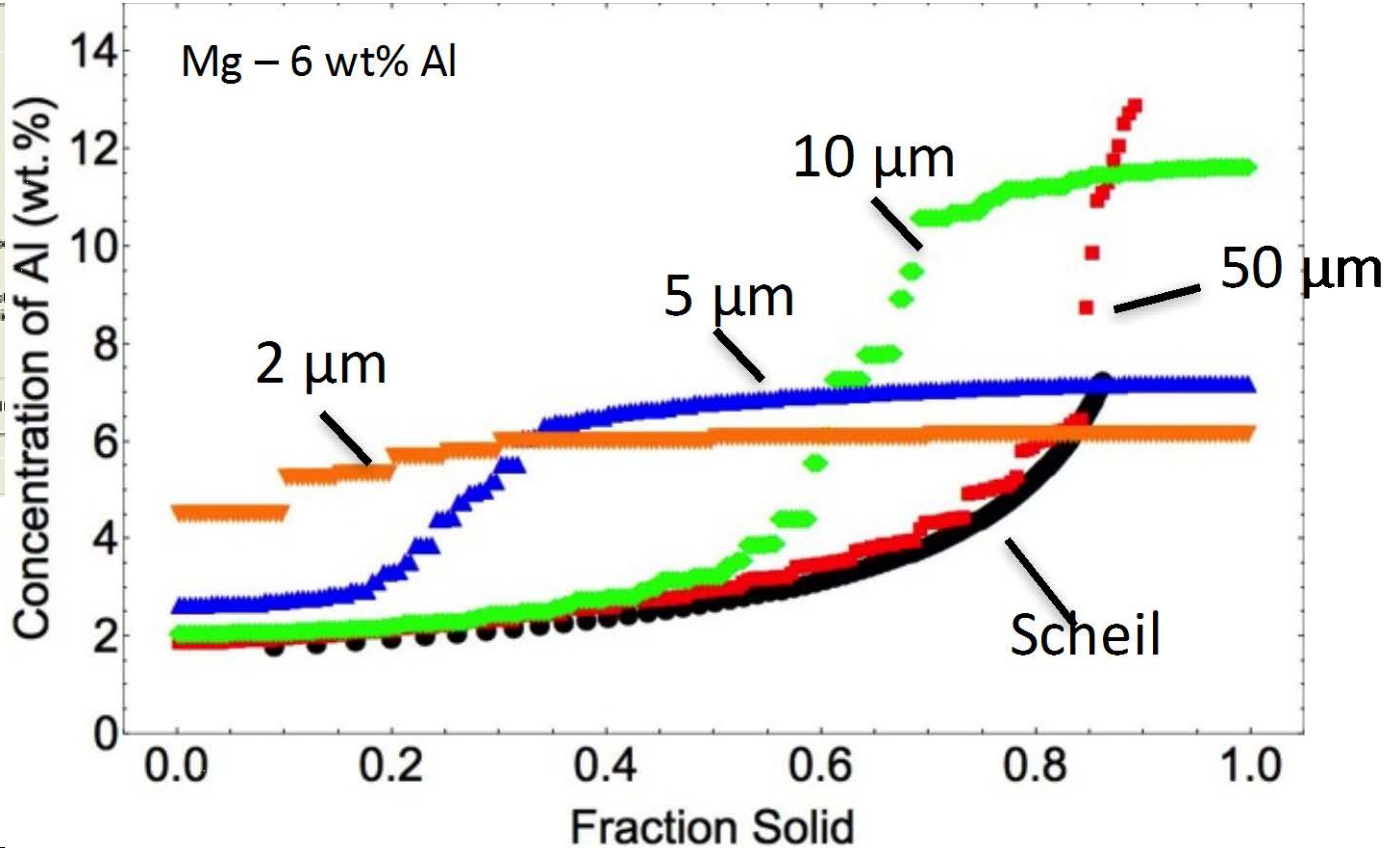
Developing a HPDC Solidification Model



- 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

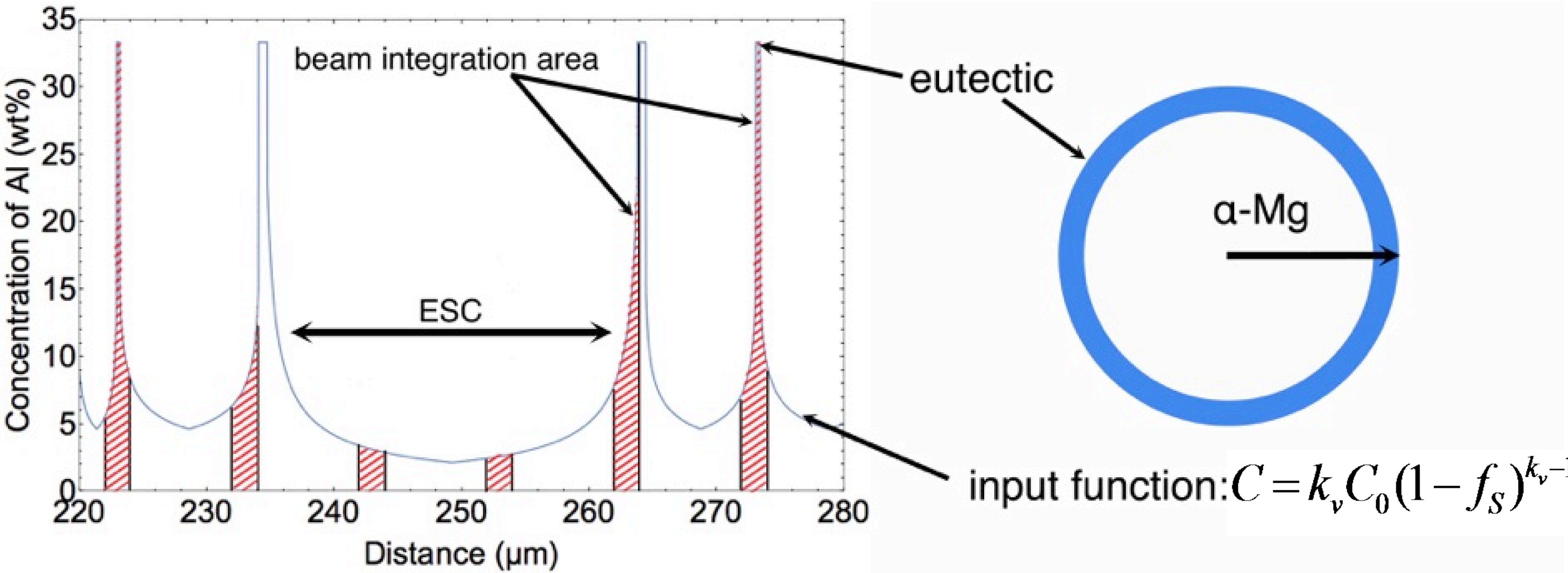
Technical Progress: Model Accounts for Influence of Beam Size





- If grain size >> beam size, you can measure the true microsegregation profile with EPMA
- grain size ~ beam size, the microsegregation profile becomes smeared out
- grain size < beam size, composition begins to appear uniform

EPMA Forward Model Simulation: Input



Forward Model Variables	Value Used
Eutectic Fraction	1/3 predicted by Scheil (ThermoCalc®)
Grain size distributions	from EBSD data, function of thickness & location
ESCs	grains larger than 30 μm; microsegregation according to Scheil

A composition measurement is simulated by integrating over the beam width

Model: Solidification Rate Dependent Partition Coefficients

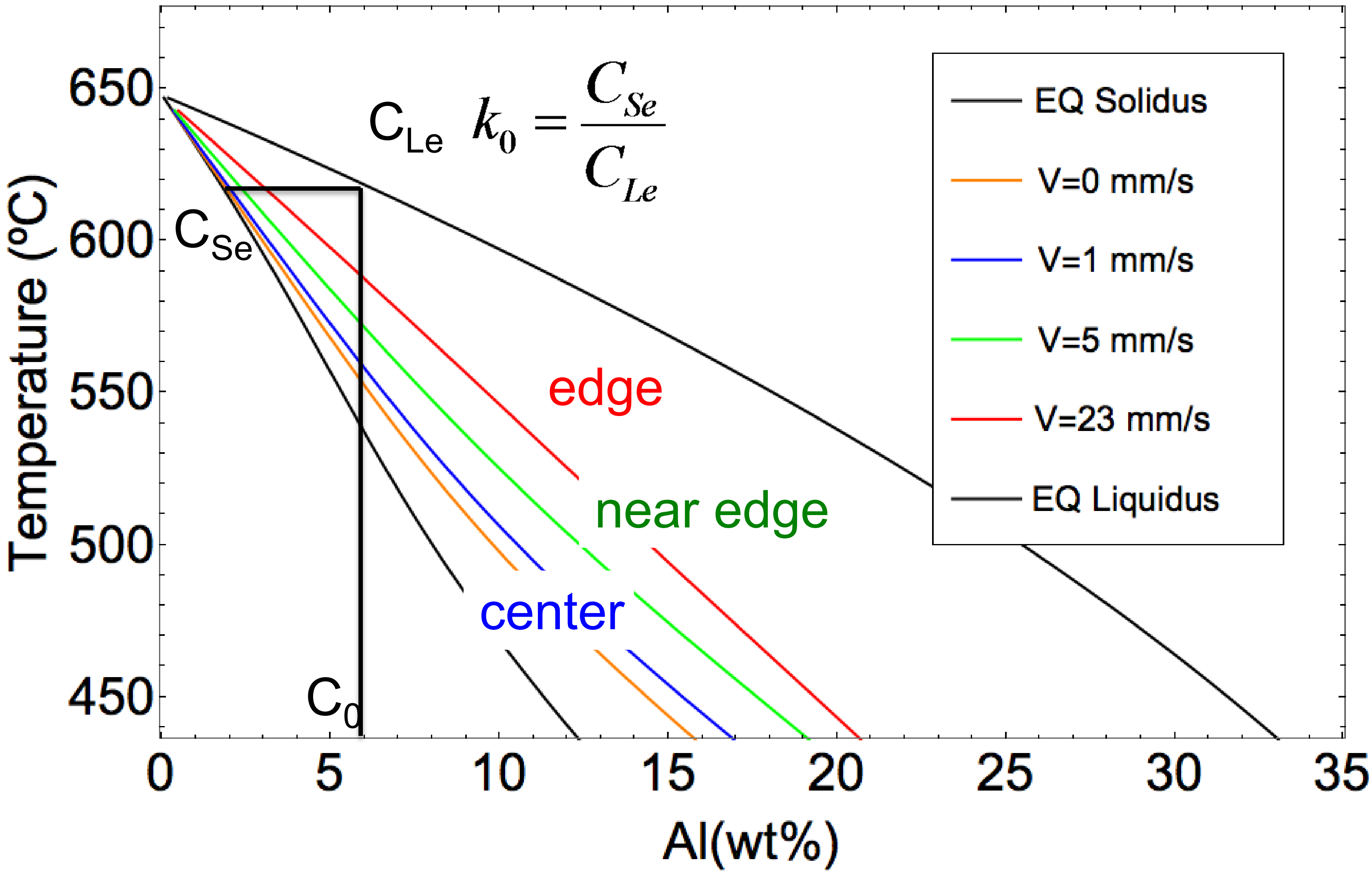
$$k_v = \frac{Bv + \kappa_e}{Bv + 1 - (1 - k_e) c_{Lv}}$$

$$c_{Lv} = c_{Le} - \frac{v}{v_0} \frac{1}{1 - k_e} \left(\frac{1 - k_e}{1 - k_v + \left[k_v + (1 - k_v) \beta \right] \ln \left(k_v / k_e \right)} \right)$$

- v_0: calibration parameter, related to the speed of crystallization
- β: solute drag (0 = no, 1 = yes)

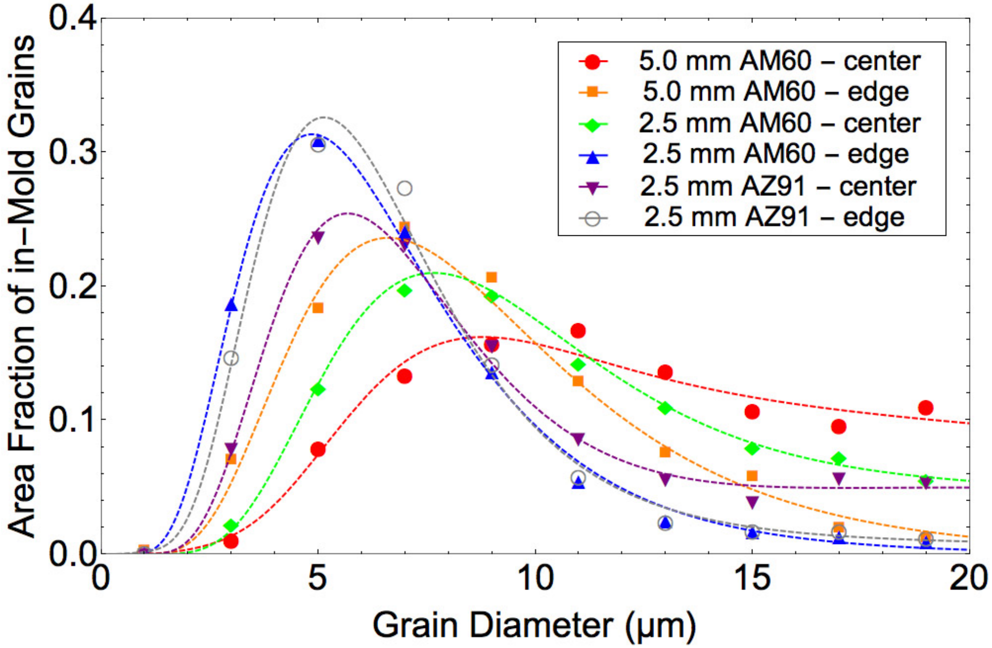
[Baker1970, Aziz1988, Carrard1992, Kraft1996]

Effective Solidus Curves for HPDC Mg-Al



- Assumes "dilute alloys"

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