Development of Advanced High Strength Cast Alloys for Heavy Duty Engines

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Caterpillar Inc.

Vehicle Technologies – Annual Merit Review
June 10, 2015

Project ID: PM 059

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CATERPILLAR® QuesTek® INNOVATIONS LLC *Materials By Design®* UAB THE UNIVERSITY OF ALABAMA AT BIRMINGHAM Argonne NATIONAL LABORATORY

Overview

Timeline

- Project start - December 2012
- Project end - December 2016
- Percent complete ~ 50%

Budget

- Total project funding: $5.08M
  - DOE share: $3.48M
  - Contractor share: $1.6M
- Expenditure of Gov't Funds:
  - FY2013: $524,942
  - FY2014: $816,519
  - FY2015: $334,390 through Mar.

Barriers

- Efficiency: material and process must achieve 214 MPa endurance limit to enable higher cylinder pressure and temperature combustion regimes.
- Power Density: achieve 25% increase in strength over A842 compacted graphite iron.
- Cost: no more than 110% of production A48 gray iron cast units

Partners

- Interactions/ collaborations
  - **QuesTek Innovations**
  - **University of Alabama at Birmingham**
  - **Argonne National Laboratory**
  - Northwestern University
  - Jonkoping University
  - Elkem
- Project lead – Caterpillar Inc.

Objectives

- New high-strength ferrous materials with at least 25% improvement in component strength relative to components made with A842 (Compacted Graphite Iron).
- Cost should not exceed 120% of the cost of components using A48 (Gray Iron).
- Material must be produced using sand or investment casting processes.
- Evaluate the performance of existing ICME codes to accelerate the development of new alloys and processing techniques.
- Develop comprehensive cost models demonstrating costs relative to established grey cast iron baselines and identifying a path to meet incremental cost targets.
- *Contractor specific requirement:* achieve fatigue endurance limit of 214 MPa.

Relevance

- Advanced materials that are lighter and/or stronger are essential for boosting the fuel economy and reducing emissions of modern vehicles while maintaining performance and safety.
  - Increased powertrain efficiency can be obtained by enabling engine components to withstand the high pressures and temperatures of high efficiency combustion regimes.
  - Offset weight penalties from advanced emissions-control equipment, safety devices, integrated electronic systems and power systems such as batteries and electric motors for hybrid, plug-in hybrid, or electric vehicles.
    - For example, using lighter and/or higher strength materials to achieve a 10% reduction in vehicle weight can result in a 6% – 8% fuel-economy improvement.
- Cost penalties need to be minimized to accelerate adoption by industry and creating consumer demand.
- ICME tools need to be developed and utilized to accelerate to the design and validation of new materials.

Milestones

- FY13 and FY14

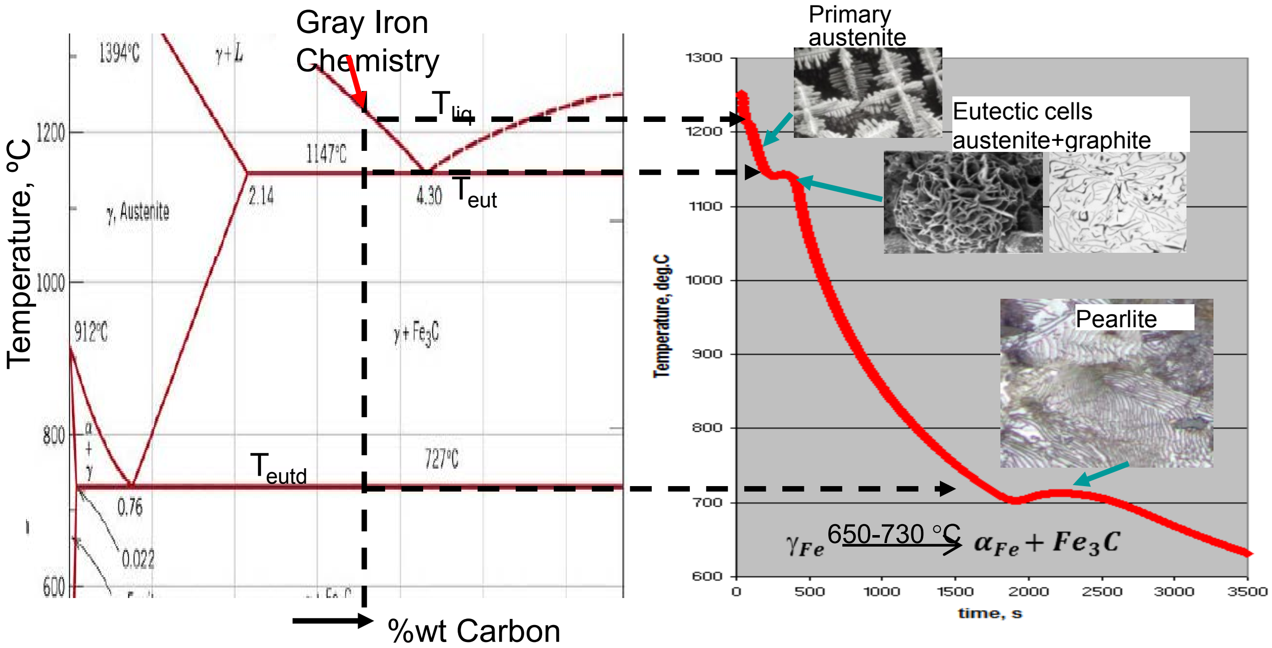
Milestone	Measure	Date	Status
Updated Project Management Plan + Reporting	Monthly + Quarterly	Ongoing	Ongoing
Definition of Alloy Requirements	Performance Requirements	Mar. '13	Complete
Generate Alloy Design Concepts	4 main areas of investigation	Aug. '13	Complete
Design Prototype Alloy Concepts	~16 prototype concepts	Dec. '13	Complete
Machinability Baseline for Current High Strength CGI	Tool wear & cutting force response surface for 450 MPa CGI, milling + drilling	Dec. '13	Complete
Design and Produce Prototype Castings	~ 16 prototype casting samples	Dec. '14	Complete
Evaluate Material Properties of Prototype Casting Alloys	> 100 KSI tensile, 140 – 245 HB	Mar '15	Complete
Refine Design of High Potential (HP) Alloy Concepts (Iteration 2)	~4 HP alloy concepts	Apr. '15	Ongoing
Produce Prototype Castings for HP Alloy Concepts	~4 HP alloys	Jun. '15	Ongoing
Evaluate Mechanical Properties of HP Alloy Concepts	> 100 KSI tensile, > 31 KSI fatigue limit	Jul. '15	
Evaluate Castability of HP Alloys	Minimize difference from HSGI, fluidity & Hot Tear	Aug. '15	

- FY15 – evaluate HP alloys, down-select and optimize final alloy design
- FY16 – validate final alloy design, demo component design
- FY17 – variation analysis, cost model

Approach

- Utilize an Integrated Computational Materials Engineering (ICME) approach that has been proven to accelerate alloy development time by applying mechanistic materials models within a systems-engineering framework to computationally engineer new material compositions and manufacturing processes.
- Prototype melts will be produced and characterized iteratively for an alloy design within a stage-gate process.
- Standard characterization and material testing will be done to validate the alloy performance against goals and provide feedback to ICME models.
- Utilize the Advanced Photon Source (APS) at Argonne National Labs to conduct innovative measurements to map 3D graphite networks and identify nucleation sites. In-situ measurements of phase evolutions and damage during heating and cooling under various loading conditions is also planned.
- Utilize an integrated modeling approach that simulates the material, processing, and performance to optimize the design of a concept engine component to demonstrate the potential benefits of the new material.
- Cost models analyzed at the material and the component/system level as a function of annual production volumes.

Introduction - Microstructure of Cast Iron



Relevant features:

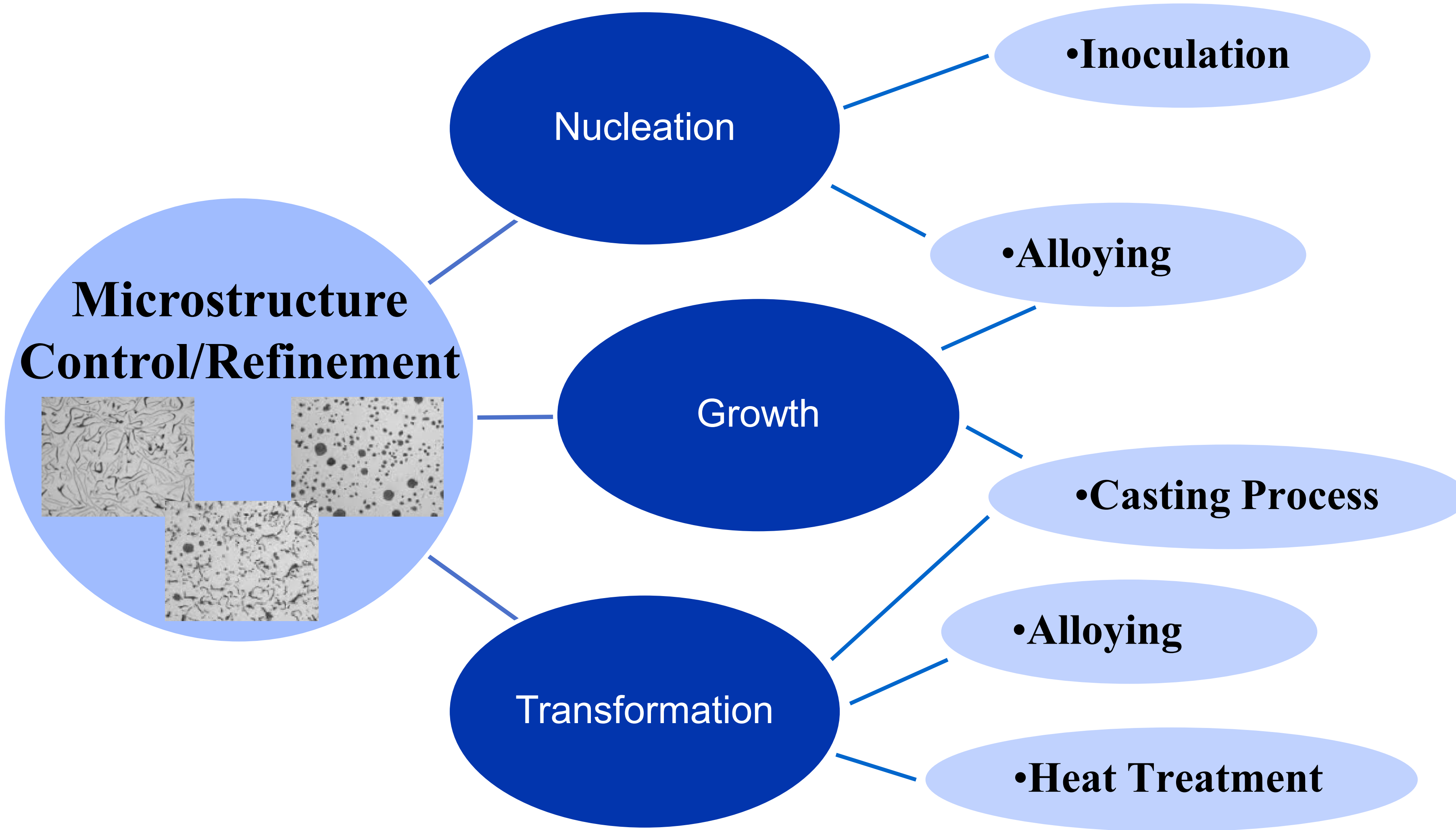
- **Solidification Stage:**
  - ***austenite*** (primary & eutectic)
  - ***graphite***

- **Room Temp.:**
  - ***pearlite*** *- provides strength and hardness*
  - ***graphite*** *– provides thermal conductivity, vibration dampening, decreases tensile & fatigue strength*

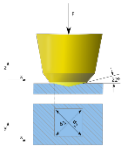
Possible Approaches to Improving Strength (UTS, FS) of Cast Iron:

- Refining the primary austenite dendrites
- Refining the eutectic cell size:
  - ***Inoculation (find most potent inoculants)***
  - ***Solidification at the limit or outside the Eutectic Coupled Zone***
- Improve the strength of the metal matrix:
  - Pearlite refining (alloying with Cu, Mo…)
  - Ferrite strengthening (nano-precipitates)

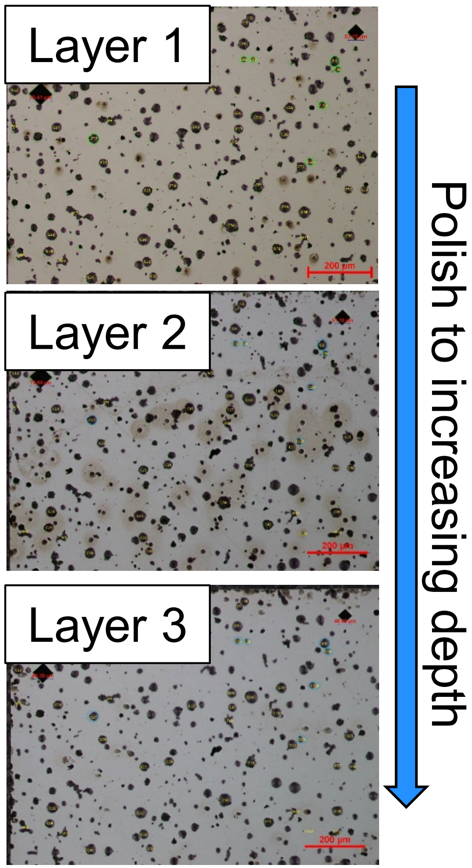
Approach


Microstructure Control/Refinement
Nucleation
Growth
Transformation
•Inoculation
•Alloying
•Casting Process
•Alloying
•Heat Treatment

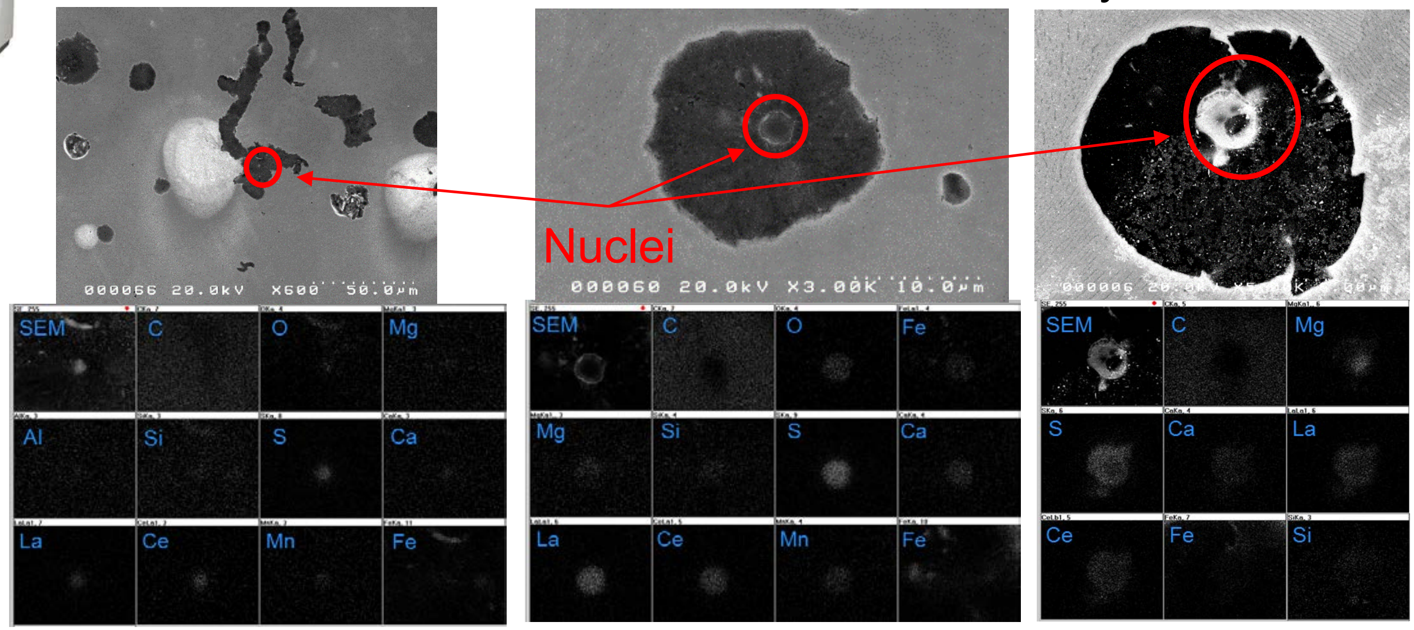
Graphite Nuclei Identification by Serial Sectioning



$$h = \frac{d}{2\sqrt{2}\tan\frac{\theta}{2}} \approx \frac{d}{7}$$

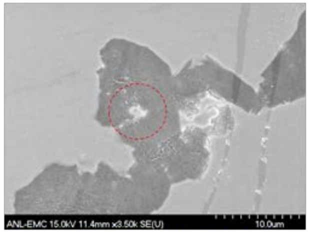


Vermicular Nucleus | Nodular Nucleus | Layered Nucleus

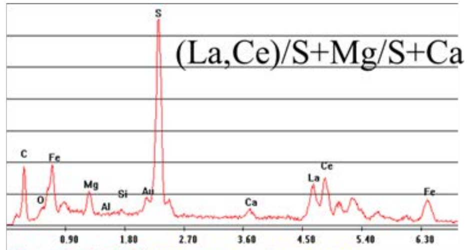


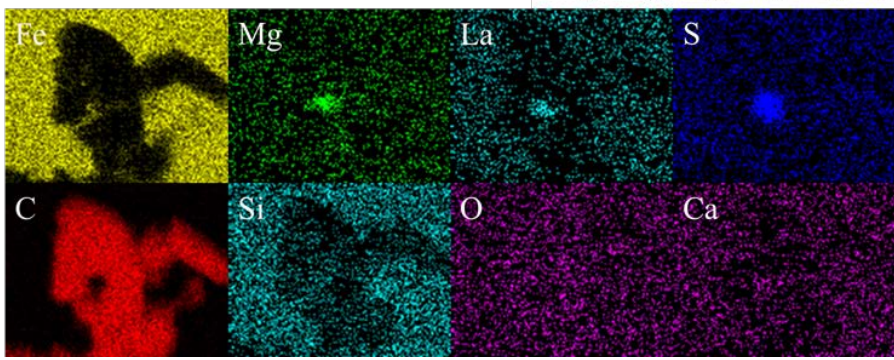
- Serial polishing to locate/track nuclei
  - ~0.6 micron layer removed for each polish (micro-indent tracking)
  - Graphite particles tracked for 3 layers (ongoing work)
- Nuclei for nodular and vermicular graphite revealed
  - EDS for particle ID: ***(La,Ce)S particles found***
  - Layered particles found: sulfide with oxide core

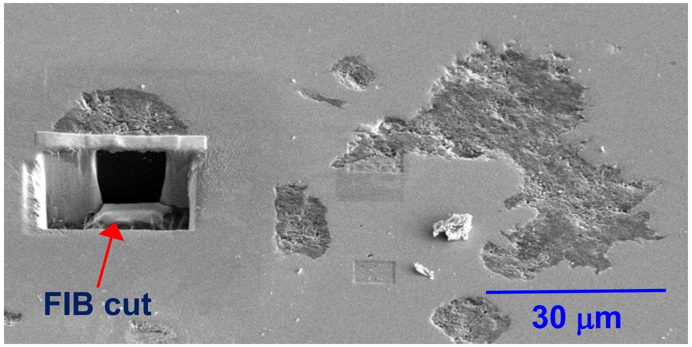
Graphite Nuclei Identification – FIB/SEM/EDS

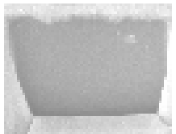


Element	Wt %	At %
O K	0.6	2.76
MgK	1.47	4.47
AlK	0.05	0.15
SiK	0.15	0.39
S K	12.98	29.99
CaK	0.91	1.69
LaL	25.91	13.81
CeL	37.71	19.93
FeK	20.21	26.81
Total	100	100

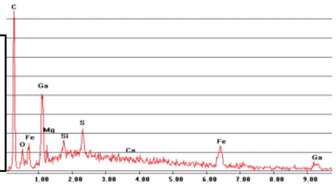


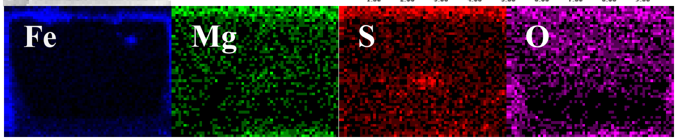


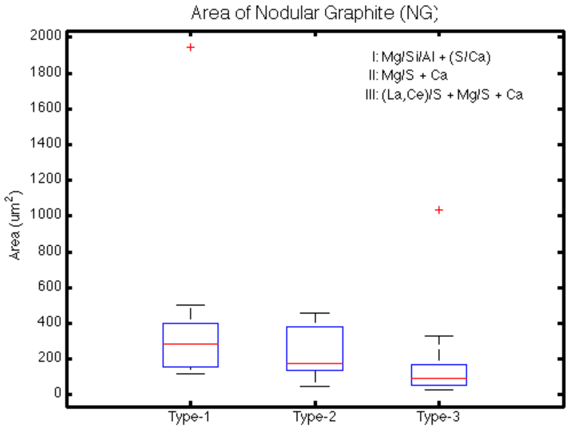


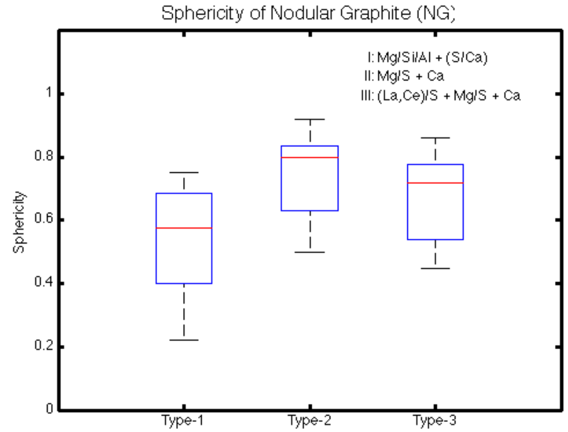


Core contains **Mg/S**









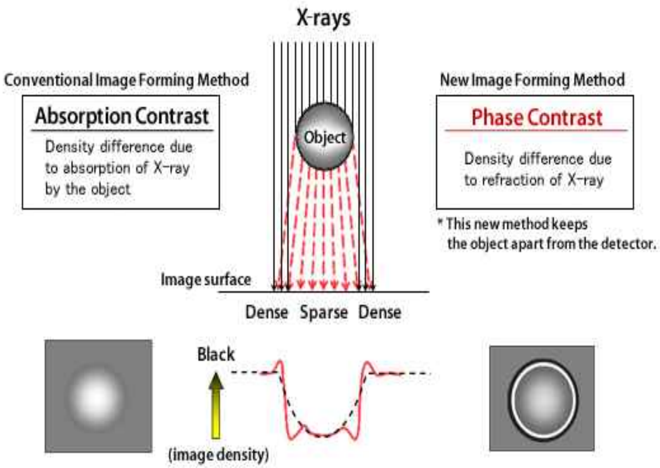
Type of nucleus identified :

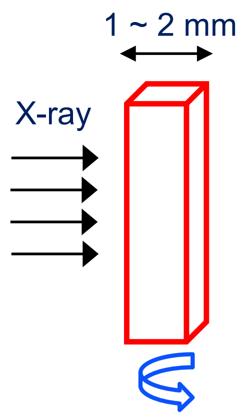
Type-I: Mg/Al/Si

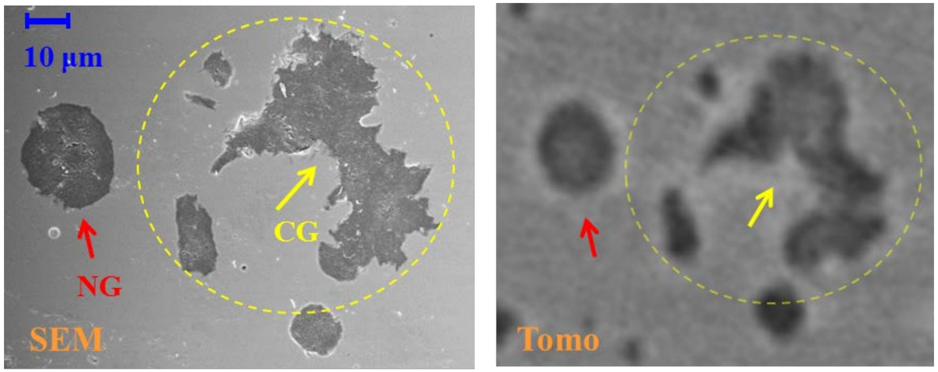
Type-II: Mg/S + Ca

Type-III: (La,Ce)/S + Ca

Non-Destructive 3D Tomography







Tomography reveals same feature as traditional metallography, but non-destructively and in 3D.

Absorption/phase contrast tomography

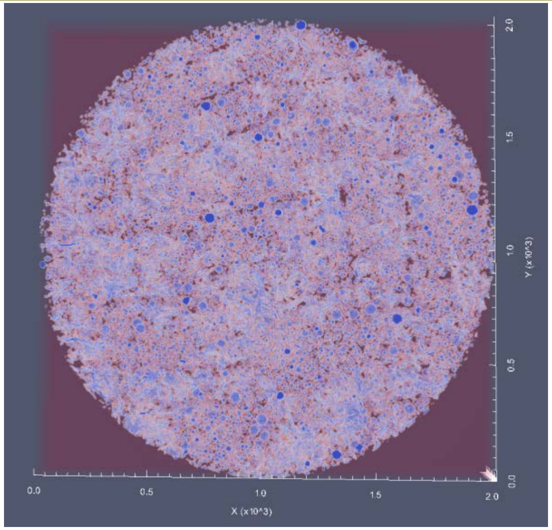
- Full field 2D image (mm^2)of direct beams
- Absorption contrast (near) or phase contrast (far) by changing sample-detector distance
- Take image and rotate -180 ~ 180
- Reconstruct 3D volume with resolution ~ 1μm

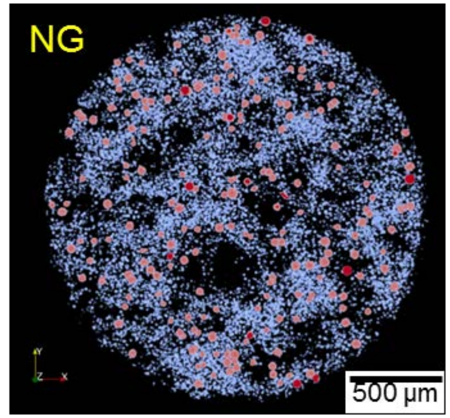
Video Here

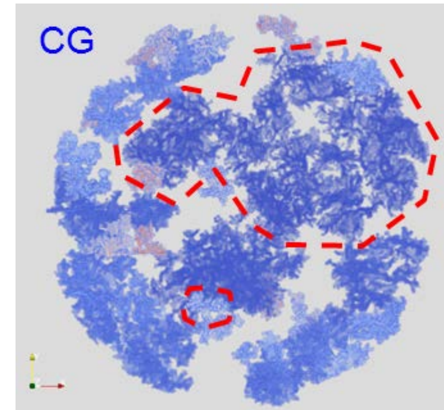
Video Here

Tomography reveals entire 3D structure.

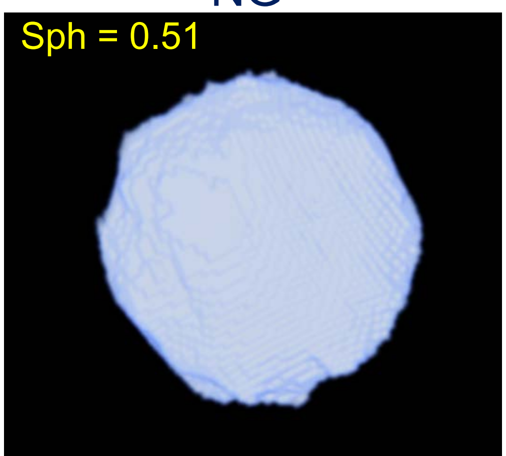
3D Quantitative Analysis of Graphite Morphology



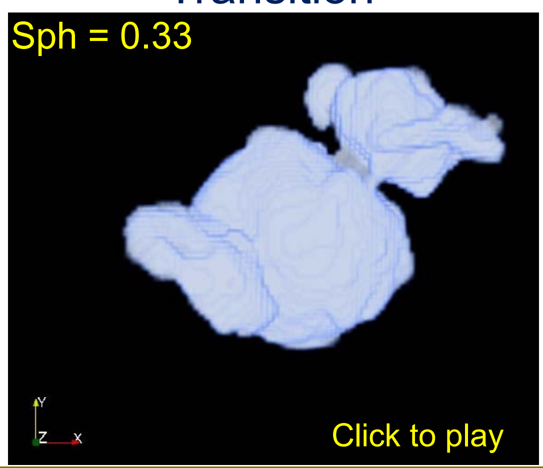




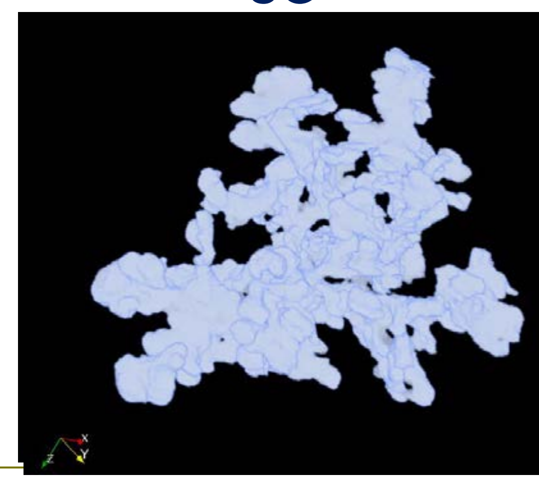
NG



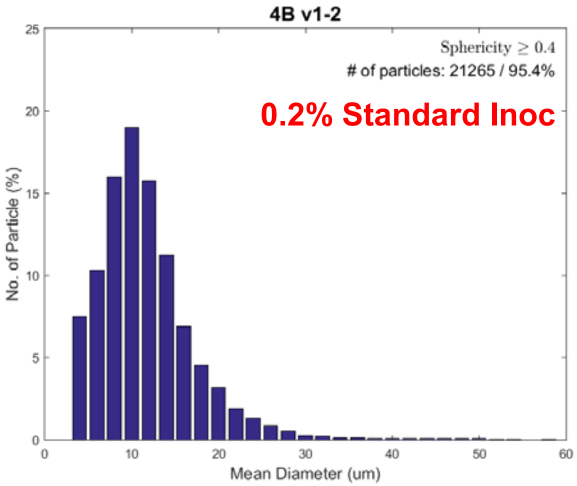
Transition

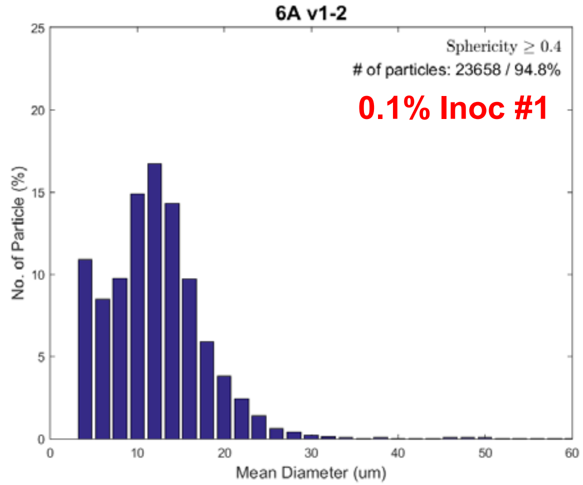


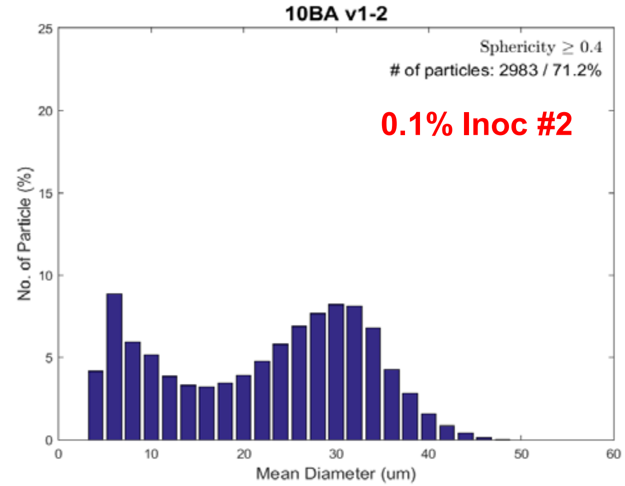
CG

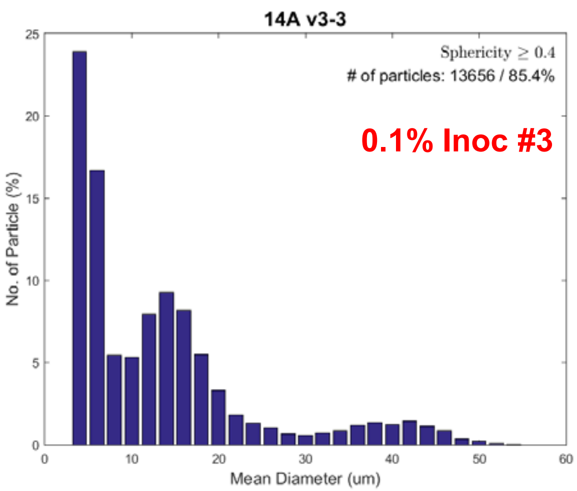


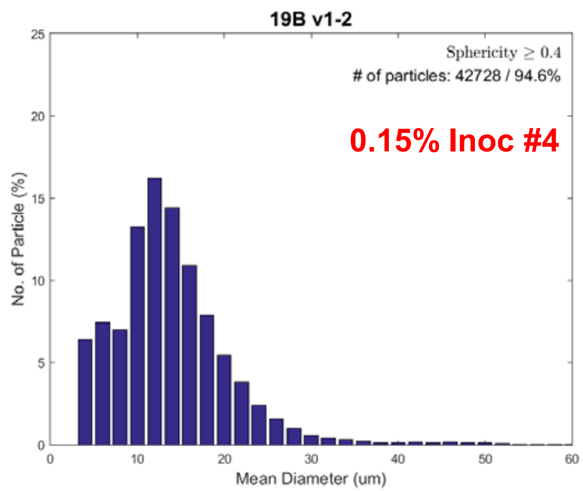
Nodule Size Distributions – Inoculation DOE

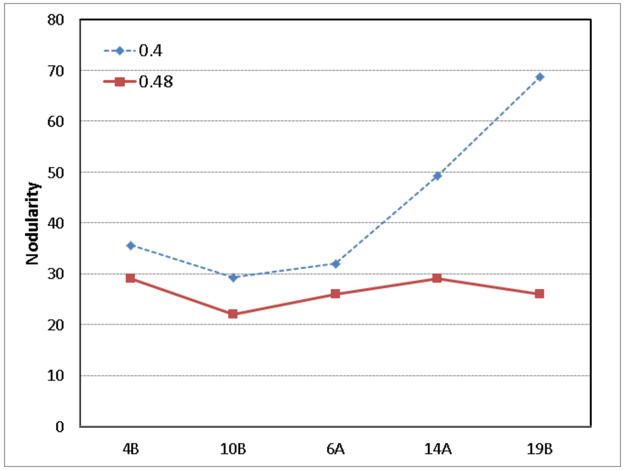




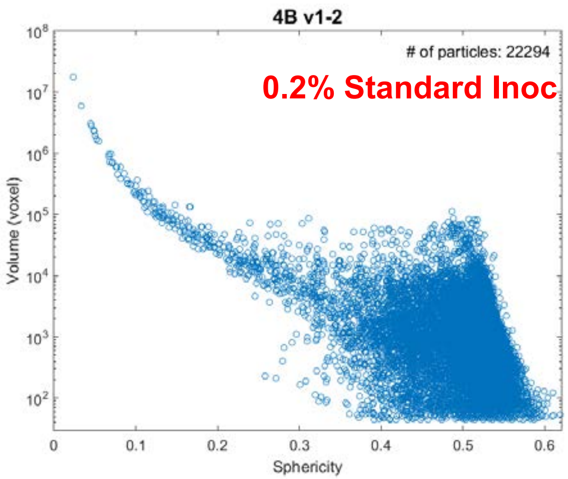


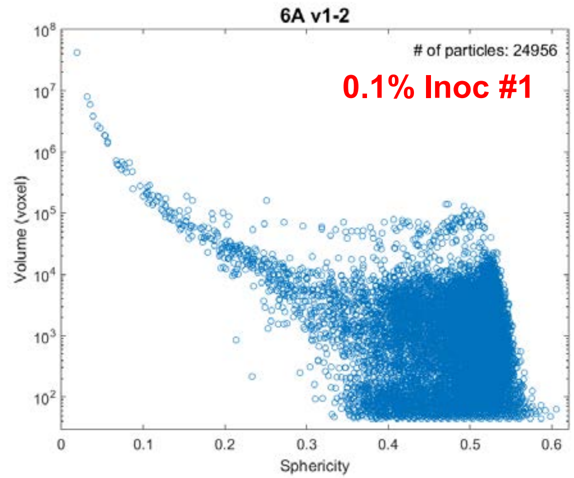


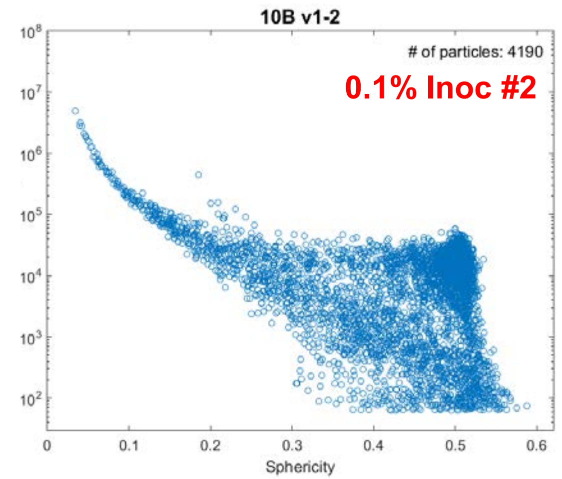


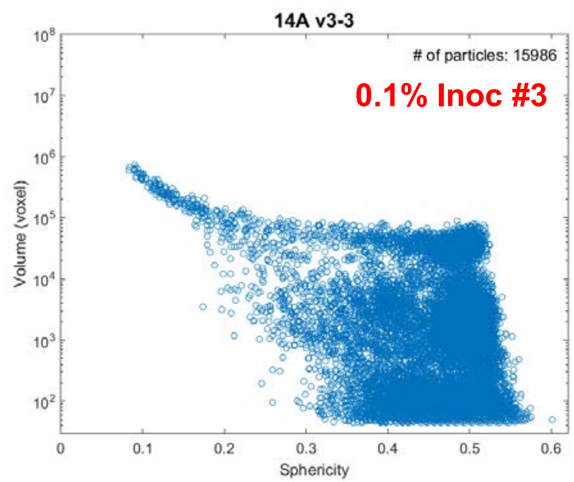


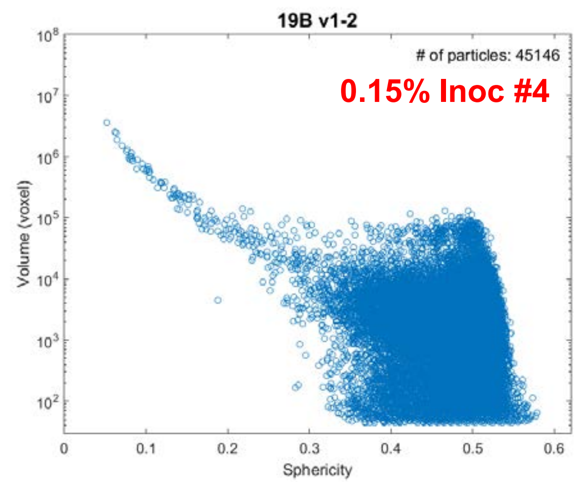
Volume Distribution of all Graphite – Inoculation DOE









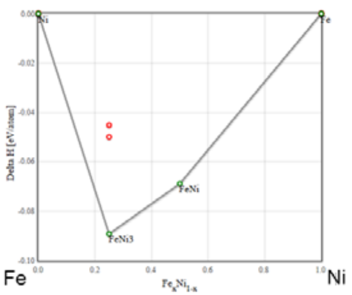


High-Throughput DFT Search for Austenite Inoculation

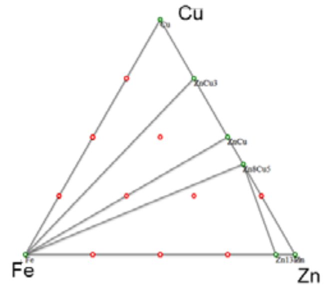
HT-DFT Search

ab initio Phase Diagrams

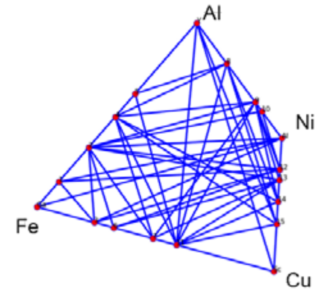
Binary ΔH: FeNi



Fe-Zn-Cu (ternary)



Fe-Al-Cu-Ni (quaternary)

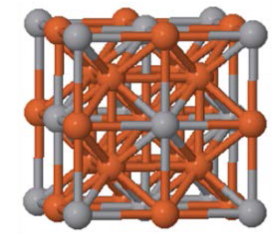


* Developed by the Wolverton Group: *www.oqmd.org*

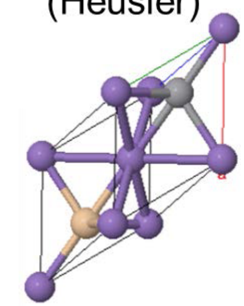
DFT stable structures* ⇨ Screen elements ⇨ Screen structures ⇨ Sort by LC matching

Austenite Matches

VFe_3 ($D0_3$)

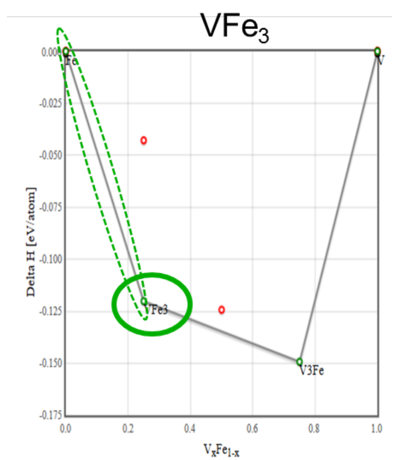


Mn_2VSi (Heusler)



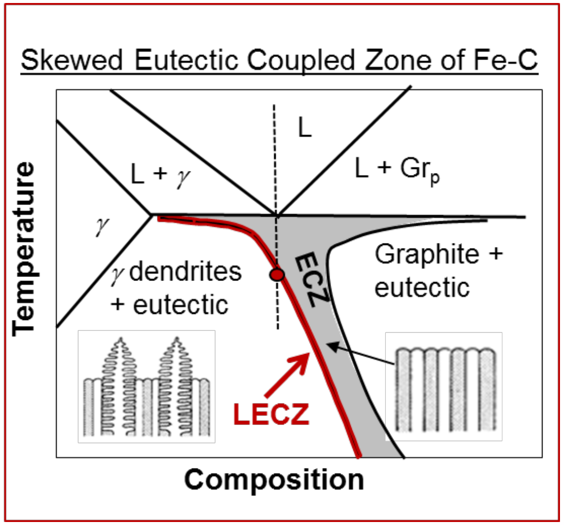
- Find new structures to inoculate austenite
  - Lower undercooling and promote fine prior austenite grain size
- OQMD*: Open Quantum Materials Database
  - Use *ab initio* phase diagrams to screen for stability
- 212 initial matches screened to 6
  - Screen for chemistry; lattice constant, crystal structure, etc.
- Matches for B1 and B2 structures: ***Mn-based; V-based; TaC***
  - Currently melting prototypes to determine stability
- Will utilize for graphite inoculation and ferrite strengthening

Binary *ab initio* convex hull showing VFe_3 stability in Fe

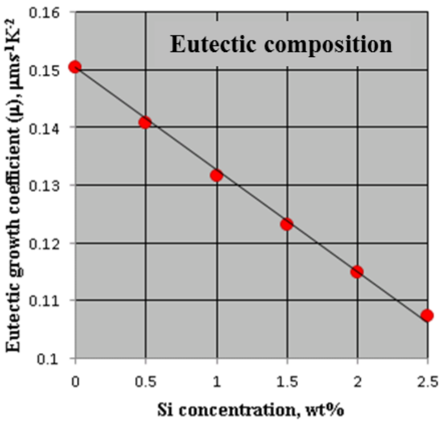


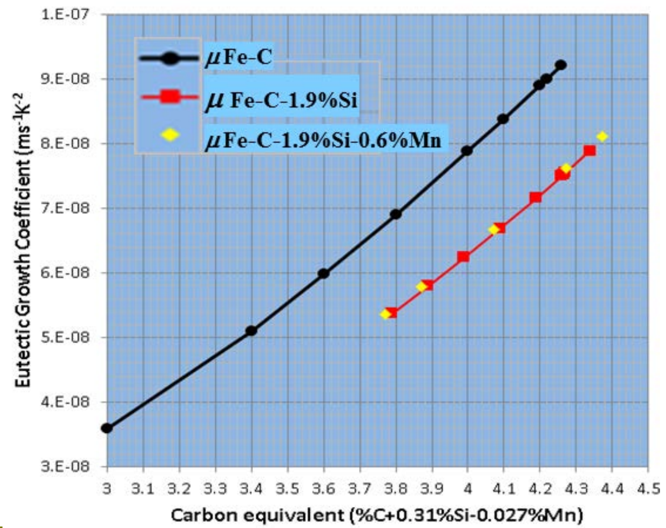
Eutectic Coupled Zone (ECZ) Solidification

- Inside the ECZ graphite and austenite grow in a cooperative manner as alternating lamellae; the graphite flake (lamella) size is comparable to that of eutectic cell size.
- Solidification outside the ECZ leads to refining of graphite flakes and increased tensile and fatigue strength of the material.
- The Eutectic Growth Velocity can be calculated with the relationship $V = \mu \cdot \Delta T^2$, where μ is the eutectic growth coefficient and ΔT is the undercooling
- **Developed model that allows studying the influence of composition on μ.**
- **Current findings**:
  - The developed model shows that eutectic microstructures at hypo-eutectic composition grow slower comparing to those of eutectic composition
  - Silicon additions decrease the eutectic growth velocity
  - Manganese does not have a sensitive influence on the eutectic growth velocity



$$\mu = 4.552 \cdot 10^{-8}\%C + 5.583 \cdot 10^{-9}\%Si$$
$$-1.163 \cdot 10^{-10}\ \%Mn - 1.0272 \cdot 10^{-7}$$





Eutectic Coupled Zone (ECZ) Solidification

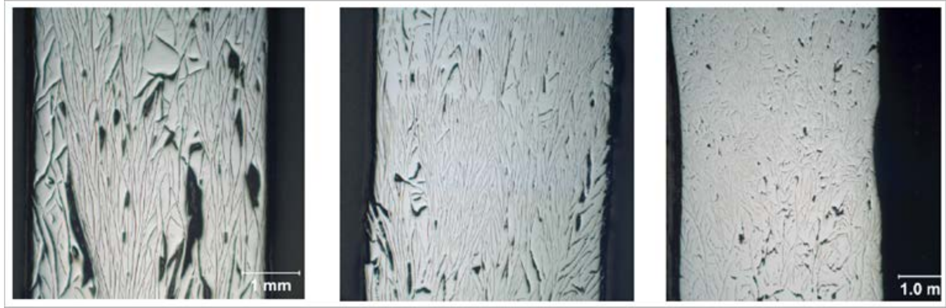
Eutectic Lamellar Spacing: Theoretical Calculations vs. Experimental Measurements

- Directional solidification experiments have been conducted at The University of Alabama at Birmingham

Chemistry of experimental samples

Specimen ID	Sorel Pig	#1	#2	#3	#4
C	4.52	3.83	3.42	3.57	3.45
Si	0.21	1.67	2.07	2.01	2.08
Mn	0.025	0.075	0.095	0.593	0.669
P	0.008	0.017	0.015	0.012	0.016
S	0.009	0.007	0.01	0.009	0.011
Cr	0.045	0.049	0.05	0.054	0.054
Mo	0.057	0.06	0.06	0.06	0.06
Ni	0.149	0.141	0.135	0.136	0.138
Cu	0.065	0.08	0.086	0.095	0.09
$C_{equivalent}$	4.59	4.39	4.11	4.23	4.13

Examples of microstructure obtained in directional solidification at V=0.5 μm/s



Sorel Pig   Specimen #2   Specimen #3

- **Current findings**:
  - The calculated eutectic spacing agrees well with experimental measurements when the chemistry of the alloy is not too far removed from the eutectic value (*e.g.*, specimens #1, #2, #3, #4)
  - For the highly hyper-eutectic specimen (*e.g.*, Sorel Pig) the agreement between the experiment and theoretical calculations is relatively poor but still within the range of the measurements errors

Eutectic Spacing Results (Exp. & Calculated)

Specimen ID	V (μms^{-1})	Exp. average spacing (μm)	Exp. std. dev. (μm)	Calc. average spacing (μm)
Sorel Pig	0.5	70.6	15.4	53.75**
#1	1	32.8 (48.2)*	6.5	29.4
#2	0.5	47.2	6.7	51.8
#3	0.5	45	7.3	46.2
#4	0.5	43.8	7.7	44.4

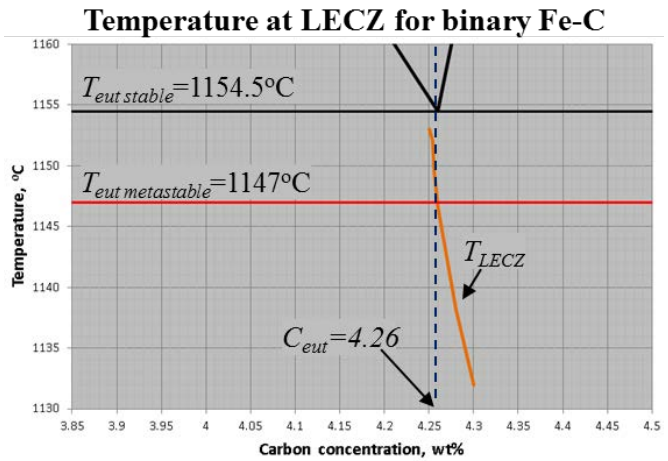
*) a bimodal spacing was observed due to the presence of austenite dendrites

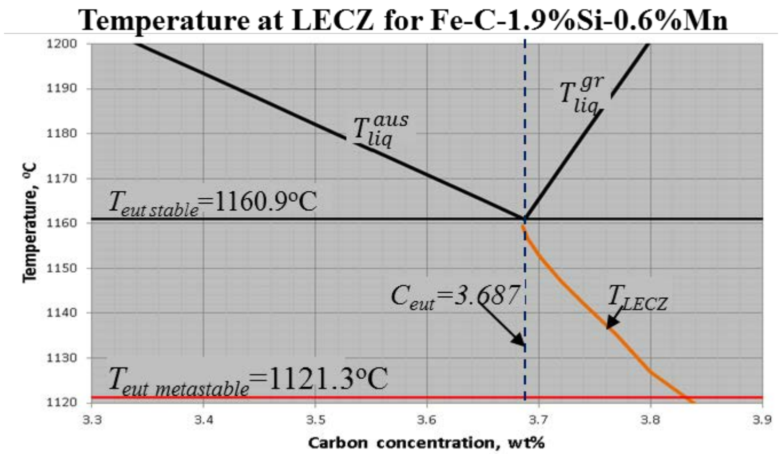
**) calculated as $(\lambda_{ex} + \lambda_M)/2$

Eutectic Coupled Zone (ECZ) Solidification

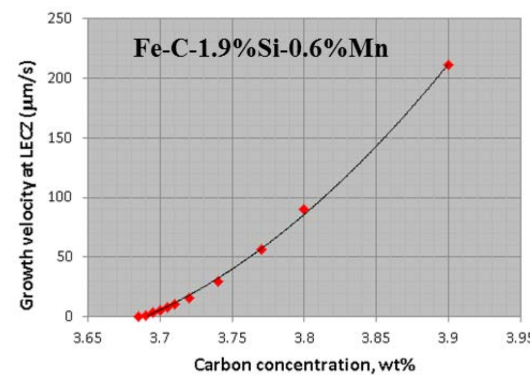
Calculation of Limit of Eutectic Coupled Zone (LECZ) in Fe-C Based Alloys

- LECZ was theoretically determined by using the developed eutectic growth model and the dendritic growth model available in literature (Lipton *et al*, 1984) which was extended to multicomponent alloys



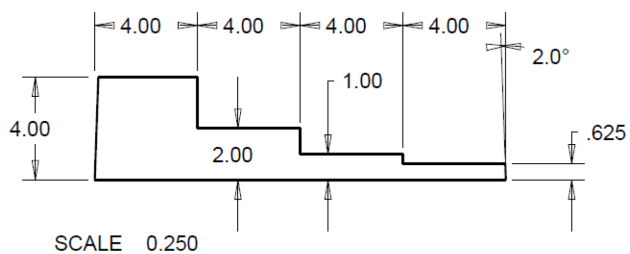


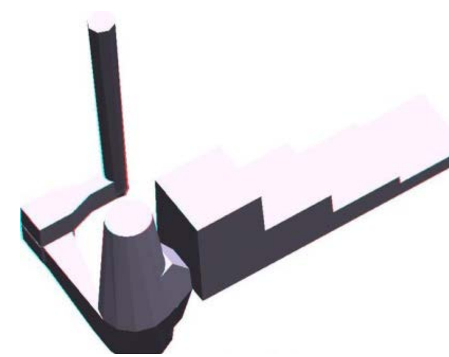
- **Current findings**:
  - Current LECZ model matches JK (*Jones & Kurz, 1980*) model at $C >= C_{eut}$, but strongly deviates from JK model at hypoeutectic compositions
  - The reason for this deviation is constant eutectic growth velocity used in JK model while the new model employs a chemistry dependent growth velocity
  - According to the developed model the dendritic austenite is always the leading phase at hypoeutectic compositions



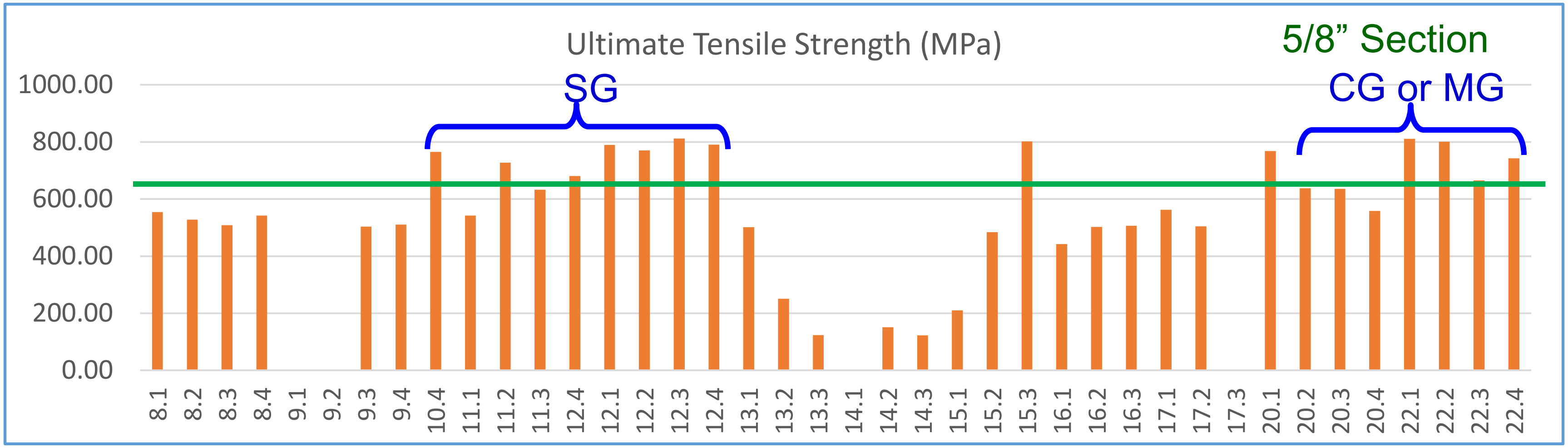
Casting Trials

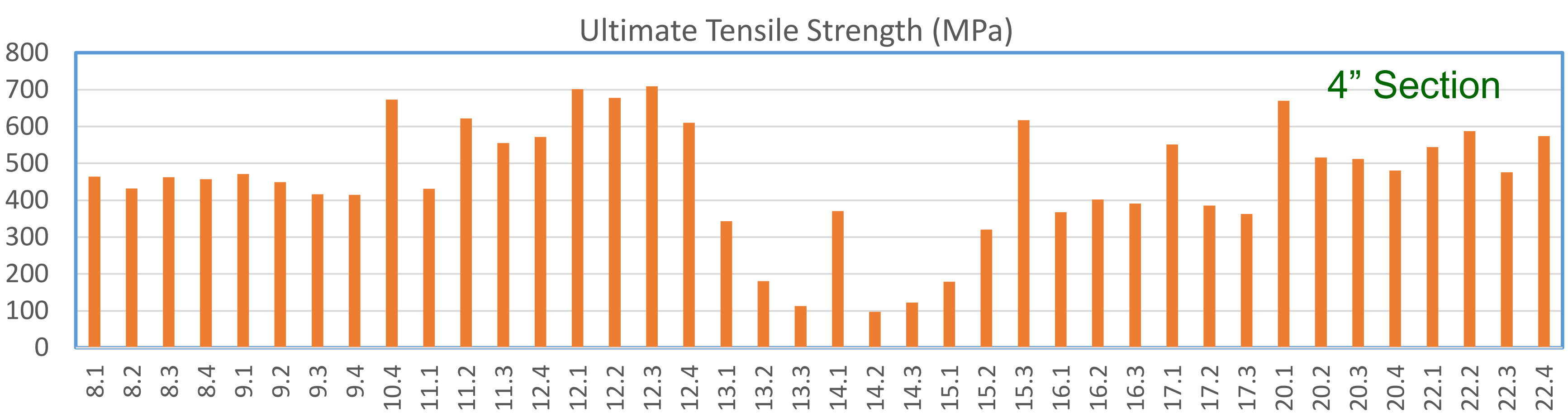
- Scaled up casting trials being performed at UAB's experimental foundry **(22 total heats poured, 3-4 variants per heat)**
  - Step block geometry chosen for test casting to provide a range of solidification rates
  - Inoculation
    - 5 graphite inoculants, 1 austenite inoculant
  - Alloying
    - Fe-C-Si-Al-S-Bi-Cu-Sn-Sb-V-Mo
  - Structure-Property
    - Tensile, Yield, Elongation, Modulus
    - Phase Fraction %, Carbide %,
    - Nodularity, Graphite Count, Graphite Size
    - *Eutectic Cell Size, Fraction Eutectic/Primary*





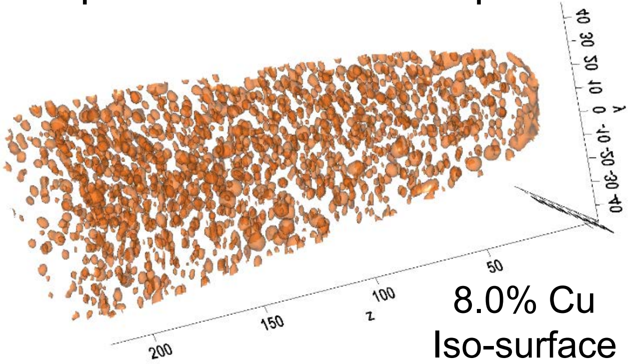
Casting Trials





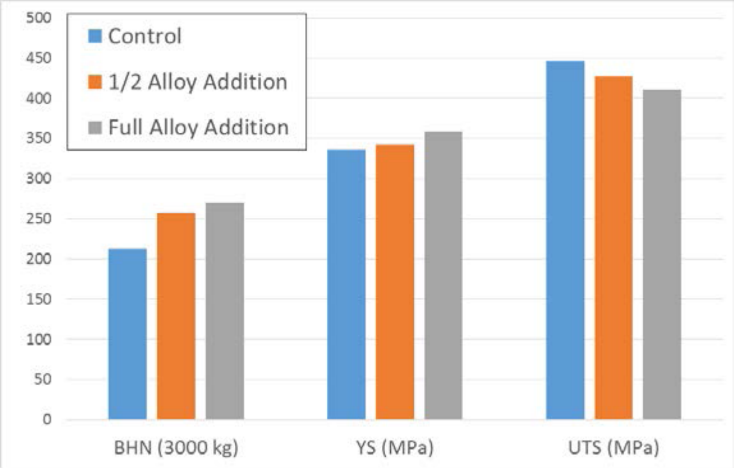
Cu-Ni-Al Precipitate Strengthening

Atom Probe (LEAP) of Cu-Ni-Al precipitates in button samples



8.0% Cu Iso-surface

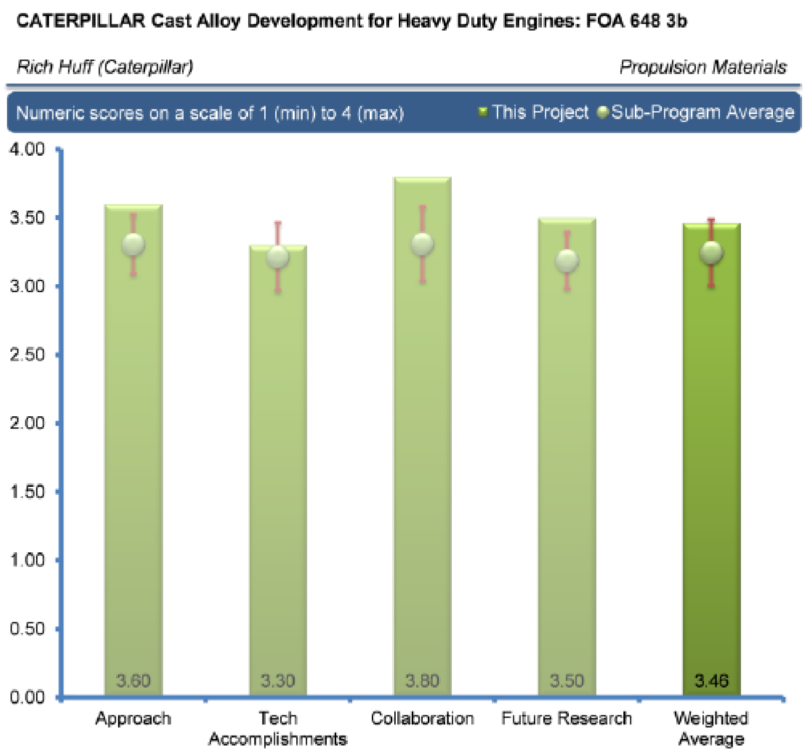
Step Block Properties (as-cast)



- Stabilize bcc-structure (B2) in sub-scale buttons
  - Cu-based precipitates identified with atom probe
  - Increase shear resistance
- Target alloys cast during scale-up activities
  - ***Increased hardness and YS achieved***
  - LEAP planned for precipitate confirmation
  - No observed tempering response
- Next Steps: Optimization of as-cast microstructure for improved UTS

Response to Reviewers Comments

- Comments from last year were generally all positive.
  - Liked approach
  - Good collaboration
  - Leveraging DOE Lab capability

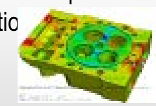


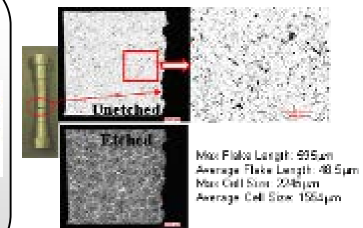
Collaboration – Project Team

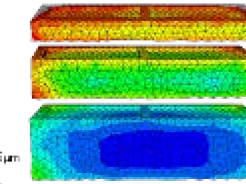
Project Lead →

CATERPILLAR®

- Material and Process Development
- Material Characterizatio
- Design Integration
- Integrated Simulation
- Design Optimization
- Concept Design Cost Model



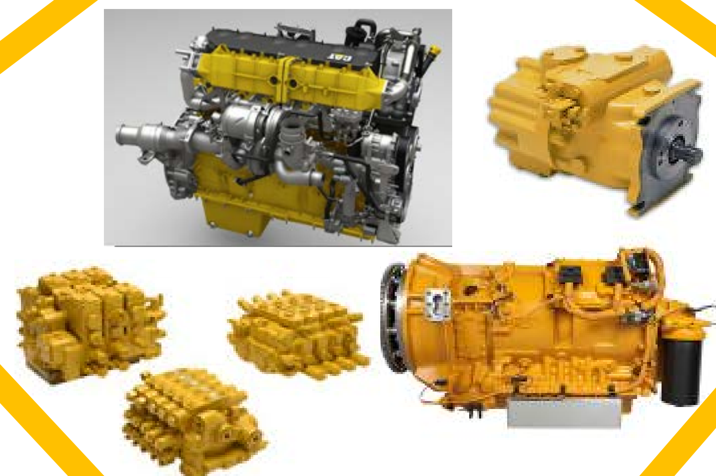




215 – 300 MPa

Argonne NATIONAL LABORATORY

- Material Evaluation using Advanced Photon Source (APS) X-Ray and MTS Testing Machine
- In-Situ Microstructure and Damage Measurements

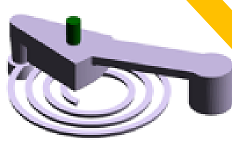


QuesTek INNOVATIONS LLC

- Computational Material Design
- Solidification Design
- Transformation Design
- Nano Design
- Material Characterization

Addition Support:

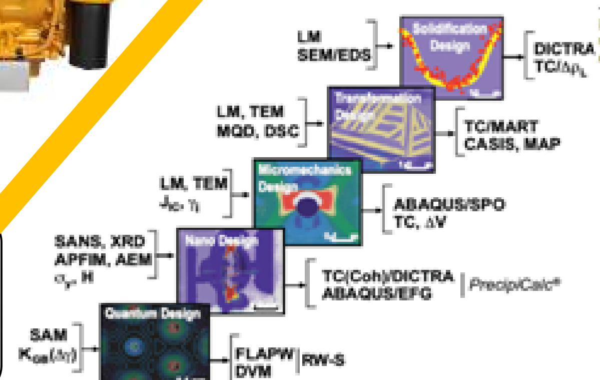
- Northwestern University
  - Dr. P. Voorhees
  - Dr. C. Wolverton
- Bradley University
  - GI Fatigue Testing



Fluidity Spiral Test Casting

UAB THE UNIVERSITY OF ALABAMA AT BIRMINGHAM

- Experimental Casting Material Samples
- Liquid Metal Fluidity Evaluation
- Hot Tear Resistance



Remaining Challenges and Barriers

- Nucleation and growth of austenite + graphite is not completely understood, especially for CGI.
  - There could be a theoretical limit to the nucleation density possible for graphite and the resulting microstructure refinement
  - Primary phase nucleation may be only refinement mechanism, extremely difficult to find austenite nucleants
  - Level of control of the eutectic growth that is attainable through control of alloying and casting conditions has not been quantified
- Material design that meets the mechanical properties targets while simultaneously satisfying thermal conductivity and manufacturing cost requirements is a significant challenge.
  - Can mechanical properties targets be achieved with a vermicular graphite morphology?
  - Strength-Conductivity trade-off
  - Strength-Fatigue relationship
- Satisfactory ICME tools for the fundamental material modeling to design cast iron alloys are not available.

Future Steps

- Nucleation
  - Continue serial sectioning to identify nucleants and better understand inoculation
  - *In-situ* solidification experiments using X-ray radiography
  - Refine precipitation models of strengthening phases and inoculants
  - DFT calculations to identify new inoculation strategies
- Growth
  - Directional solidification + quenching experiments for CGI
    - Quantitative evaluation on the influence of alloying elements and solidification conditions on the formation sequence of CGI
  - Improve and validate "Divorced Eutectic" solidification model (Austenite +CG)
- Casting Trials
  - Continue step block trials for variations of the high strength alloying concepts already identified
  - Develop experimental process for increasing the solidification rate of step blocks
  - Define property limits and strength vs. thermal conductivity trade-offs
  - Investigate fatigue performance of the high-strength alloy concepts
  - Cast step block samples of new precipitation strengthening and inoculation concepts

Summary

- Project is relevant to the development of high-efficiency, low-emission heavy-duty engines
  - Improved material properties are needed to enable heavy-duty engines to operate in optimal combustion regimes
  - Increased mechanical properties will allow the mass of heavy-duty engines to be reduced, which will improve fuel-economy at the vehicle level
- FY13-15: focus on identifying and modeling the critical mechanisms governing the microstructure development during solidification and high-strength alloy concepts
  - Nucleant identified in several graphite particles and categorized three types by size.
  - First CG nucleants found, determined CG nucleants are the same as SG nucleants
  - X-ray methods established to reveal complex 3-D graphite network in cast irons and quantified the distribution of graphite particles (size and shape).
  - DFT work in progress to identify potential new inoculant strategies for austenite and graphite phases and potential precipitation strengthening phases.
  - Eutectic Coupled Zone (ECZ) solidification model extended to multi-component alloys and validated through directional solidification experiments. Extending to CGI growth.
  - Casting trials have identified alloy concepts with strengths ranging from 650 to 820 MPa tensile strengths for CG->MG->SG.

Technical Back-up Slides

Approach

Updated systems-design chart for cast iron