Materials Synthesis: Enabling and Benefitting from High B Experiments

Ryan Baumbach[1,2]
1. National High Magnetic Field Laboratory
2. Florida State University, Tallahassee

- What synthesis methods at MagLab?
- When are magnetic fields useful?

The challenge of developing new materials

- Discover of extraordinary phenomena requires:
  - Familiarity with fundamental degrees of freedom
  - Deep knowledge of chemical/structural/electronic/magnetic trends
  - Sifting through the chemical/structural phase space
  - Creating space for serendipity

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Physics, Chemistry, and Materials Science in harmony

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Rational design of materials invites AI, data mining, fair data and more engagement with theorists

Materials Design and Synthesis at the MagLab


Christianne Beekman
MagLab-FSU


Luis Balicas
MagLab-FSU


Shalinee Chikara
MagLab-FSU


Ryan Baumbach
MagLab-FSU


Kaya Wei
MagLab-FSU

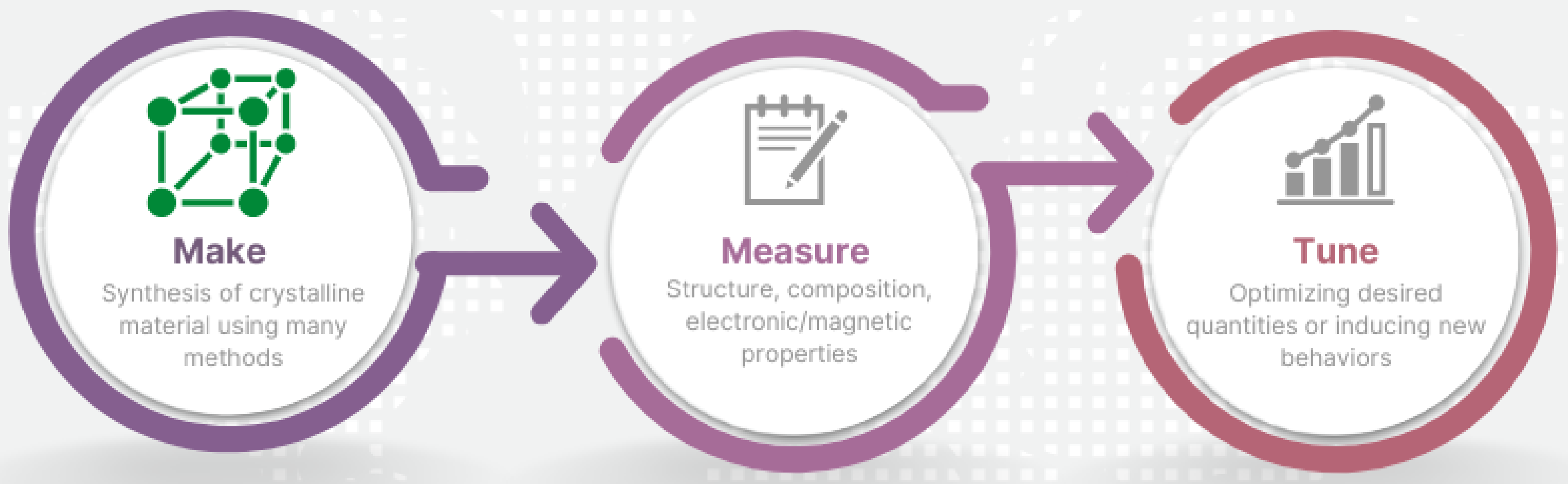

James Hamlin
MagLab-UF


Mark Meisel
MagLab-UF


Shermane Benjamin
MagLab-FSU

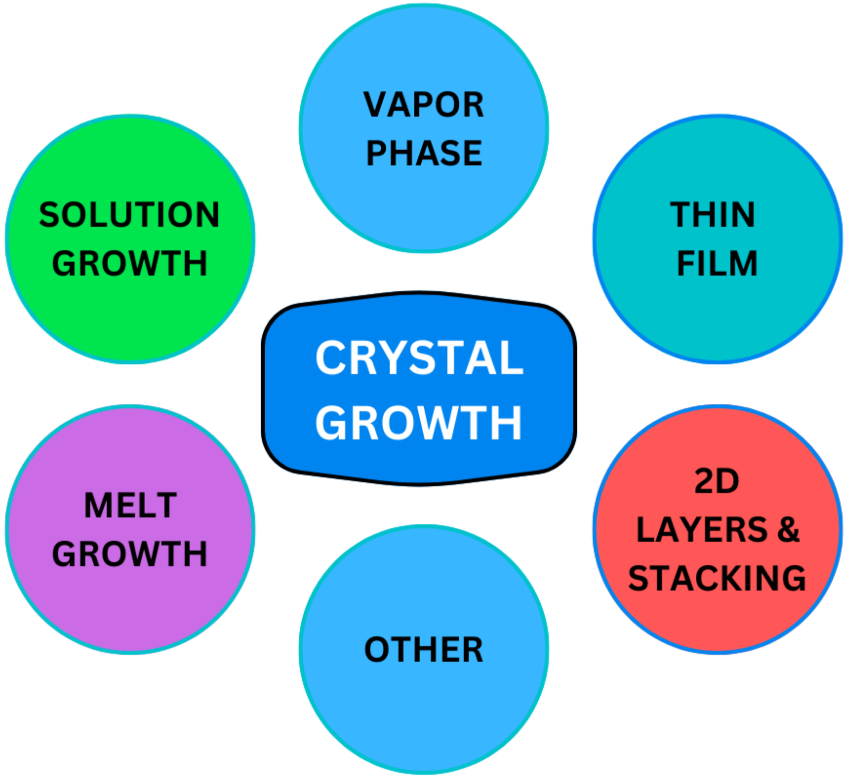

Laura Greene
MagLab Chief Sci.


Theo Siegrist
MagLab-FSU/FAMU



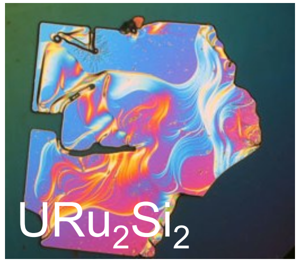
P. Canfield “**New Materials Physics**”, Reports on Progress in Physics 83, 016501 (2019).

Materials Design and Synthesis Methods

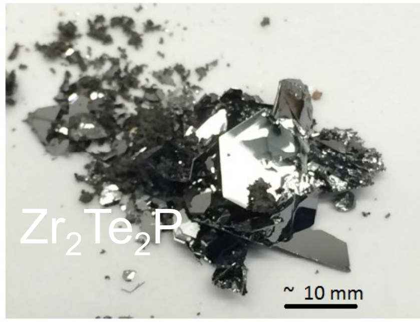


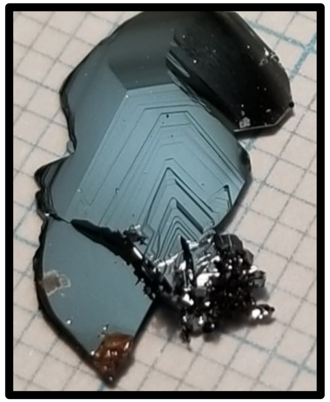
Materials Design and Synthesis Methods

Molten Metal/Salt Flux



Iodine (and other) transport

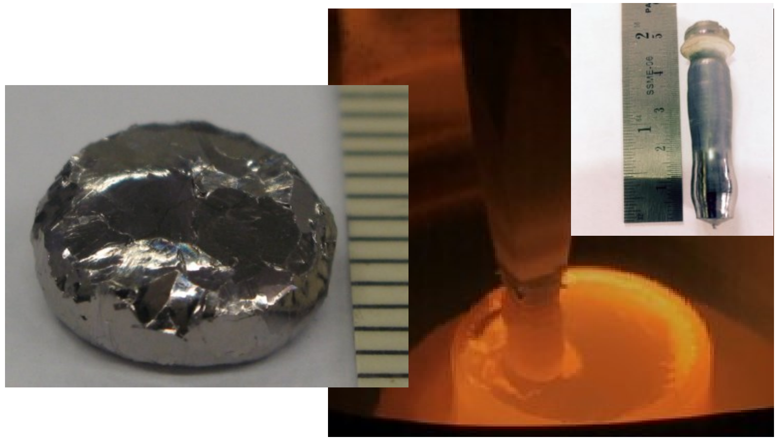




CRYSTAL GROWTH

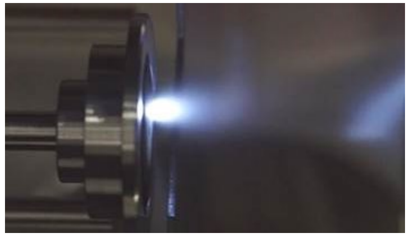
- **SOLUTION GROWTH** — Molten Metal/Salt Flux
- **VAPOR PHASE** — Iodine (and other) transport
- **THIN FILM** — Pulsed laser deposition, Magnetron sputtering
- **2D LAYERS & STACKING** — Exfoliation and devices
- **OTHER**
- **MELT GROWTH** — Arc furnace, Bridgman, Czochralski, float zone furnace

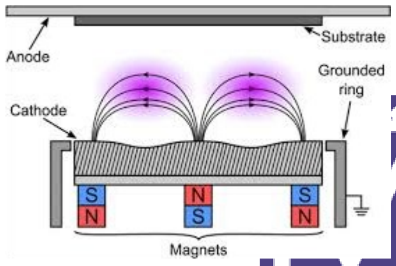
Arc furnace, Bridgman Czochralski, float zone furnace



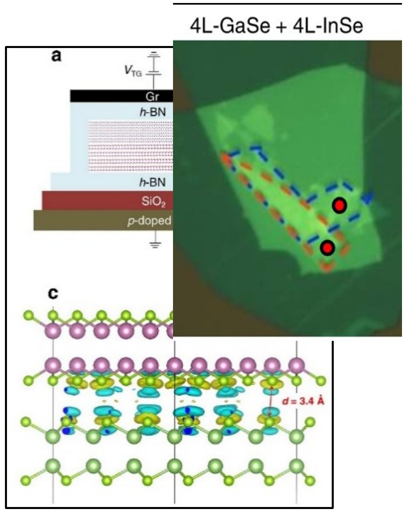
Pulsed laser deposition

Magnetron sputtering





Exfoliation and devices

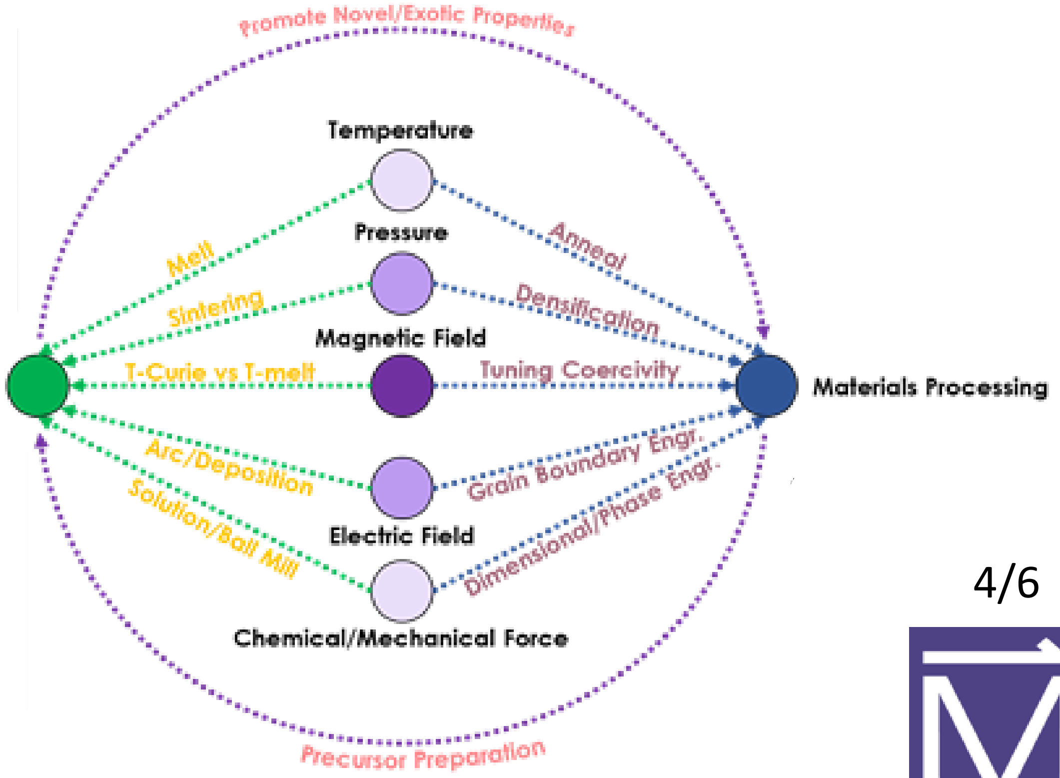


Materials Growth under Applied Magnetic Fields

HBT
DC Field

					
Laura Greene MagLab-FSU	James Hamlin MagLab-UF	Ryan Baumbach MagLab-FSU	Kaya Wei MagLab-FSU	Mark Meisel MagLab-UF	Theo Siegrist MagLab-FSU/FAMU

- **Magnetic Field as a Thermodynamic Variable in Materials Growth/Processing**
- **Motivation: The stability of a phase is given by the Gibbs Free Energy**
  $dG(p,V,T,H) = dU + pdV - TdS + HdB$
- **Ground State of a System depends on H**



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MagLab-FSU/FAMU

Magnetic Fields have Small Energy Scales.

However: Some effects will go at B^2 and increase from 1 T to 18 T is a factor of 324, and further extensions to the Series Connected Hybrid is 18 T to 36 T is 4.

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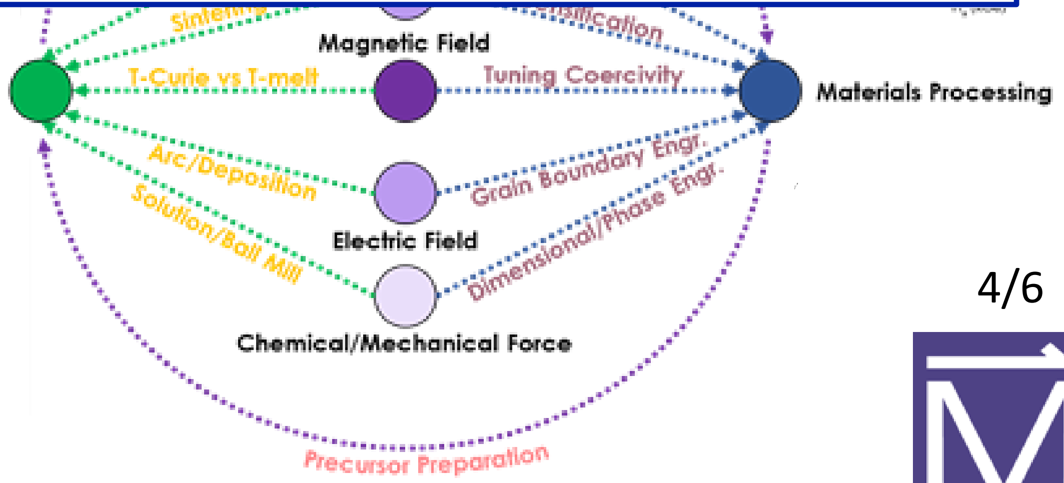

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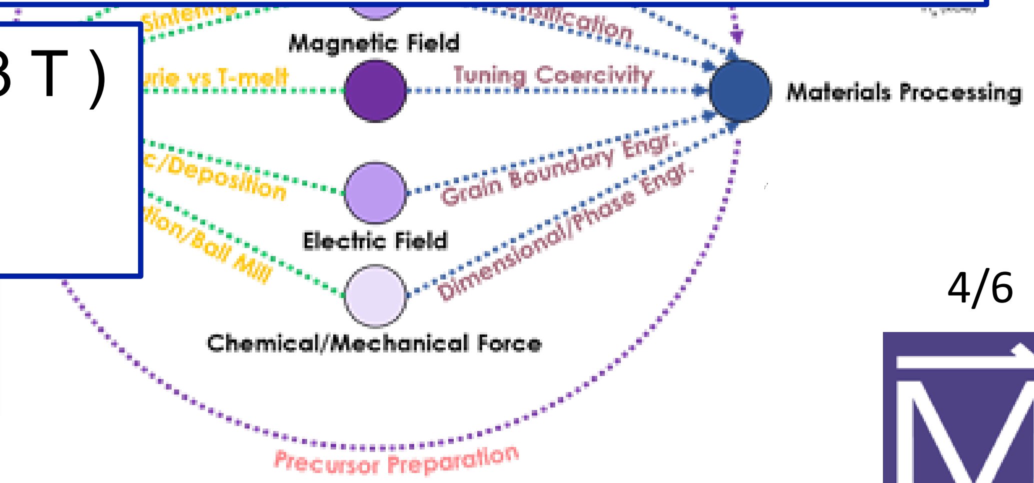
Energy: $g\,\mu_B\,B = k_B\,T$ (where $\mu_B / k_B \approx 2$ K / 3 T)

So: g = 5 in $B = 18$ T $\rightarrow$ $T \approx 60$ K



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HBT

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$B\Delta B$ gradient fields

Separation chemistry

❖ **Ground State of a System depends on H**

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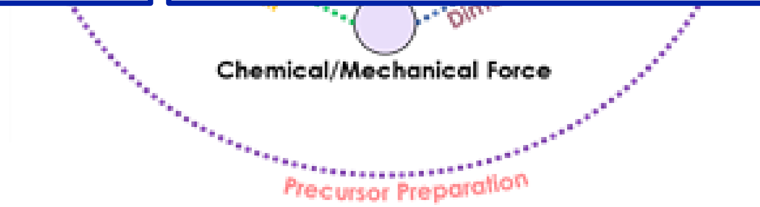
$B\Delta B$ gradient fields
Separation chemistry

Selecting metastable structural phases

on H

ng

4/6



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$B\Delta B$ gradient fields
Separation chemistry

Selecting metastable structural phases

Other Impacts: (i) Reduced convection, (ii) preferred growth direction

International Efforts are Under Way

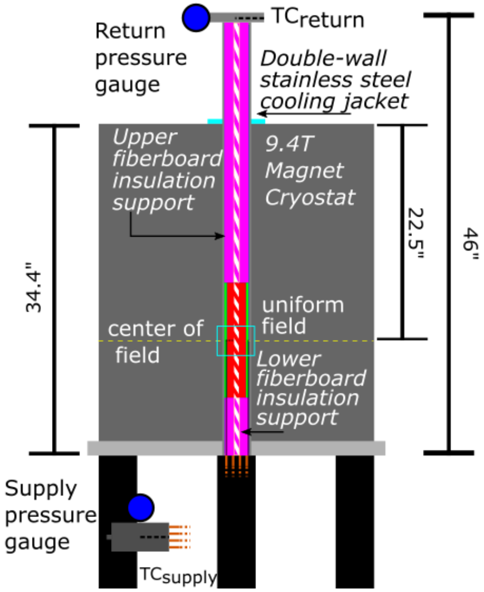
High Magnetic Field Laboratory
- Hefei, China
Natl. Lab. of High Magn. Field, CNRS/LNCMI
- Grenoble, France
National Institute for Materials Science
-Tsukuba, Japan

1. Magnetic force
2. Lorentz force and Magneto-hydrodynamic effect
3. Thermodynamic effect under magnetic fields
4. Magnetic orientation
5. Analytic technique using magnetic fields
6. Separation technique using magnetic fields
7. Spin chemistry and photophysics
8. Biological phenomena under magnetic fields
9. Magnetic field generation
10. Other phenomena related to magnetism or magnetic fields

5/6

Expanding Capabilities and User Community

UF High B x T Facility



Temperature < 1200 °C

½" I.D., 12" furnace chamber

Adjustable sample position

B_{max} = 9 T

or $(\nabla B)_{max}$ (0.7 T cm^{-1})

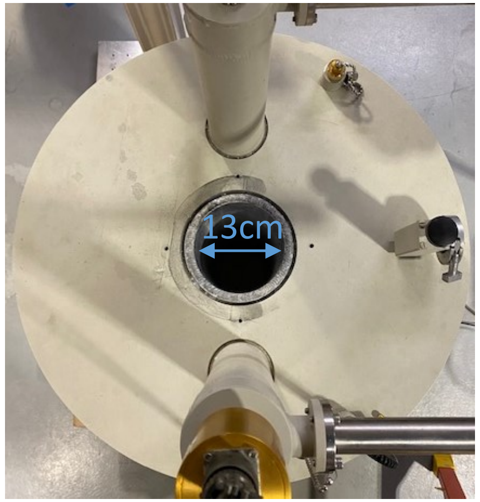
Automatic safety monitoring and fault response

FSU DC Field Facility

13 cm I.D.

Adjustable sample position

B_{max} = 5 T



External Funding

- * DOE EERE DE-EE0009131, PI: Michele Manuel, UF MSE, with James Hamlin & Mark Meisel, UF Physics, as Co-PIs.
- NHMFL-UCGP Funding proposal, PI Kaya Wei, MagLab-FSU
- ***Other funding opportunities?***

Personnel

- Steven Flynn, Postdoctoral Researcher, Caeli Benyacko, Jared Lee, Undergrad. REU. PIs: James Hamlin, Mark Meisel, MagLab-UF
- Benny Schundelmier, Grad. Student. PI: Kaya Wei, MagLab-FSU
- Caleb Bush, Undergrad. REU. PI, Ryan Baumbach MagLab-FSU
- ***Additional personnel – e.g., for sustained user support***

MagLab Users Committee Meeting

18 September 2023

Mark W. Meisel
Department of Physics and MagLab High B/T Facility
University of Florida, Gainesville

Development of High Energy Density Thermomagnetic Processing Technology for Intensification of Industrial Heat-Treatment and Increased Material Performance

DOE AMO EERE DE-EE0009131

Technology Summary:

Induction-coupled Thermomagnetic Processing (**ITMP**) of steels provides a means of realizing energy savings and achieving previously elusive material properties.

Impact of ITMP:

+ Enables rapid processing of steels, reducing processing time.
+ Provides energy savings, reducing process energy consumption by up to 96% during thermal treatments.
+ Shifts thermodynamic equilibrium of the steels, increasing yield strength and ductility of heat-treated steels.
+ Reduces lifecycle energy and resource impacts of manufactured goods.
+ Improves the U.S. heat treating industry competitive advantage through improvements in energy efficiencies, as well as operational and environmental impacts.

Michele Manuel UF MSE | Tori Miller UF MSE | Mike Tonks UF MSE | Richard Hennig UF MSE | Gerry Ludtka UF MSE | James Hamlin UF Physics | Mark Meisel UF Physics | Mike Kesler ORNL | Dallas Trinkle UIUC MSE | Harrison Kim UIUC IESE | Ling Li VATech | Charlie Li | Lynn Ferguson DANTE Solutions

University of Florida

Post-Docs and Students: Ramon Padin-Monroig, Megan Hurley, Ming Li, Alexander Donald

University of Illinois at Urbana-Champaign

Post-Docs and Students: Luke Wirth, Kangcheng Lin

Oak Ridge National Laboratory

Technical Staff: Michael Thompson, Bart Murphy, Dante Quirinale, Cory Fletcher

Beamline Scientists: Matthias Frontzek, Jeffrey Bunn, Lisa DeBeer-Schmitt

Virginia Tech

Students: Ravi Bollineni

Program Goals

Demonstrate ITMP for significant energy intensification over conventional commercial heat treat processing.

Improve mechanical properties (UTS increase without ductility loss).

Reduction in Quench and Temper energy.

Provide baseline information to industrial partner’s targeted application.

Technical and Scientific Objectives

Demonstrate utility of ITMP technology for manufacturing applications.

Generate zero-field and field assisted diagrams of phase stability and phase transformations.

Increase predictive capability of high field related effects on steels.

Develop and employ multi-scale modeling and simulation tools to drive performance and optimize design and operation of ITMP using an Integrated Computational Materials Engineering (ICME) approach.

Demonstrate industrially-relevant ITMP at system-scale.

Development of High Energy Density Thermomagnetic Processing Technology for Intensification of Industrial Heat-Treatment and Increased Material Performance

University / National Lab / Industry Partnership

11 senior investigators
+ 14 early career scientists
+ 6 additional industrial partners

dante | loukustech | John Deere | Ajax TOCCO Magnethermic | Vision Materials | CATERPILLAR

Main objective of this research program is to demonstrate the use of Induction Coupled Thermo-Magnetic-Processing (ITMP) technology on the **heat treatment of primarily ferrous alloys to yield higher energy efficiencies through temperature/time reductions.**

Leadership Team
Michele Manuel (UF)
Michael Tonks (UF)
Michael Kesler (ORNL)

Task 1: Michael Kesler (Lead)
Victoria Miller (UF)
Mark Meisel (UF)
Michele Manuel (UF)
Michael Tonks (UF)

Task 2: Michael Tonks (UF)
Richard Hennig (UF)
James Hamlin (UF)
Dallas Trinkle (UIUC)
Reza Mirzaeifar (VT)
Charlie Li (DANTE)

Task 3: Michele Manuel (UF)
Mark Meisel (UF)
Michael Kesler (ORNL)
Harrison Kim (UIUC)

BRIDGE
Michael Tonks
James Hamlin

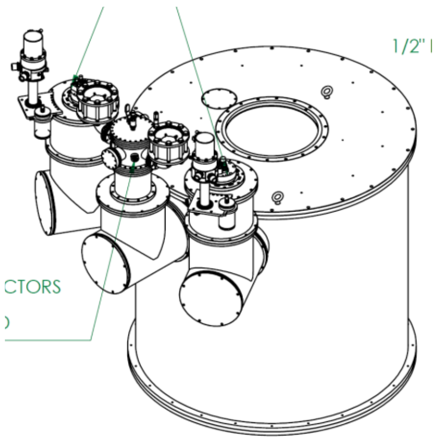
BRIDGE
Mark Meisel
Michael Kesler

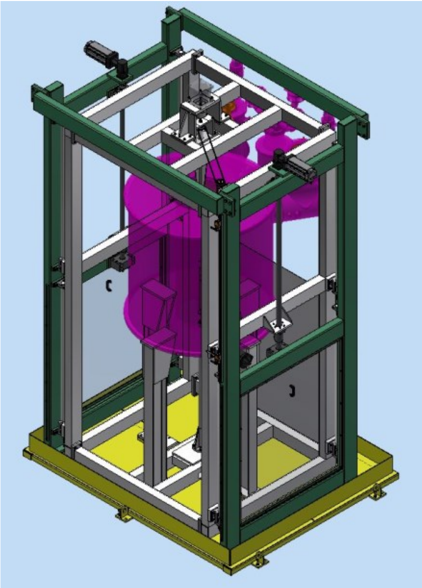
Task 1: Conventional (steel and alloys) and advanced (new and novel) materials synthesis and annealing/treatment in ***magnetic fields*** followed by physical/mechanical and magnetic characterization from which the resulting microstructural and physical properties will inform/validate the computation effort in **Task 2** and leverage the predictive computational capability to design routes for enhancing industrial alloys. Facilities in ORNL and in the **High Bay Convergence Lab at UF** will be used.

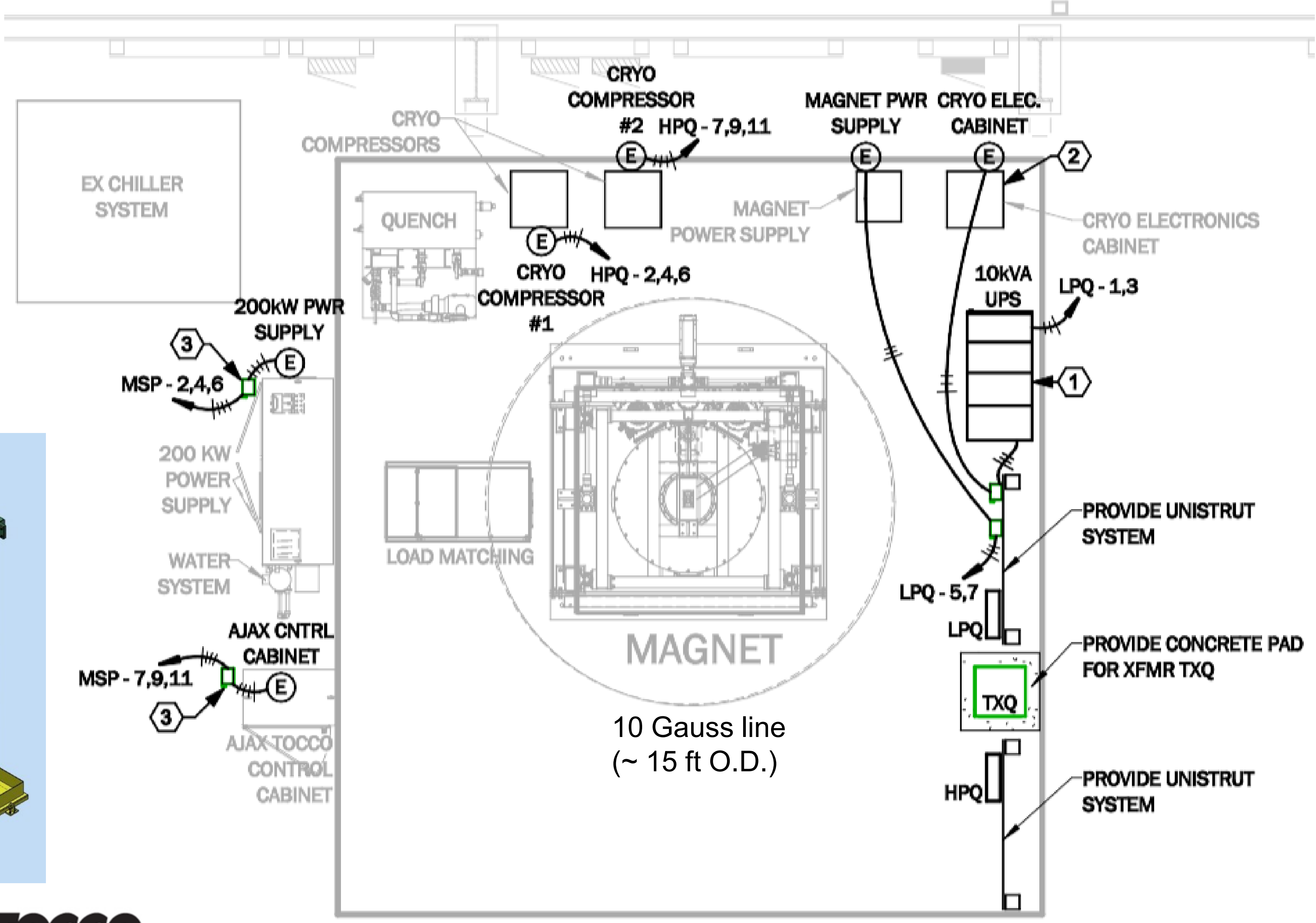
Task 2: Density Functional Theory (DFT) and Calculations of Phase Diagrams (CALPHAD) computational/simulation tools to model the data generated by **Task 1** to generate phase, Time-Temperature-Transformation (TTT), and Continuous-Cooling-Transformations (CCT) diagrams as ***functions of the magnitude of the applied magnetic field***.

Task 3: Design and install an industrial-scale prototype system (***20-inch inner diameter, 2 Tesla superconducting magnet*** with 200 kW induction power supply) to demonstrate ITMP technologies to rapidly accelerate the commercialization of this ***energy efficient***, high-temperature materials processing technology. The system will be installed in UF Department of Materials Science and Engineering.

Development of High Energy Density Thermomagnetic Processing Technology for Intensification of Industrial Heat-Treatment and Increased Material Performance



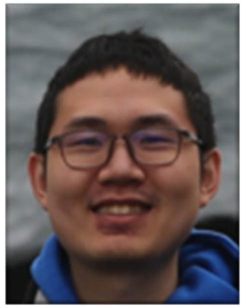




Inside section (60% drawings) of Powell Lab Building, East Campus, UF

Life Cycle Assessment (LCA): Carbon Steel

Harrison Kim



Kangcheng Lin

ILLINOIS

ISE | Industrial & Enterprise Systems Engineering

GRAINGER COLLEGE OF ENGINEERING

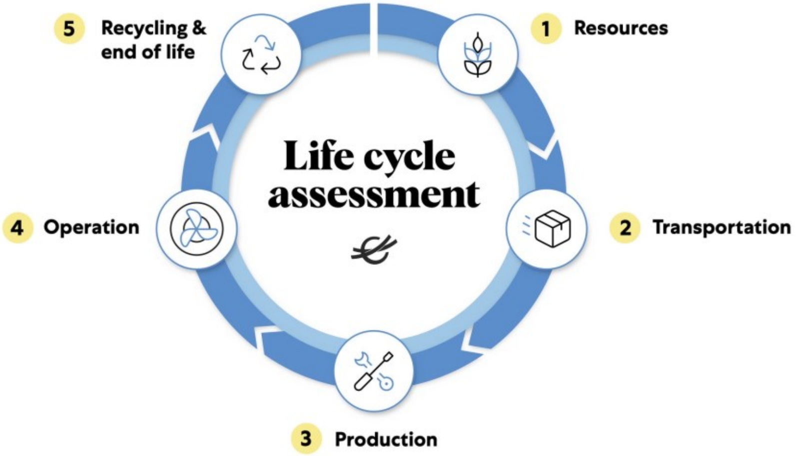


Image Source: https://climeworks.com/

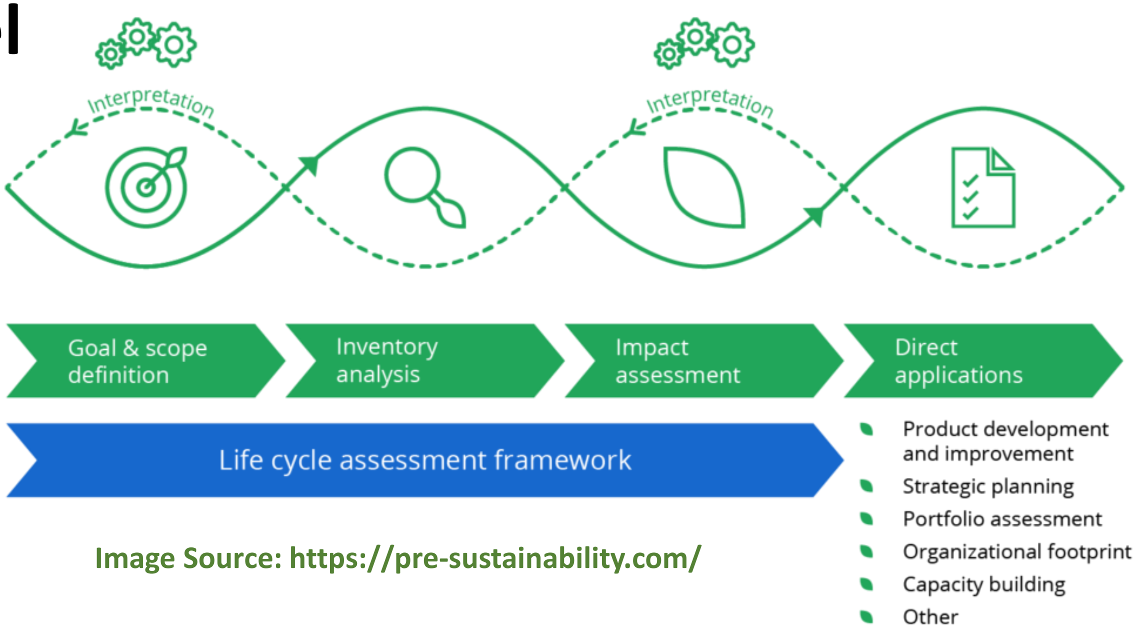


Image Source: https://pre-sustainability.com/

ITMP technique reduces the ***Environmental Impact*** of producing 1 kg of carbon steel by **≈ 90%,** when compared to conventional methods, and by **≈ 86%** for the heat treatment of a specific, 10 kg part specified by an industrial partner.

Preliminary LCA of CO2 production of NbTi with energy sources from coal, natural gas, wind, and fuel. – K. Lin and H.M. Kim, in preparation (2023).

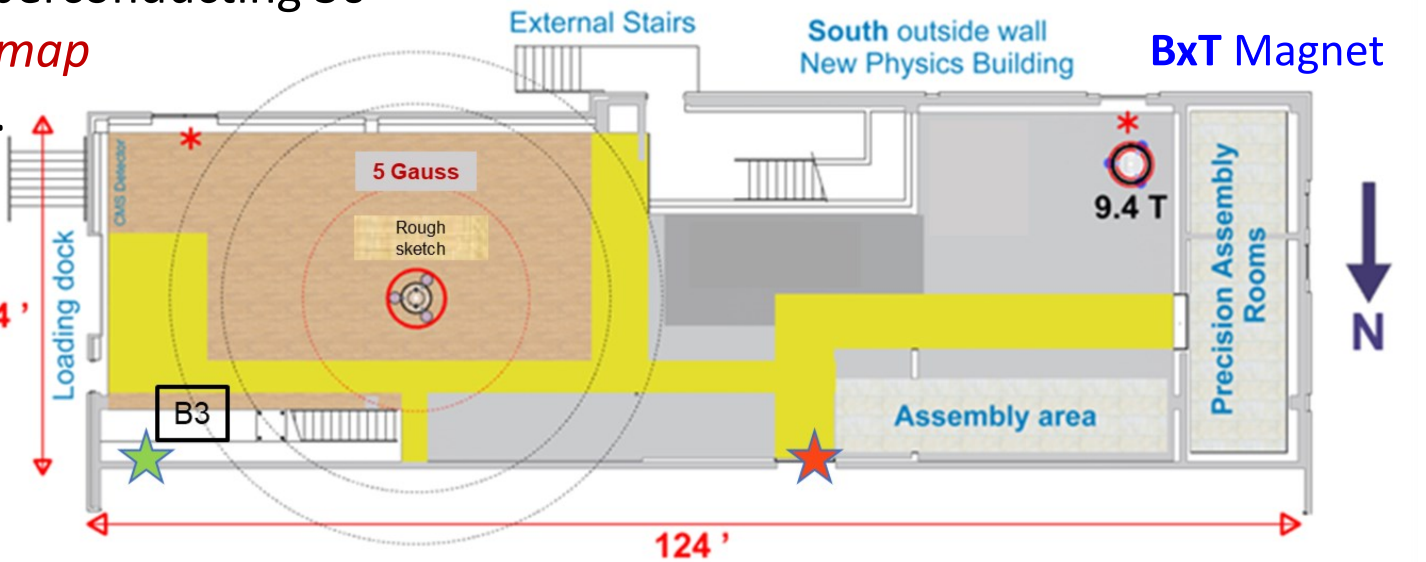
U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy
PI: Michele Manuel DE-EE0009131

High Bay Convergence Lab “a” vision

“a” vision: *Starter Place* for next generation all-superconducting 30+ Tesla magnet and a *“how to / best practices” roadmap* for other locations and AMRIS + HBT User Science.

Present: **BxT Facility** (9 Tesla x 1100º C)
Materials Processing in High B (or B∇B) at High T
Funded by **DOE AMO EERE DE-EE0009131**

PI: Michele Manuel, UF MSE, CoPIs, J.J. Hamlin, M.W. Meisel



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UF UNIVERSITY of FLORIDA · UNIVERSITY OF ILLINOIS URBANA-CHAMPAIGN · OAK RIDGE National Laboratory · VIRGINIA TECH

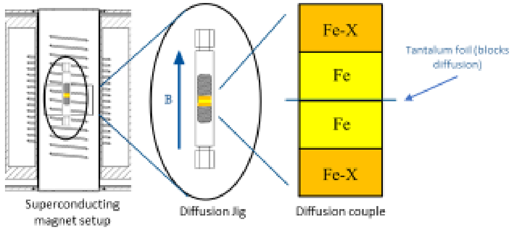
Diffusion Couple Experiments to 9 Tesla: inductive heater at ORNL, resistive furnace in BxT Facility at UF – Data Matrix

Ramon F. Padin-Monroig *et al.*

- In field experiments to be performed at NPB (All Quenched)

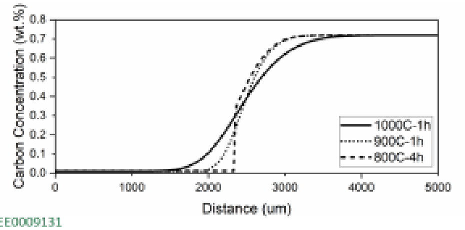
Specimens	Temperature/Time	Field
Fe/Fe-0.63wt.%C	1000°C-1h	0T*, 2T, 5T, 9T
Fe/Fe-0.63wt.%C	900°C-1h	0T*, 2T, 5T, 9T
Fe/Fe-0.63wt.%C	800°C-4h	0T*, 2T, 5T, 9T

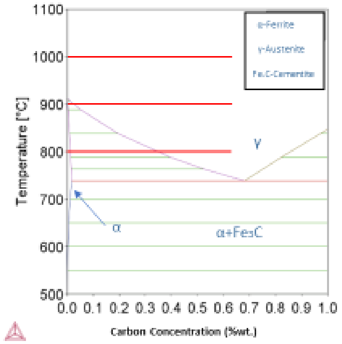
*Lab Box Furnace,
BxT furnace to be scheduled.



Green- Data Acquired   blue- Characterization Ongoing
Orange- Anneal Completed   Red- TBD

DICTRA Simulations based off TCFE12 database





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Task 1 (examples) Fe-C Diffusion Data and Tempered Martensite Hardness guiding
Task 2 numerical-simulation modelling

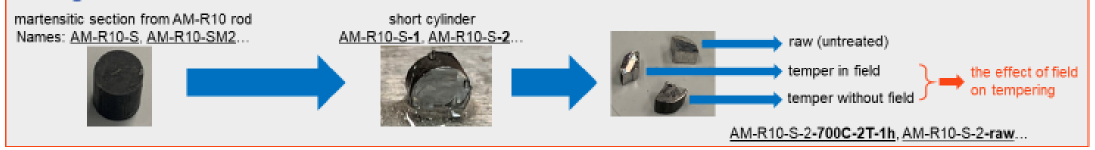
Contrasting Environments

UF BxT: to 9 tesla (resistive element heating)

ORNL: to 9 tesla (inductive heating)

Tempered Martensite Study to 9 Tesla: resistive furnace in BxT Facility at UF – The Sample / Data Matrix

Zhongwei Li *et al.*

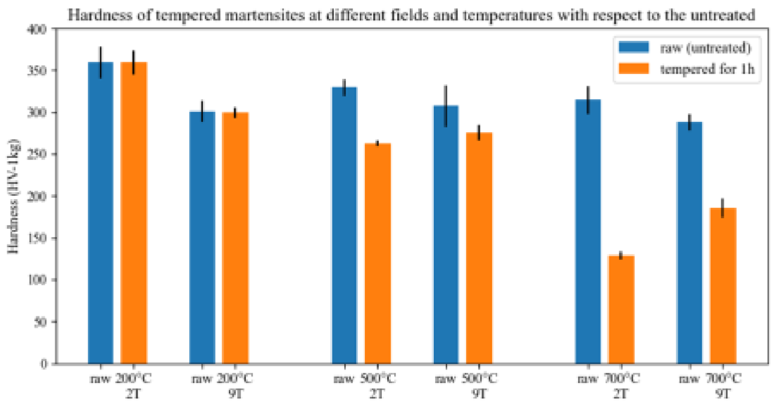


sample name	AM-R10-S-2-			AM-R10-S-3-			AM-R10-S-4-		
	raw	700C-2T-1h	700C-0T-1h	raw	500C-2T-1h	500C-0T-1h	raw	200C-2T-1h	200C-0T-1h
finish date	230228	230302	230808	230228	230301	230717	230225	230301	230711
hardness (HV-1kg)	314±17	129±5	*ongoing*	329±10	263±4	*ongoing*	359±14	359±19	*ongoing*
sample name	AM-R10-M2-3-			AM-R10-M2-2-			AM-R10-M2-1-		
	raw	700C-5T-1h	700C-0T-1h	raw	500C-5T-1h	500C-0T-1h	raw	200C-5T-1h	200C-0T-1h
finish date	230515	230628	230808	230512	230625	230808	230511	230615	230808
hardness (HV-1kg)	*ongoing*								
sample name	AM-R10-SM2-2-			AM-R10-SM2-3-			AM-R10-SM2-1-		
	raw	700C-9T-1h	700C-0T-1h	raw	500C-9T-1h	500C-0T-1h	raw	200C-9T-1h	200C-0T-1h
finish date	230323	230324	230808	230323	230322	230726	230315	230316	230713
hardness (HV-1kg)	288±10	185±12	*ongoing*	307±25	275±9	*ongoing*	301±12	299±6	*ongoing*

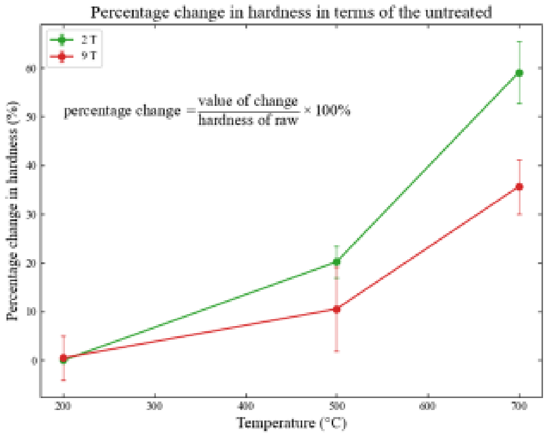
Tempered Martensite Study to 9 Tesla: resistive furnace in BxT Facility at UF – Preliminary Results

Zhongwei Li *et al.*

Hardness of tempered martensites at different fields and temperatures with respect to the untreated



Percentage change in hardness in terms of the untreated



Magnetic field suppresses the softening during tempering.

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PI: Michele Manuel   DE-EE0009131

Carbon-Steel properties vary with heating / magnetic field protocols

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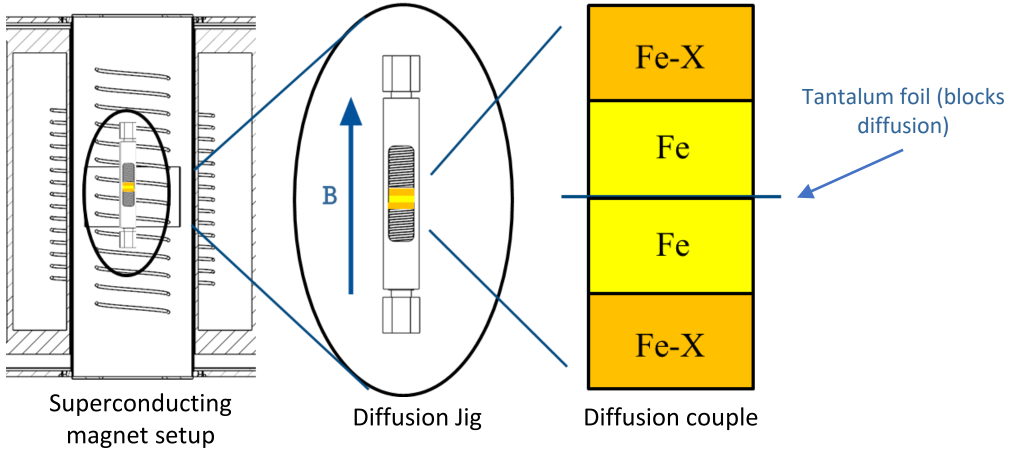
- In field experiments to be performed at NPB (All Quenched)

Specimens	Temperature/Time	Field
Fe/Fe-0.63wt.%C	1000°C-1h	0T*, 2T, 5T, 9T
Fe/Fe-0.63wt.%C	900°C-1h	0T*, 2T, 5T, 9T
Fe/Fe-0.63wt.%C	800°C-4h	0T*, 2T, 5T, 9T

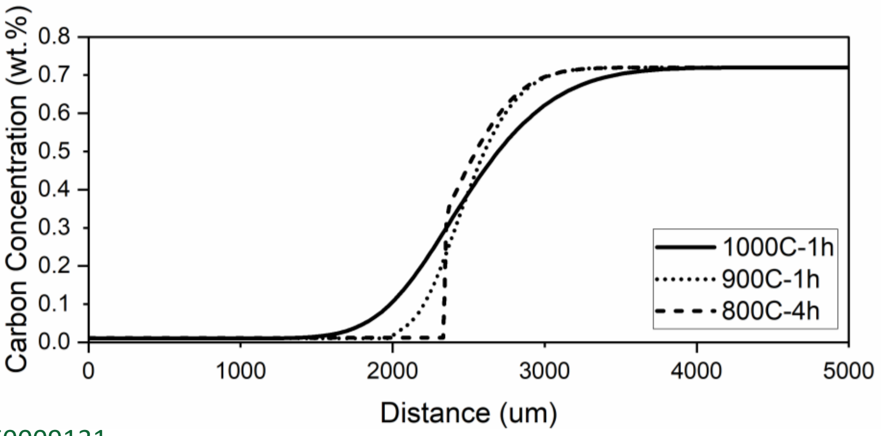
*Lab Box Furnace,
BxT furnace to be scheduled.

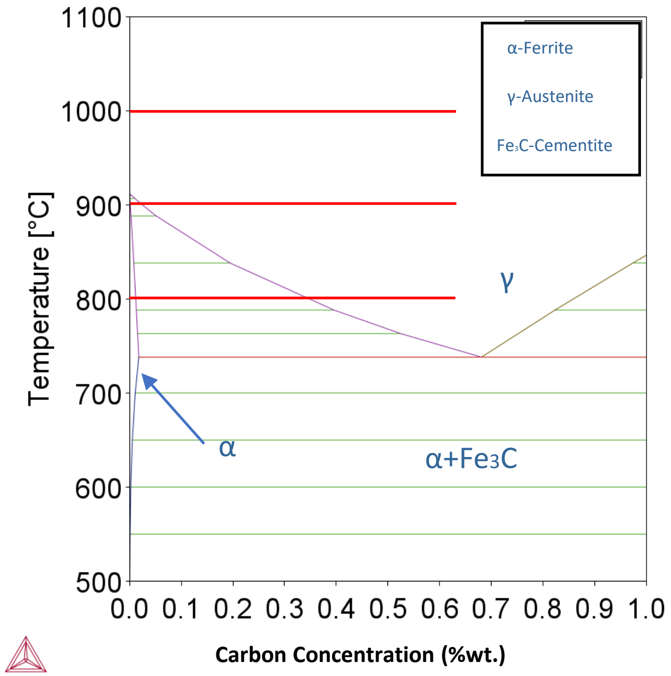


Superconducting magnet setup   Diffusion Jig   Diffusion couple

Green- Data Acquired   blue- Characterization Ongoing
Orange- Anneal Completed   Red- TBD

DICTRA Simulations based off TCFE12 database





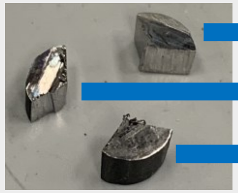
Tempered Martensite Study to 9 Tesla: resistive furnace in BxT Facility at UF – The Sample / Data Matrix

Zhongwei Li *et al.*

sample processing flowchart

martensitic section from AM-R10 rod
Names: AM-R10-S, AM-R10-SM2…

short cylinder
AM-R10-S-**1**, AM-R10-S-**2**…



- raw (untreated)
- temper in field
- temper without field

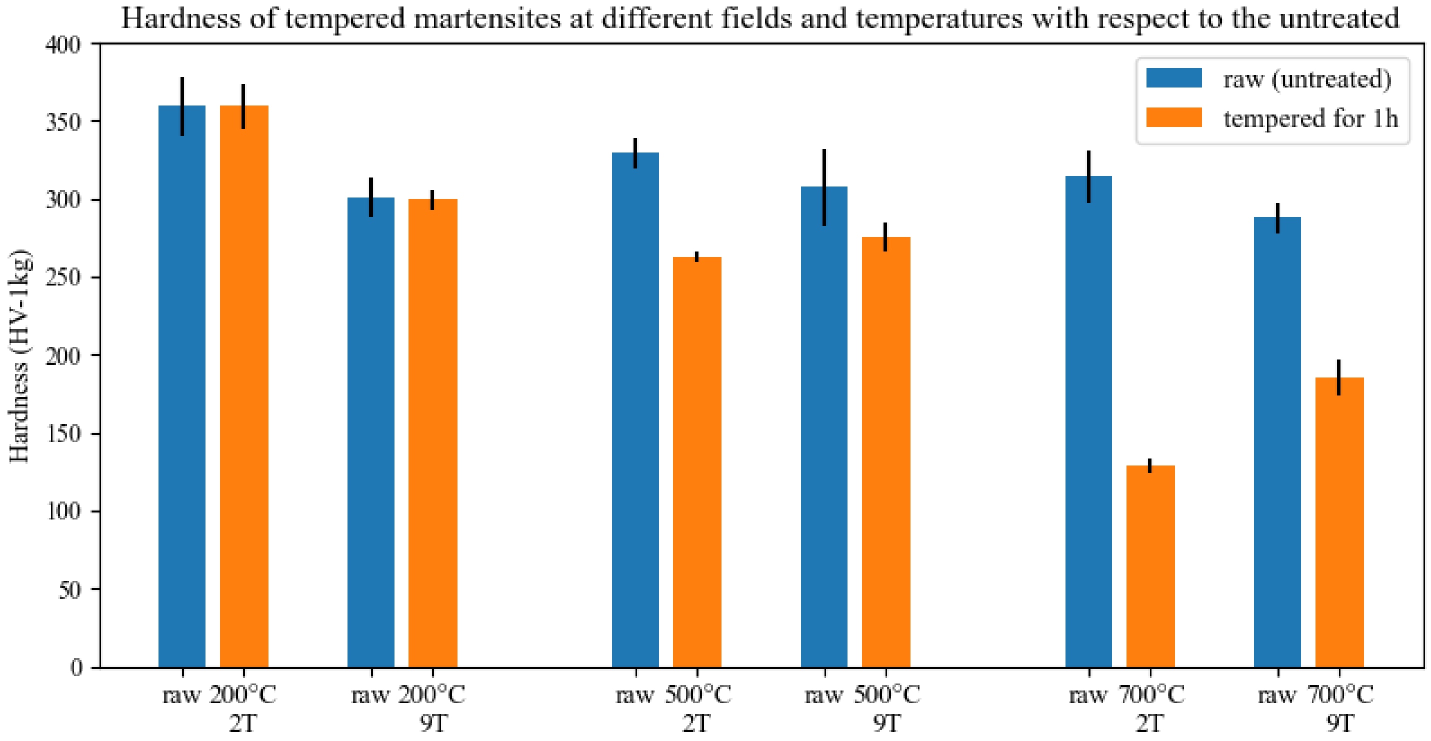
→ the effect of field on tempering

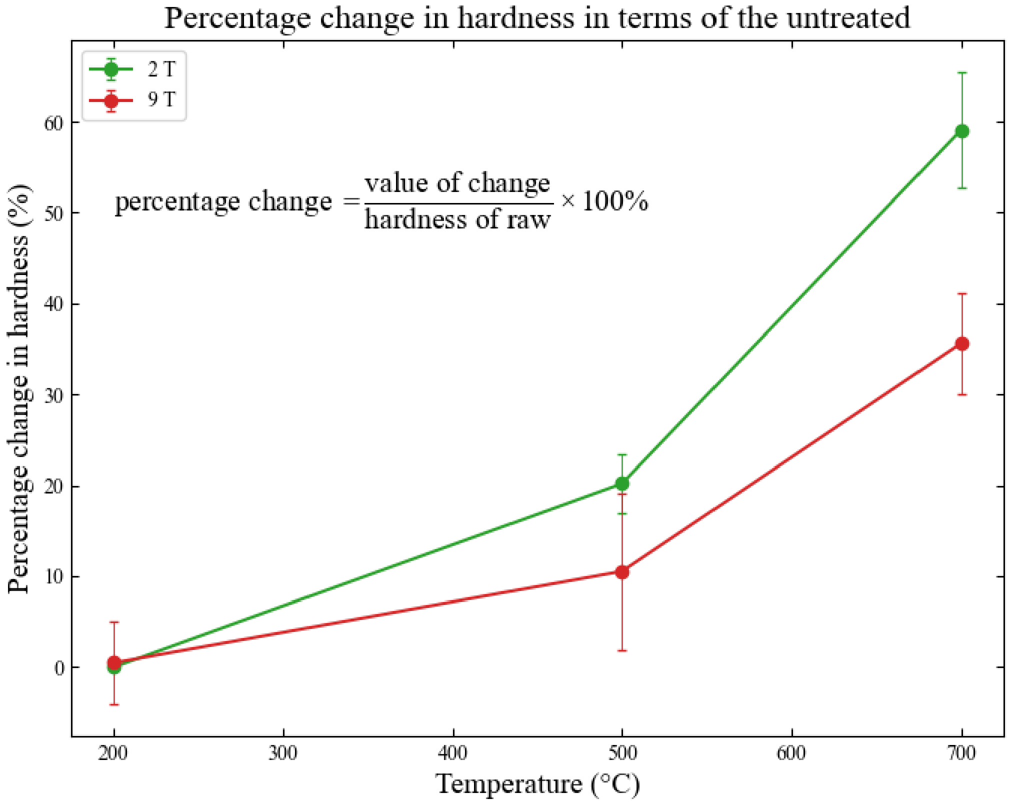
AM-R10-S-2-**700C-2T-1h**, AM-R10-S-2-**raw**…

sample name	AM-R10-S-2-			AM-R10-S-3-			AM-R10-S-4-		
	raw	700C-2T-1h	700C-0T-1h	raw	500C-2T-1h	500C-0T-1h	raw	200C-2T-1h	200C-0T-1h
finish date	230228	230302	230808	230228	230301	230717	230225	230301	230711
hardness (HV-1kg)	314±17	129±5	*ongoing*	329±10	263±4	*ongoing*	359±14	359±19	*ongoing*
sample name	**AM-R10-M2-3-**			**AM-R10-M2-2-**			**AM-R10-M2-1-**		
	raw	700C-5T-1h	700C-0T-1h	raw	500C-5T-1h	500C-0T-1h	raw	200C-5T-1h	200C-0T-1h
finish date	230515	230628	230808	230512	230625	230808	230511	230615	230808
hardness (HV-1kg)	*ongoing*								
sample name	**AM-R10-SM2-2-**			**AM-R10-SM2-3-**			**AM-R10-SM2-1-**		
	raw	700C-9T-1h	700C-0T-1h	raw	500C-9T-1h	500C-0T-1h	raw	200C-9T-1h	200C-0T-1h
finish date	230323	230324	230808	230323	230322	230726	230315	230316	230713
hardness (HV-1kg)	288±10	185±12	*ongoing*	307±25	275±9	*ongoing*	301±12	299±6	*ongoing*

Tempered Martensite Study to 9 Tesla: resistive furnace in BxT Facility at UF – Preliminary Results

Zhongwei Li *et al.*

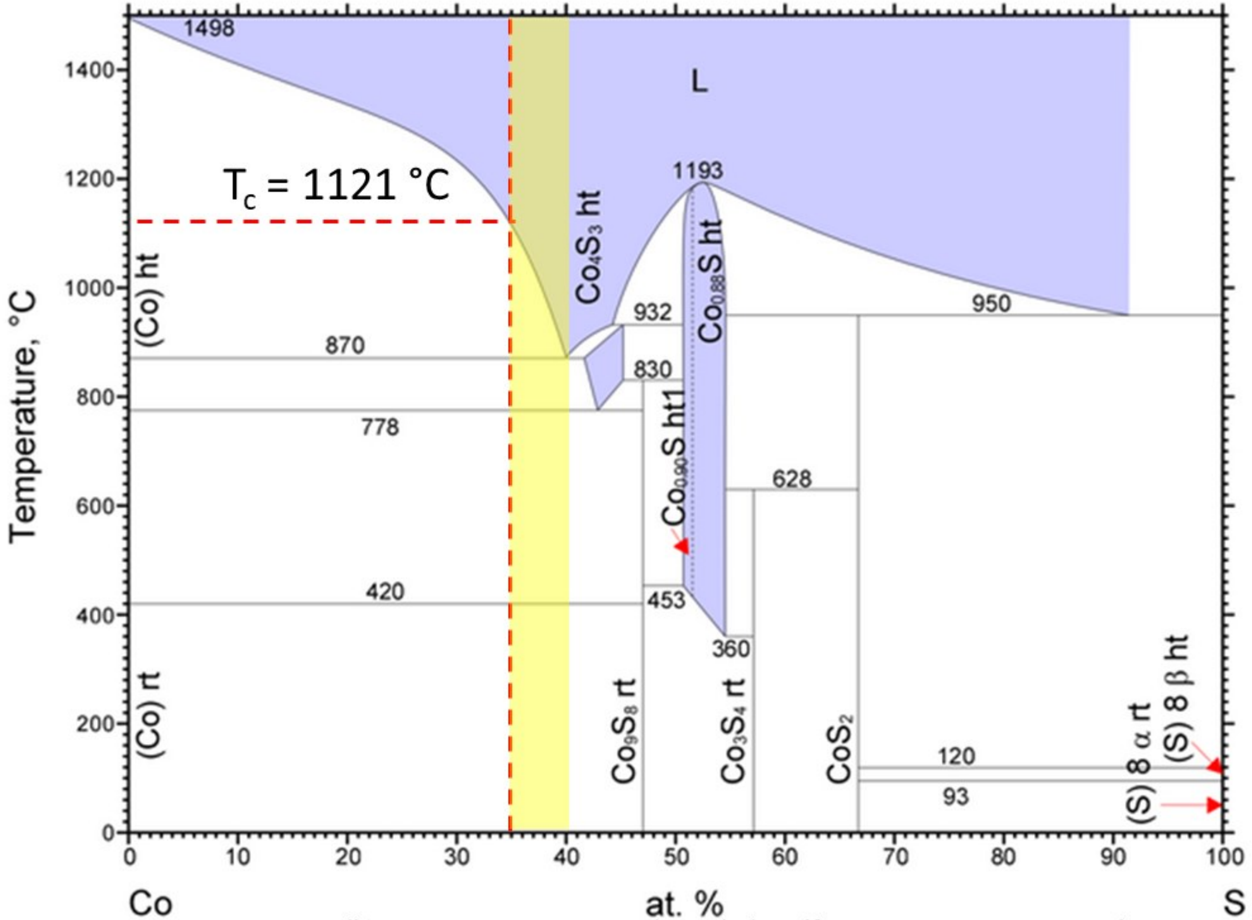




Magnetic field suppresses the softening during tempering.

Magnetic Properties of Ferromagnetic Cobalt Grown in an Applied Magnetic Field

Steven Flynn *et al.*



Kuznetsov, V. G.; Palkina, K. K.; Popova, Z. V. The Cobalt-Sulfur System. *IZV AKAD Nauk SSSR NEORGAN Mater.* **1965**, *1* (5), 675–689.

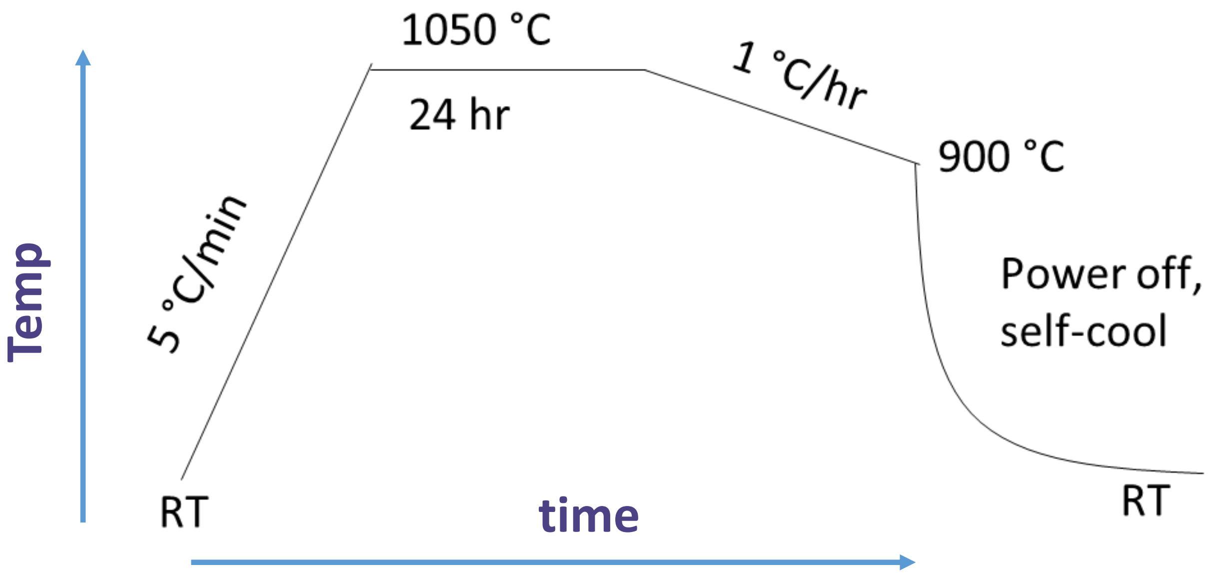
From: T.B. Massalski, *Binary Alloy Phase Diagrams*, 2nd ed., ASM Inter., 1992.

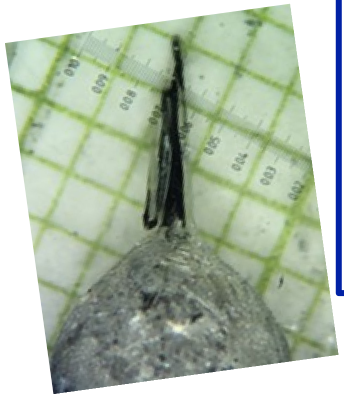
[1] X. Lin, S.L. Bud'ko, P.C. Canfield, *Philos. Mag*, 92 (2012) 2436.

[2] S. Kaya, *Rep. Tohoku Univ.*, 17 (1928) 1157.

MagLab support via NSF DMR-1644779 (2018-2022) and the State of Florida.

Co single crystals can be grown from a Co-S flux below its T_C (ferromagnetic state) [1]





9 T cobalt dendrite and boule.

Crystal structure and magnetic anisotropy [2] characterized and compared experimental saturated moment with *Materials Project*, https://materialsproject.org/.

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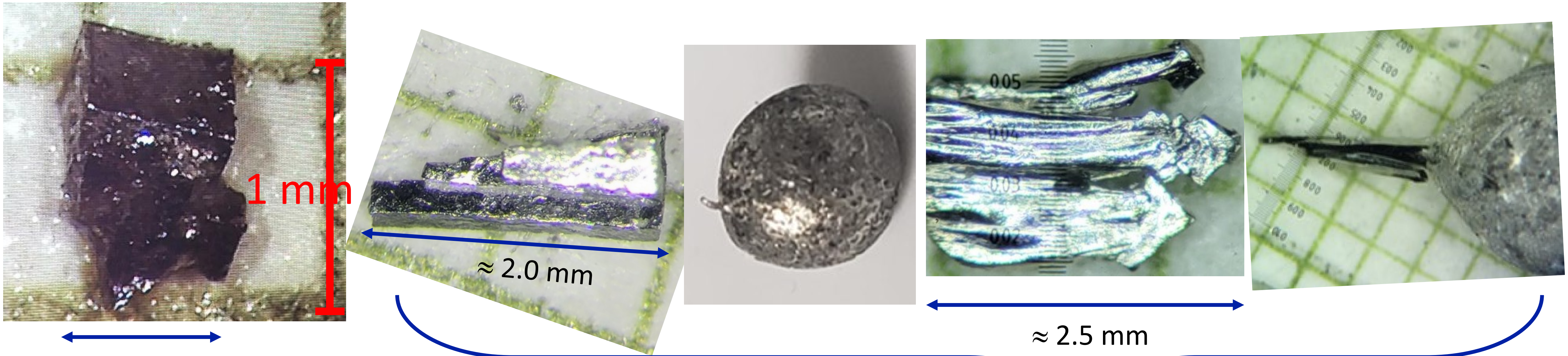
Impacts of an Applied Field on Morphology

Steven Flynn *et al.*

High BxT at UF – Now Open For Collaborative Access

0 T **3 T** **9 T**



Primary Co dendrites parallel to applied field were observed in Co-Sn alloys field-cooled and none observed in absence of applied field [3].

Tendency of grains to align and elongate with applied synthesis field as its magnitude increased.

[3] F. Gaucherand, E. Beaugnon, *Physica B Condens. Matter*, 294-295 (2001) 96.

MagLab support via NSF DMR-1644779 (2018-2022) and the State of Florida.

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The 9th International Workshop on Materials Analysis and Processing in Magnetic Fields, MAP9, will take place in Miyazaki, Kyushu, Japan, from December 11-14, 2023. This series of workshop succeeded in Grenoble (2018), Providence (2016), Okinawa (2014), Grenoble-Autrans (2012), Atlanta (2010), Tokyo (2008), Grenoble (2006) and Tallahassee (2004).

https://www.sci.kagoshima-u.ac.jp/koyama/map9/Welcome.html

Accepted for Oral Presentation: **ITMP Initiative**

Development of High Energy Density Thermomagnetic Processing Technology for Intensification of Industrial Heat-Treatment and Increased Material Performance

Michele V. Manuel [1,†], Michael R. Tonks [1,§], Michael S. Kesler [2,‡], James J. Hamlin [3], Richard G. Hennig [1], Harrison H. Kim [4], Ling Li [5], Zhichao Li [6], Mark W. Meisel [3,7,*], Dallas R. Trinkle [8]

Accepted for Poster Presentation: **Co-S Project**

Texturing of Co Grown from Co-S in High Magnetic Fields

Steven Flynn [1,2,†], Caeli L. Benyacko [1,2], Jared Lee [1,‡], Fuyan Ma [3], Michael Bates [1,§], Shubham Sinha [1], Khalil Abboud [3], Mark W. Meisel [1,3,*], James J. Hamlin [1,*]

MWM to suggest MAP10 (2025) to be in Florida

https://nationalmaglab.org/research/industry/

Development of High Energy Density Thermomagnetic Processing Technology for Intensification of Industrial Heat-Treatment and Increased Material Performance

University / National Lab / Industry Partnership

loukustech

dante

John Deere

Ajax TOCCO Magnethermic Corporation

Caterpillar

Vision Materials

Leadership Team
Michele Manuel (UF)
Michael Tonks (UF)
Michael Kesler (ORNL)

Task 1: Michael Kesler (Lead)
Victoria Miller (UF)
Mark Meisel (UF)
Michele Manuel (UF)
Michael Tonks (UF)

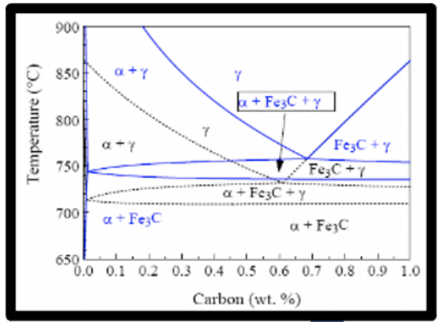
Task 2: Michael Tonks (Lead)
Richard Hennig (UF)
James Hamlin (UF)
Dallas Trinkle (UIUC)
Ling Li (VT)
Charlie Li (DANTE)

Task 3: Michele Manuel (Lead)
Mark Meisel (UF)
Michael Kesler (ORNL)
Harrison Kim (UIUC)

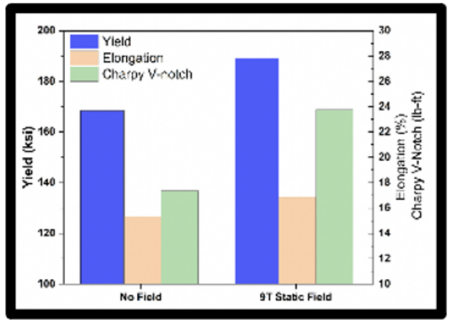
BRIDGE
Michael Tonks
James Hamlin

BRIDGE
Mark Meisel
Michael Kesler

Alters thermodynamics and kinetics



Improves properties and yields high energy savings



DOE Acknowledgements and Disclaimers

Acknowledgement

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