Investigation of Ionic Liquid Isolated Iron for Ductile Iron Castings

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About the Presenter

- **Blake C. Stewart**
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  - **Ph.D. candidate**
  - **B.S. Mechanical Engineering**
- **Highlights and Interests**
  - **Steelmaking and cast iron**
  - **In-situ resource utilization (ISRU)**
  - **Extraterrestrial manufacturing methods**
  - **Experience in manufacturing, field engineering, and experimental research**

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About the Presenter

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Motivation

- In-situ resource utilization (ISRU) crucial for future exploration and colonization beyond low-Earth orbit
- Mechanical components needed from resources available in Lunar and Martian environments
- Martian regolith rich in metallic elements but found in silicates and oxides

Team SEArch+/Apis Cor rendering of structure manufactured on Martian surface [1]

[1] NASA, Team SEArch+/Apis Cor, Phase 3: Level 4 software model

Overview

- Chemical analysis to determine the composition of
  Ionic liquid-sourced iron (IL-Fe)
- Ductile iron ingot cast using commercial materials with a composition simulating the use of IL-Fe
  - Carbon (C) sourced from Bosch reactor at MSFC
  - Cast material referred to as Sim-IL ductile iron (DI)
- Produced ingot compared to commercially available ductile iron
  - Characterized microstructure
  - Continuous cooling transformation (CCT) and time temperature transformation (TTT) diagrams
  - Mechanical property (hardness)
- Investigation suggested quality ductile iron can be produced from IL-Fe and Bosch C

Literature Review: Ionic Liquids, Bosch Carbon, and Martian Regolith

- **Ionic liquids (IL) currently studied at MSFC to extract and recover metallic elements [2]**
- **Bosch process currently studied at MSFC as a life support system for oxygen regeneration with a by-product of elemental C [3]**

Average Chemical Composition of Martian regolith [4-9]	
SiO_2 (n=40)	47.75 ± 6.08
MnO (n=20)	0.31 ± 0.15
Fe_xO_x (n=40)	15.11 ± 4.15
Ni (n=11)	0.0192 ± 0.01
MgO (n=40)	6.04 ± 2.07
Al_2O_3 (n=40)	12.13 ± 4.76
CaO (n=40)	7.80 ± 1.96
Cr_2O_3 (n=14)	0.24 ± 0.18
	(wt.%) ± st.dev

[2] E. Fox, et. al, Astronomy on Tap Club, May 2019
[3] M. B. Abney, et. al, ICES, 2012.
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Literature Review: Ductile Iron Alloying Elements

- Iron (Fe), C, silicon (Si) and manganese (Mn) are primary elements in ductile iron
  - Magnesium (Mg) needed to “spheroidize” graphite [10]
    - Characteristic feature separating gray cast iron from ductile iron
- Phosphorus (P), often considered an impurity, increases castability, machinability, and tensile properties [11]
- Nickel (Ni) increases tensile properties, hardness at the expense of ductility [12]
  - Elongation and impact energy increase with Ni up to 0.71 wt.% [13]
- Less than 0.55 wt.% molybdenum (Mo) can increase Ni effects on hardness, and decreases ductility [14]
  - Omitted as variable due to desire for minimal alloying

[10] Hot Topics, Ductile Iron Society, 2003.
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[12] C. Hsu, et. al, Mater. Sci. Eng. A, 2007.
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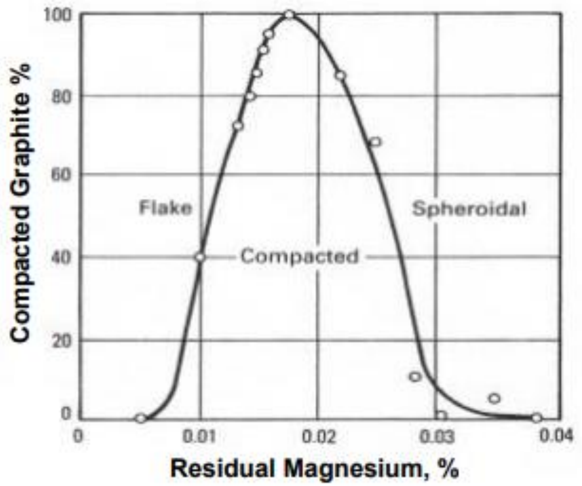
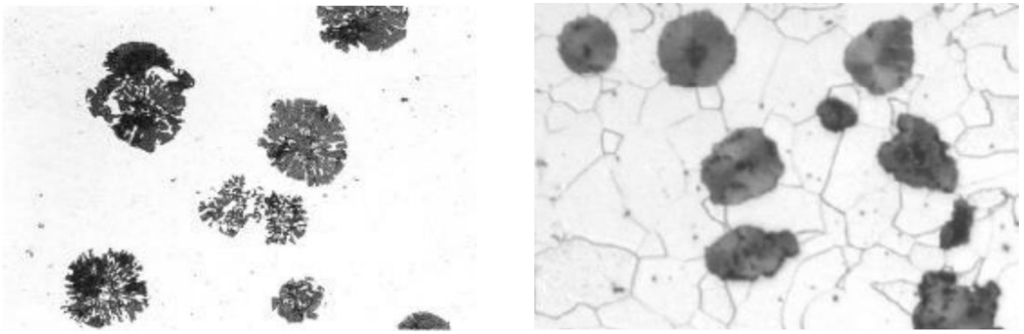
Literature Review: Mg in Ductile Iron[10]

- Mg is necessary to spheroidize graphite from flakes to nodules
- Mg requirement varies with sulfur (S) composition
- Forms MgS first
- "Residual Mg" is excess after sulfides form
- %Mg = 0.020 + ¾ (%S)
- Too much Mg results in "exploded" graphite, porosity, and degraded performance
  - Max of 0.040 wt.%

[10] Hot Topics, Ductile Iron Society, 2003.



Effects on graphite shape with varying Mg



Exploded graphite (left) [10], spherical graphite (right)

Literature Review: Magnesium Additions to Ductile Iron

- Ingot was cast using pure (99+ %) bulk material at MSU
- Mg sourced from commercially available “master alloy”
  - Master alloys used where a low melting temperature elements is required
  - Mg vapor temperature is ~1100 ˚C while melting temperature of pure Fe is ~1600 ˚C
- Master alloy reduces volatile reaction, enabling for greater recovery rates when casting [15]
  - Recovery rate obtained within this experiment was ~33%

5.5Mg-48Si-Fe w/ trace lanthanum (La) and Ca

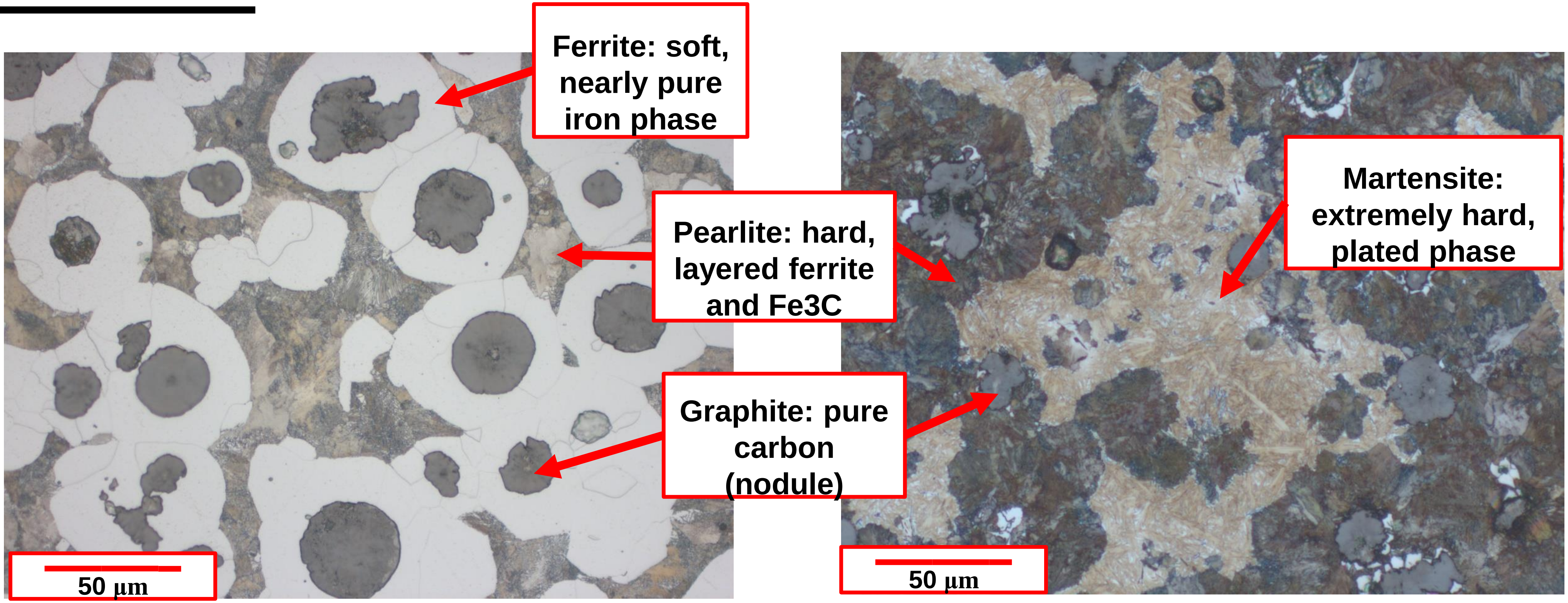
[15] kbmaffilips.com, 2020

Initial Evaluation of Commercial Ductile Iron

- Two grades of ductile iron purchased [16-17]
  - 65-45-12 and 100-70-03
    - Nearly identical chemical composition
  - Naming convention from minimum properties
    - (Tensile strength in ksi – yield strength in ksi – elongation in percent)
- Sample of 100 grade melted and cooled at 1.0˚C/s and compared to 65 grade indicated both grades were produced with heat treatment alone

[16] “Dura-Bar 65-45-12 Continuously Cast Ductile Iron Bar Stock ASTM A536”, Matweb.com, 2019.
[17] “Dura-Bar 100-70-03 Continuously Cast Ductile Iron Bar Stock ASTM A536”, Matweb.com, 2019.

Microstructural phases typically present in ductile iron

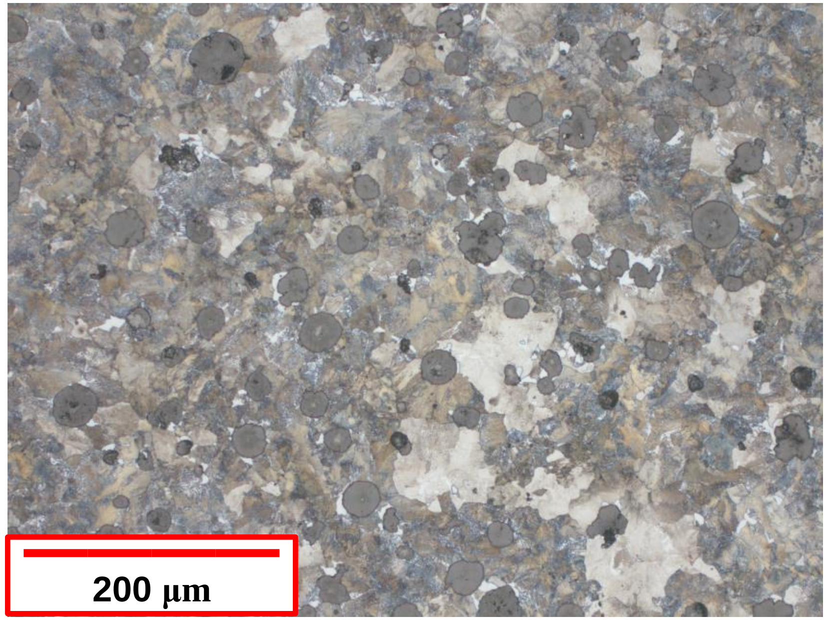


65-45-12 cooled at 1°C/s

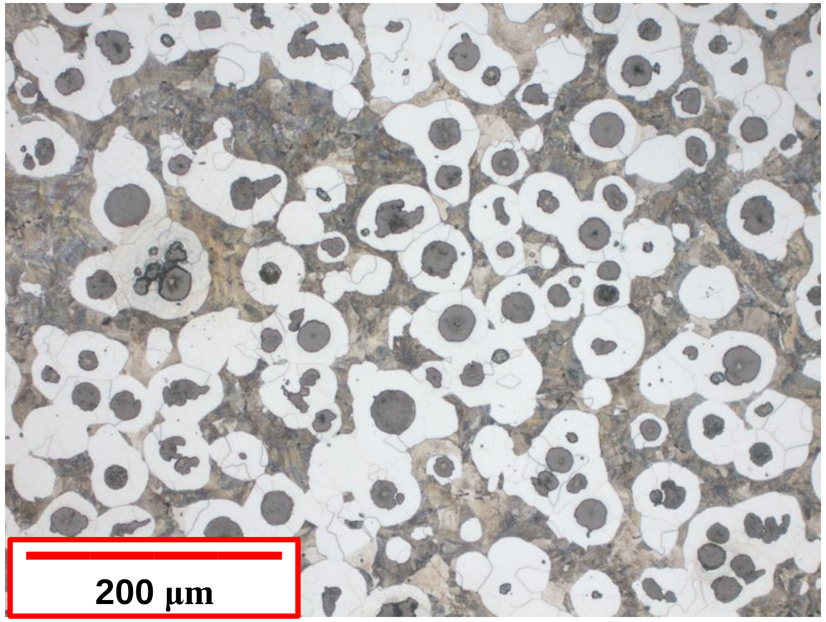
65-45-12 cooled at 10°C/s

Phases formed due to different cooling rates and/or elemental additions. I.E., Ferrite, pearlite, martensite (slow to fast cooling).

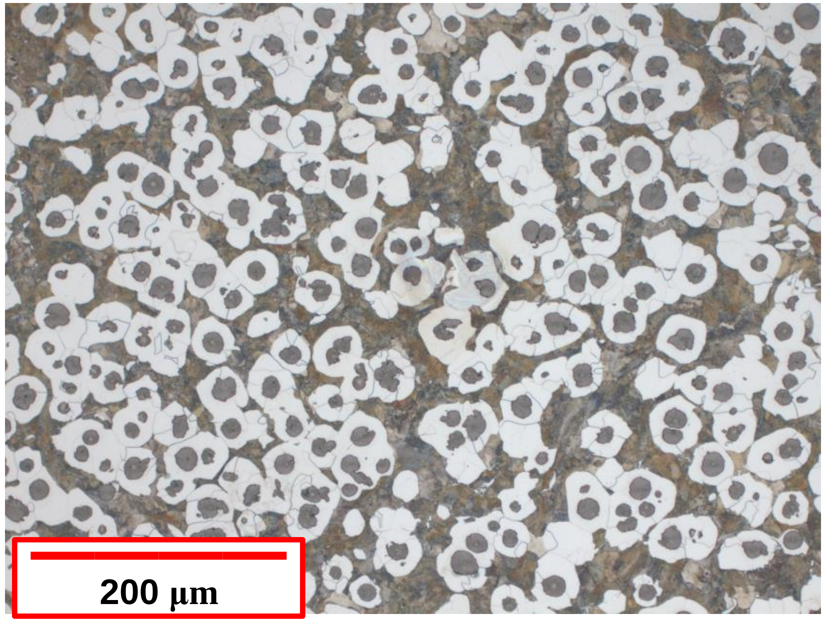
Testing suggested both commercial grades are similar with differences attributed to heat treatment



100-70-03 As-received



100-70-03 recast and cooled at 1.0°C/s

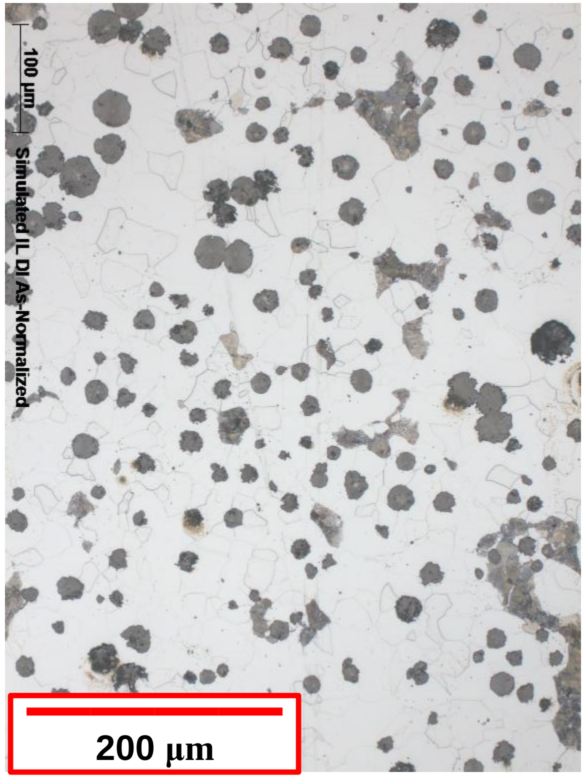


65-45-12 As-received

Hereafter, only 65-45-12 is used for analysis and comparison

Sim-IL DI As-Normalized vs Commercial Ductile Iron

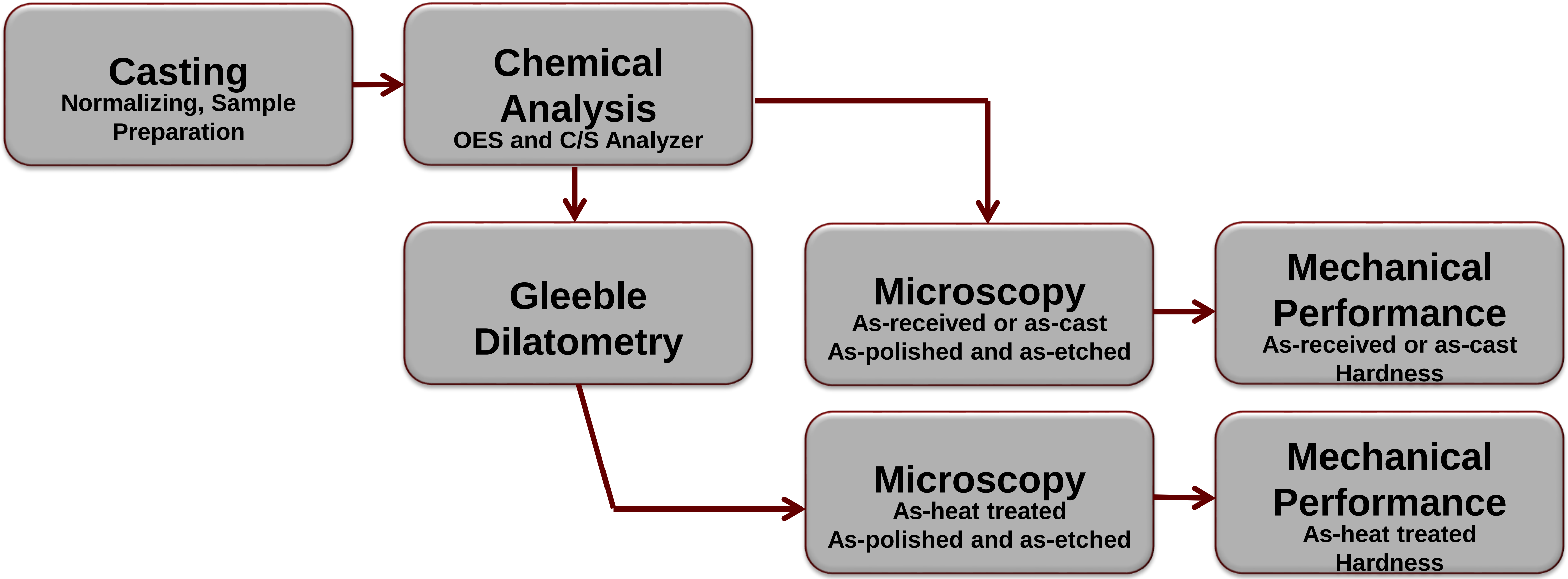
- **Sim-IL DI was cast then normalized to homogenize microstructure**
  - Soaked in 900°C furnace for 2 hours, removed and air cooled
- **Sim-IL DI showed more ferrite (white) than commercial**
  - Likely due to a faster cooling rate upon casting/heat treating of commercial material
- **Difficult to compare properties due to unknown heat treatment parameters of commercial ductile iron**



Sim-IL DI As-normalized

65-45-12 As-received

Experimental Evaluations



Chemical Composition

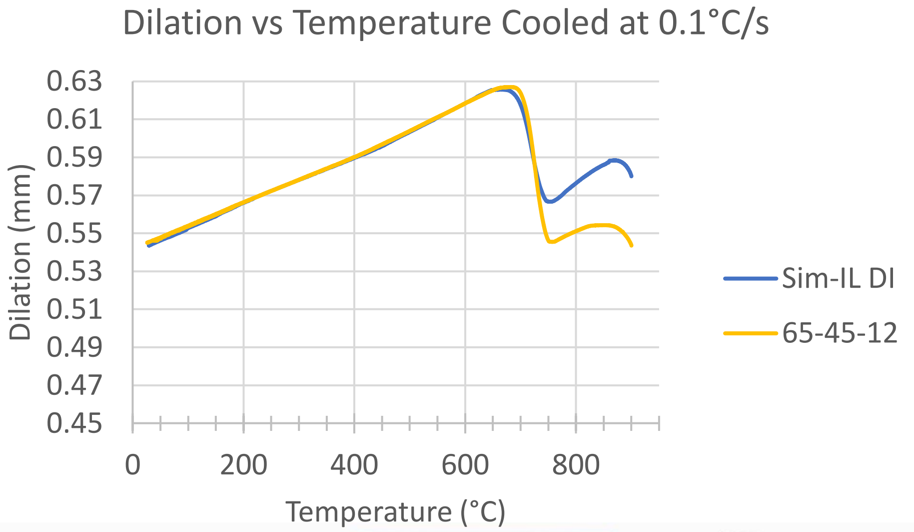
- **Chemical composition acquired via optical emission spectrometry (OES) and carbon/sulfur analyzer**
- **Commercial ductile iron compared to the target and resultant Sim-IL DI ingot as well as the composition of raw IL-Fe**
- **C difference effect negligible or offset based on properties found (lower graphite volume in Sim-IL DI)**

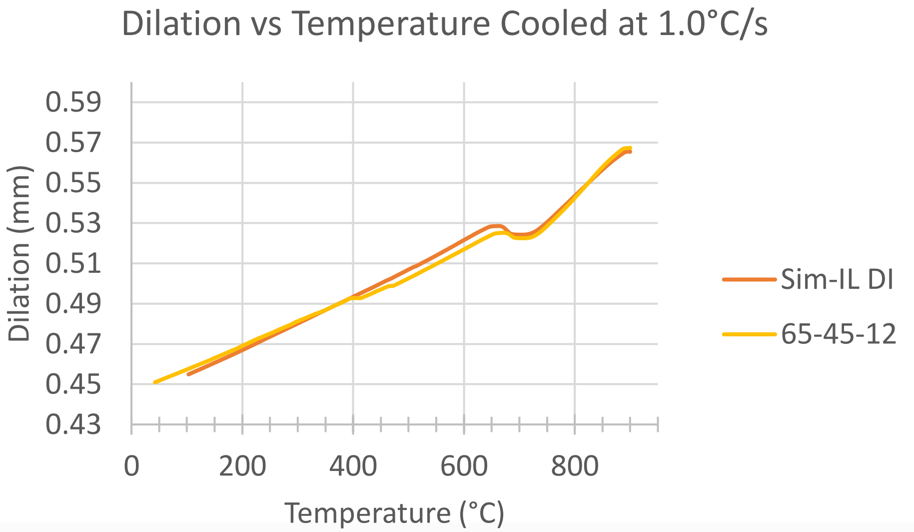
Chemical Composition Comparisons				
	Commercial	IL-Fe (n=3)	Sim-IL DI Target	Sim-IL DI Result (n=10)
C	3.83	-	3.83	3.35 ± 0.0
Si	2.65	1.40 ± 0.3	2.65	2.58 ± 0.1
Mn	0.24	0.47 ± 0.1	0.46	0.31 ± 0.0
Ni	-	0.14 ± 0.0	0.13	0.12 ± 0.0
Al	-	0.11 ± 0.1	0.14	0.04 ± 0.0
Co	-	0.08 ± 0.0	0.08	0.06 ± 0.0
Mg	0.035	2.27 ± 0.3	0.035	0.030 ± 0.00
Na	-	0.04 ± 0.0	0.04	-
Ca	-	0.11 ± 0.1	0.13	0.00 ± 0.0
Fe	bal	bal	bal	bal

(wt.%) ± st.dev

Dilatometry – How volume changes over time

- **9 mm OD x 25 mm L samples were heated to 900°C and held for 30 min to normalize microstructure and allow C to saturate austenite from graphite nodules**
  - **Austenite = common phase start point for heat treatment**
- **Cooling rate maintained (for continuous cooling transformation) with diameter monitored to visualize phase transformation temperatures**





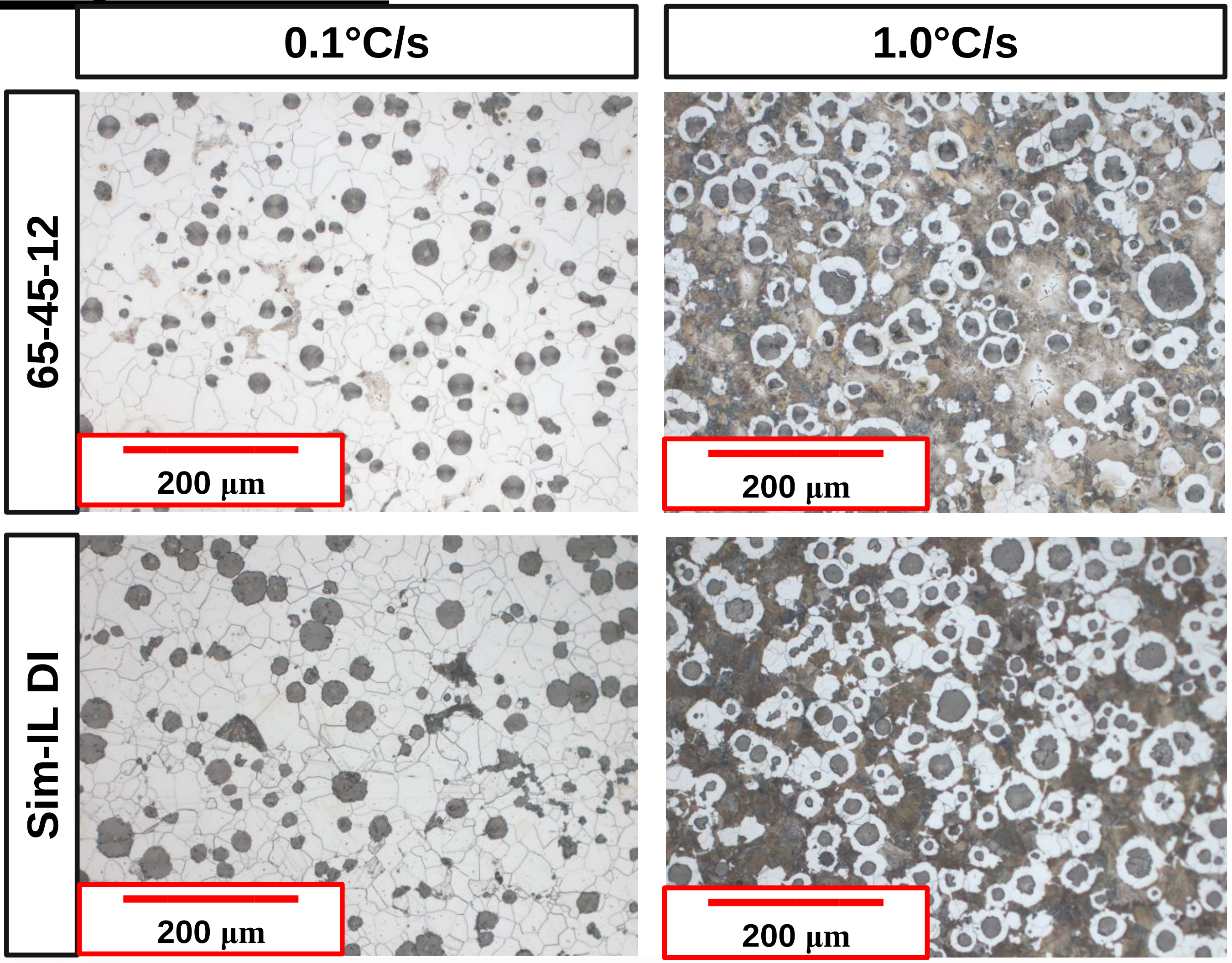
Transformation temperatures showed minimal difference

- **Transition temperatures were within 10°C for all**
- **This round of tests showed minimal differences in transition temperatures for the heat treatment rates completed**

Transformation Temperatures				
Rate	Mat'l	F_S	F_F,P_S	P_F
0.1°C/s	65-45-12	760	682	649
	Sim-IL DI	761	677	650
	Diff.	-1	5	-1
1.0°C/s	65-45-12	753	673	649
	Sim-IL DI	756	668	642
	Diff.	-3	5	7

Microstructures showed slightly greater fraction of harder phases

- Sim-IL DI showed slightly greater area fraction of harder phases, suggesting it could more readily respond to heat treatment



Microstructural area fraction verified harder phases

- **Higher pearlite content in Sim-IL DI**
  - **Could be due to increased Ni and Mn**
- **Lower graphite fraction in Sim-IL DI due to reduced carbon quantity in material**
  - **Lower recovery rate for Bosch C due to fine powder morphology versus larger chunks of commercial C feedstock**

Area Fraction Averages (n=5)				
Rate	Mat'l	Graphite	Ferrite	Pearlite
0.1°C/s	65-45-12	13.0	81.6	5.4
	Sim-IL DI	10.2	73.9	15.9
	Diff.	2.8	7.7	-10.5
1.0°C/s	65-45-12	11.7	35.0	53.3
	Sim-IL DI	10.3	32.0	57.6
	Diff.	1.4	3	-4.3

Mechanical Testing: Hardness

- **Hardness measured and converted across Brinell (HB), Rockwell-B (HRB), and Rockwell-C (HRC)**
- **Hardness values approximately equal**

Area Fraction Averages (n=5)				
Rate		HB	HRB	HRC
0.1°C/s	65-45-12	151	81	1
	Sim-IL DI	148	80	0
	Diff.	3	1	1
1.0°C/s	65-45-12	219	96	20
	Sim-IL DI	211	95	18
	Diff.	8	1	2

Measured, conversion

Sim-IL DI vs Commercial DI

- Sim-IL DI
  - More pearlite
  - Higher highness
    - Greater tensile strength
    - Lower ductility
  - Lower graphite fraction
    - Less mass fraction carbon

- Commercial 65-45-12
  - More ferrite, less pearlite
  - Lower hardness
    - Lower tensile strength
    - Greater elasticity
  - Higher graphite fraction
    - More mass fraction carbon

- Sim-IL DI performance difference possibly due to greater Ni and Mn composition
- Sim-IL DI potentially obtains similar properties as commercial material with slower cooling rates

Summary

- Dilatometry and CCT results showed similar phase transitions
  with some variation attributable to presence of Ni and larger Mn composition
- Hardness and microstructure showed Sim-IL DI responds well to heat treatment
- The use of IL-Fe and Bosch C as casting feedstock could produce ductile iron with equivalent properties to commercial ductile iron

- In summary, the use of Bosch C with IL-Fe is likely a viable option to manufacture ductile iron on the Lunar or Martian surfaces with some limitations

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