Molded Graphite Products Synthesized from Waste-Coal

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Content

- Introduction to Touchstone
- Project Overview
- Technology Background
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Who We Are

- Touchstone's "Coal-to-Products" technology achievements
  - High Density Carbon US-7,824,645
  - Twenty-seven (27) Carbon Foam related patent awards
  - Silicon Carbide US-11,186,522, 1 patent pending

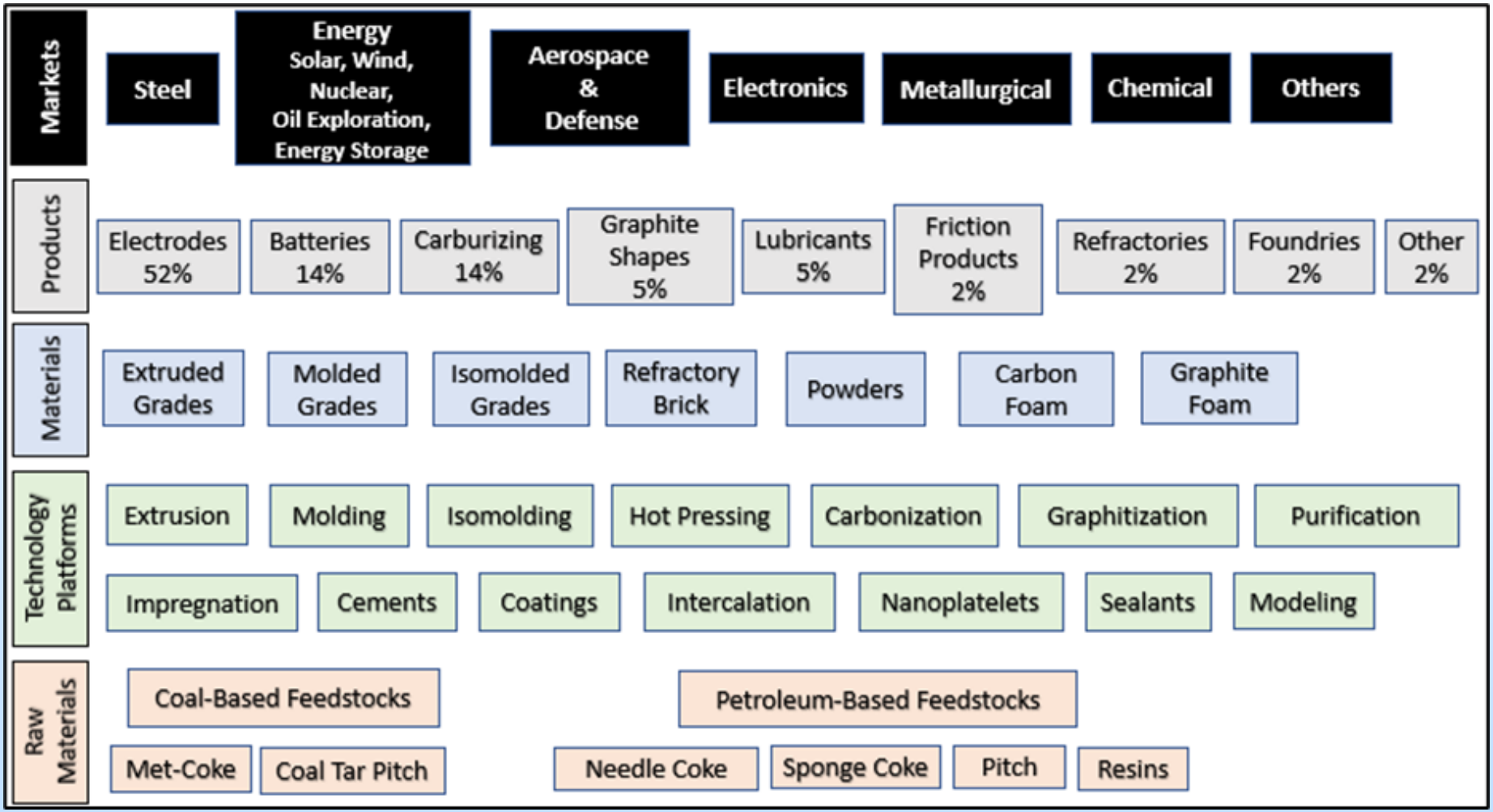
Project Overview

- Funding (DOE and Cost Share)
  - Federal = $999,990
  - Non-Federal = $250,000
  - Total Project = $1,249,990
- Period of Performance: 06/01/2022 - 02/28/2025
- Project Participants
  - Recipient: Touchstone Research Laboratory
  - Sub-recipient: Virginia Tech University

Project Objectives

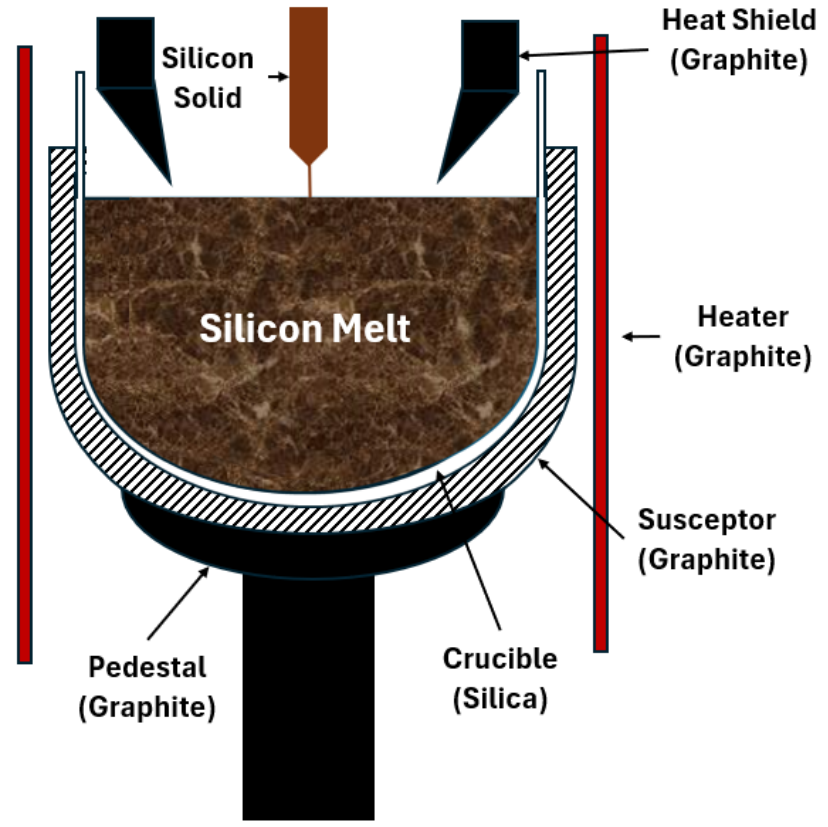
- Develop high density molded graphite from domestic waste-coal feedstock
  - Impurity ≤ 1.0% when cleaned via HHS
  - Moisture content ≤1.0%
- Coal feed particles < 40μm
  - Demonstrate processed feed is suitable for molded graphite processes
  - Newly developed graphite materials should be competitive to conventional graphite synthesized from petroleum cokes
    - Properties
    - Cost
- Transition the technology from laboratory scale proof of concept (TRL-3) to pilot scale (TRL-5).

Carbon and Graphite

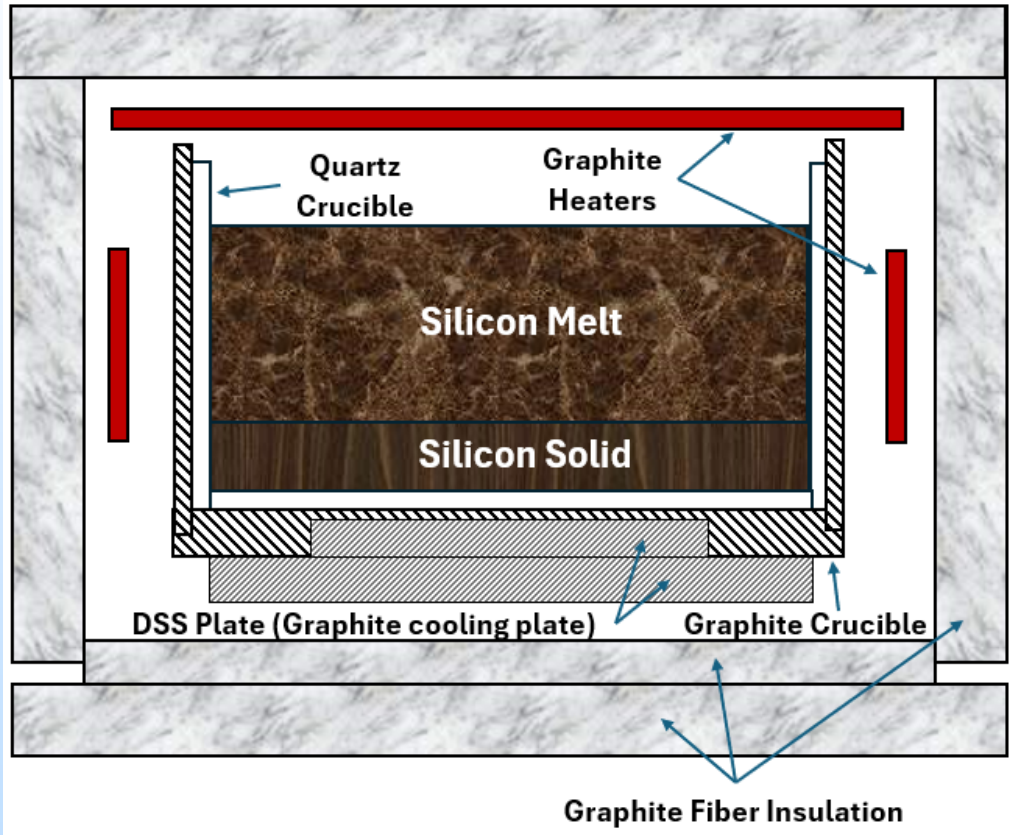


Silicon Production

Monocrystal
Czochralski Method (CZ)



Polycrystal
Directional Solid Solidification (DSS)



Graphite Classification

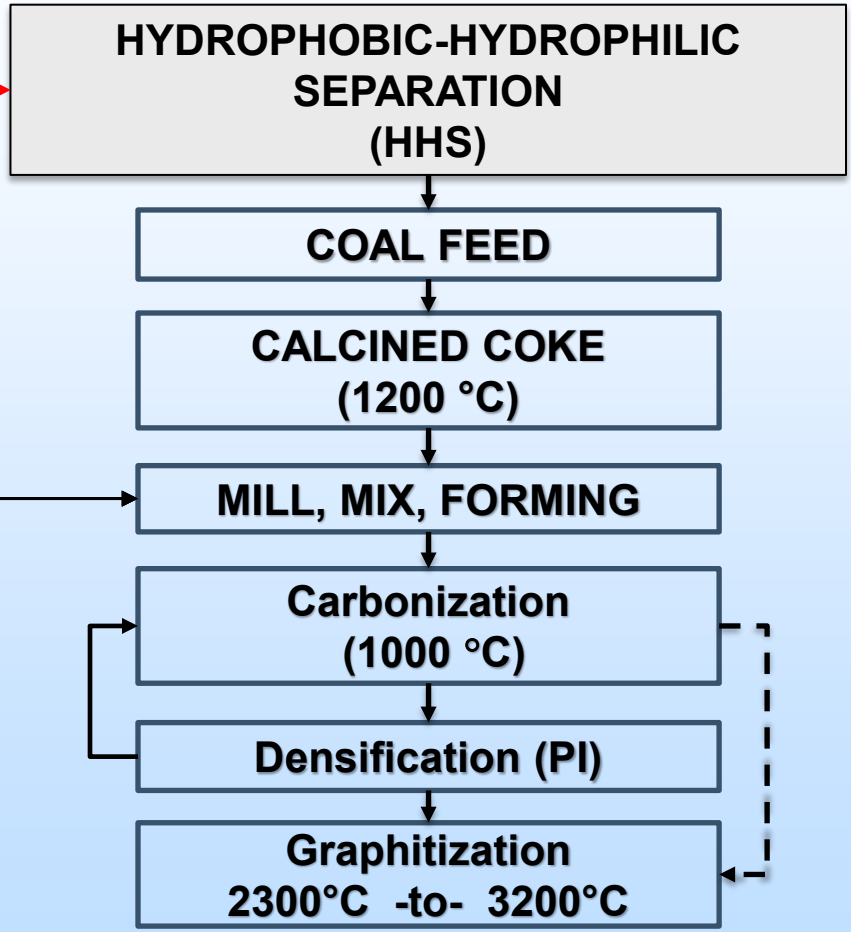
Class	ASTM D709	ASTM D7301[3,4]	Other – Specialty (Average Particle Size) [1]
Coarse	> 4 mm	Generally, > 4mm	> 100 µm
Medium-Coarse	N/A	2 mm < grain size < 4 mm	N/A
Medium	< 4mm	1 mm < grain size < 2 mm	21 µm – 100 µm
Medium-Fine	N/A	100 µm < grain size < 1 mm	N/A
Fine	< 100 µm	50 µm < grain size < 100 µm	11 µm – 20 µm
Superfine	**< 50 µm**	**10 µm < grain size < 50 µm**	**6 µm - 10 µm**
Ultrafine	< 10 µm	2 µm < grain size < 10 µm	1µm - 5 µm, depending on grade
Microfine	-	Generally, < 2 µm	N/A
Angstrofine	N/A	N/A	< 1 µm

Note: **Grain**, in manufactured carbon and graphite, particle of filler material (usually coke or graphite) in the starting mix formulation. Also referred to as granular material, filler particle, or aggregate material.

REFERENCES:
[1] Morgan, D., *Properties and Characteristics of POCO EDM Graphite,* International Symposium of Electro Machining (ISEM XIII), (May 2001)
[2] ASTM D709, titled "Standard Terminology Relating to Manufactured Carbon and Graphite.
[3] ASTM D4175, titled "Standard Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants".
[4] ASTM D7301, titled "Standard Specification for Nuclear Graphite Suitable for Components Subjected to Low Neutron Irradiation Dose.

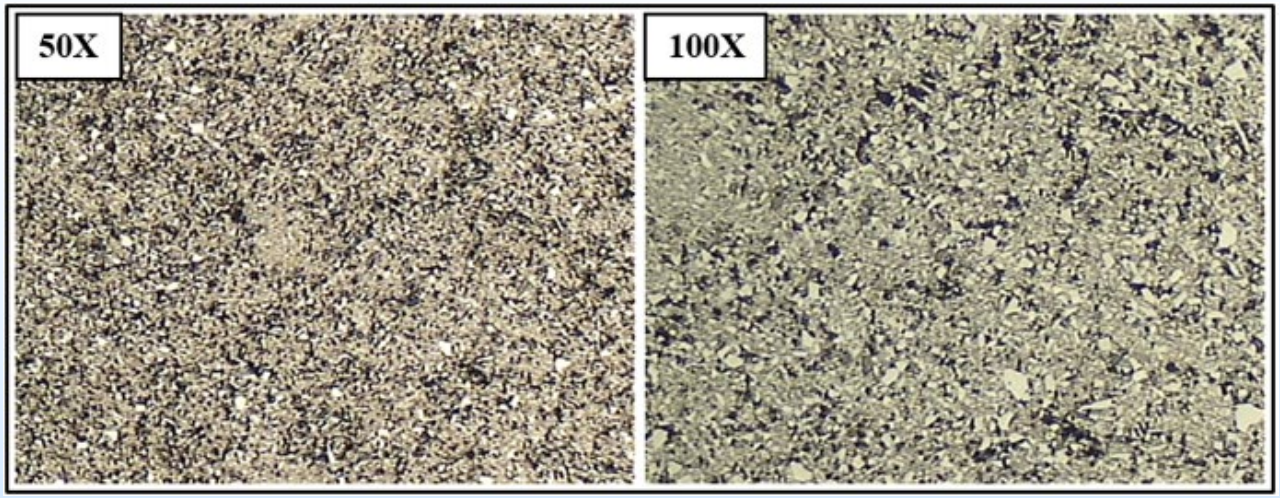
Process Summary

FACTOR	CRITERIA
Filler	• Impurity • Type • Size
Binder	• Type • Level
Additives	• Type • Level
Forming	• Method • Pressure • Temperature
Thermal Treatment	• Peak Temperature • Heating Rates • Soak

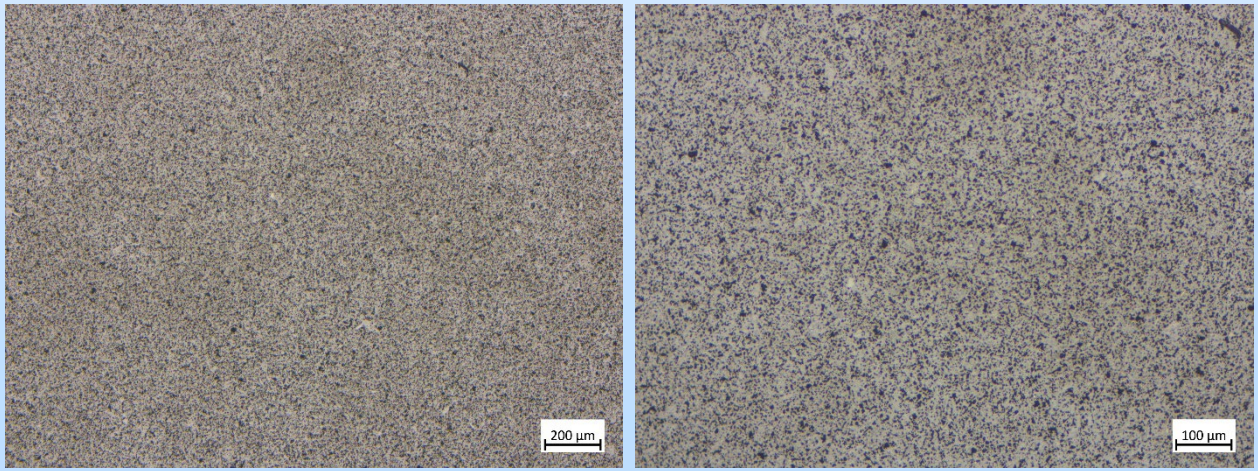


Touchstone Prior Work

Coal-based Isomold Graphite (74 µm grain Max)



Pet coke-based Isomold Graphite (5 µm grain)



Advantages/Challenges

- Advantages:
  - HHS cleaned coal with ash ~4 wt% reported ash in final graphite product at 0.1wt% (1000 ppm), well below the ≤ 1wt% project objective.
  - Final heat treatment takes some burden off the HHS process for requiring ash impurity<1wt%.
  - Feasibility demonstrated in manufacturing molded graphite from cleaned refuse coal feed.
  - Microstructures may be engineered via grain sizing and molding methodology common to SOA synthetic graphite processes.
- Challenges
  - The graphite density was reported to be 1.52 g/cm^3 that is below the stretch goal of ≥ 1.70 g/cm^3 target.
  - Pitch impregnating step required to further enhance density for achieving higher density grades.

Project Scope

- Coal cleaning via Hydrophobic-Hydrophilic Separation (HHS)
  - Low, medium, and high rank waste-coal feeds
  - Dewatering method
  - Determine purity
- Define carbon mix recipe (Milling, Mixing, & Molding)
  - Particle size and distribution
  - Binder level
  - Molding pressures
- Pyrolysis & Graphitization heat treatment
- Increase from laboratory scale to pilot scale processing
- Graphite verification via materials characterization
  - Degree of graphitization
  - Ash impurity
  - Microstructure
  - Physical properties
  - Mechanical properties

Project Tasks

1.0 Project Management and Planning (Year 1-2)

2.0 Coal Screening (Year 1-2)

- **Procure HHS cleaned coal**
- **Materials characterization**

3.0 Laboratory Scale Process Optimization (Year 1)

- **Prepare & evaluate various filler/binder mix formations**
- **Preliminary molding trial**
- **Establish Carbon & Graphite heat treatment parameters**

4.0 Graphite Pilot Processing Trials (Year 1-2)

- **Down select to best set of formulation**
- **Scale batch size to kg levels**
- **Produce prototypes and test**

5.0 Commercialization (Year 2-3)

- **Identify applications, markets, and establish potential customer base**
- **Update business case**
- **Develop commercialization plan for product launch**

Success Criteria

- Identify waste-coal fines that yield graphite.
- Clean and recover low ash and low moisture carbon from waste-coal precursor.
- Define optimal coal rank, particle size, binder levels, and pressures for molding processes.
- Scale and validate the graphite process from laboratory to pilot level capacity.
- Manage project cost, scope, schedules, business goals, and stakeholder engagement.

Milestones

Milestone	Description	Planned Completion	Actual Completion	Verification Method
A	Degree of Graphitization	9/2/2022	8/30/2022	Heat Treatment XRD
B	Mix formulations characterized	12/30/2022	11/30/2022	DSC/TGA Carbon yield
C	Mix recipe defined	5/31/2023	12/30/2022	Mill, mix, mold
D	Successfully molded shapes	8/31/2023	5/19/2023	Forming trials Microstructure Density
E	Product qualified	1/30/2025		Product testing

Accomplishments

- HHS bituminous impoundment coal fines:
  - Ash reduction
    - Superfine ash ~2%, 97-99% carbon recovery
    - Ultrafine ash <1%, 99% carbon recovery
    - Kilogram levels provided for product evaluation
- HHS anthracite silt pond feed:
  - Ash reduced to 2.8%, 87.2% carbon recovery
  - Investigations ongoing
- Graphite processing:
  - Discovery for waste-coal fines feedstock
    - Yield graphitizable carbons
    - Achieve low-enough ash and low-moisture content for process
  - Defined optimal coal rank, particle size, binder levels, and pressure for molding
  - Successfully scaled graphite process to pilot level

Synergy

Innovation Cycle

Fundamental Research	Applied Research	Product Development	Commercialization
Virginia Tech	Touchstone Virginia Tech	Touchstone Virginia Tech	Touchstone Coal Company

Waste Coal Recovery

Graphite Product

- Research institute driven
- Enhance Knowledge
- Reduce/eliminate garbage of bituminous
- Improves community environment
- Innovative HHS process research

- Conducted to apply HHS process and solve societal environmental problem
- Motivated to turn HHS process into method of reclaiming carbon from mine waste
- Benefits society via utilizing reclaimed carbon for products.
- Research institute and Industry driven

- Coal companies evaluate the commercial viability in HHS for producing high volume clean carbon
- Touchstone's objective is to develop mill, mix, forming, and heat treatment processes for producing high density molded graphite.
- Industry driven

- Touchstone motivated in bringing new product synthesized from coal to market
- Coal companies motivated in implementing "efficient" waste-coal cleaning technology and identifying markets

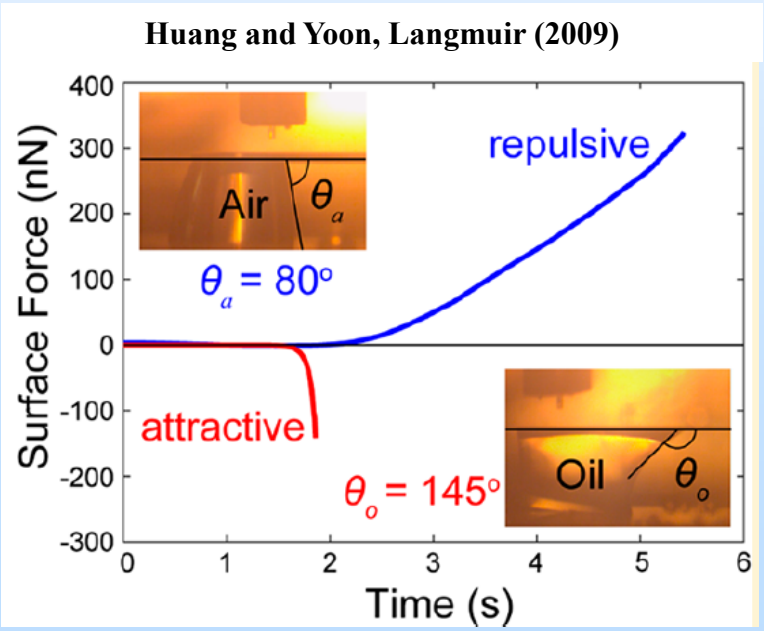
**Touchstone participates in the Mid-APPalachian Carbon Ore, Rare Earth and Critical Minerals (MAPP-CORE) Initiative focused on the expansion and transformation of the use of coal and coal-based resources – including waste streams – to produce products of high value to the 21st Century energy and manufacturing ecosystem.*

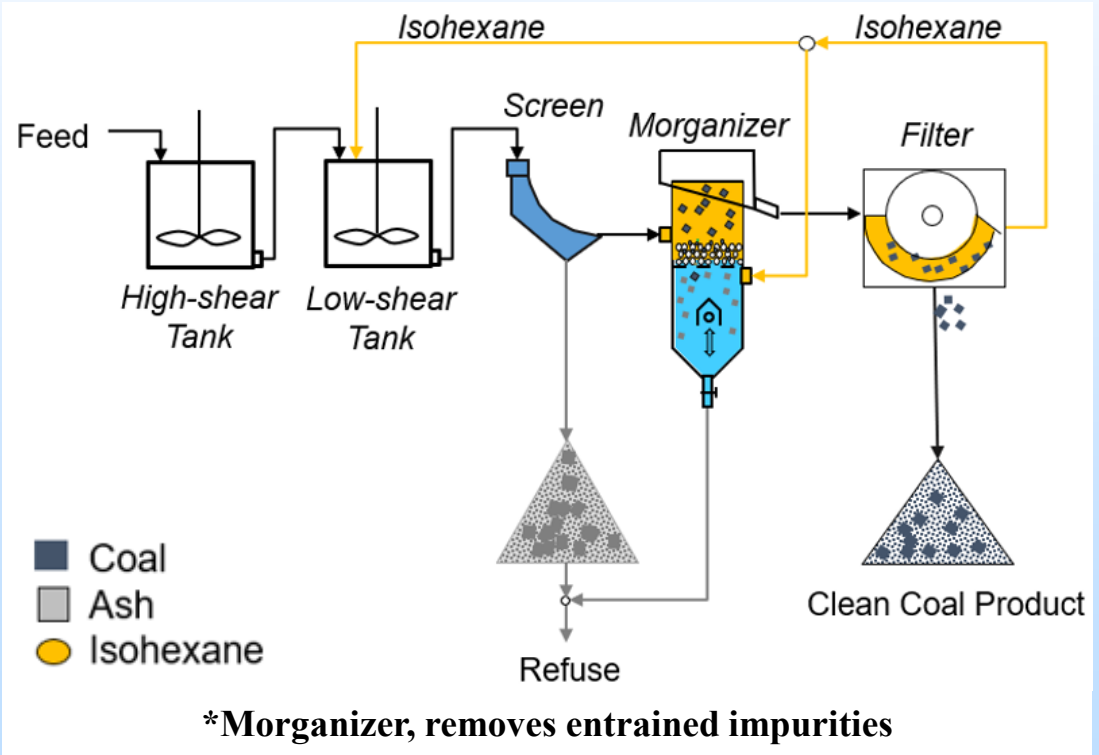
Hydrophobic-Hydrophilic Separation (HHS)

- Yoon, US Patent 9,518,241 (2016)
- Recyclable oil used in lieu of air bubbles
- Advantages of HHS
  - Oil drops increase contact angle ~2x over air bubbles
  - Surface forces
    - Repulsive for flotation
    - Attractive for HHS

High-Vol Coal

HHS Feed		HHS Product		Organic Recovery (%)	
D_{80} (μm)	Ash (%)	Ash (%)	Moisture (%)	HHS	Overall
3.6	5.89	0.80	1.98	95.23	88.00





*Morganizer, removes entrained impurities

Virginia Tech HHS

Prior Work

- Bituminous coals from the Central Appalachian coal field
  - Refuse coal accumulated more than 30 years.
  - Estimated ~4 billion tons of fine coal refuse has been discarded in the U.S.
  - Typically, one-third of a fine refuse is a recoverable carbon.
  - HHS test results obtained on the bituminous impoundment coal fines are presented in lower table.

HHS Test Results Obtained on a Bituminous Impoundment Coal Fines

Sample	Feed		Product		Reject Ash (%)	Organic Carbon Recovery (%)
	D80 (μm)	Ash (%)	Ash (%)	Moisture (%)		
As-Received	11.2	4.97	2.30	2.12	79.79	99.50
	11.2	4.97	1.93	2.37	52.71	97.28
	11.2	4.97	2.04	2.19	83.83	99.64
Ground	4.9	5.69	0.91	1.50	79.26	99.06
	4.9	5.69	0.72	1.40	81.31	99.20
	4.9	5.69	0.66	1.50	81.13	99.18

- Two anthracite samples from an operating mine in Pennsylvania.
  - Reduced ash content to 1.49% at a 0.97% moisture from a clean coal (anthracite).
  - Reduced the ash content to 2.8% at a 1.23% moisture.
  - Data suggests that had the tests been conducted after finer grinding, the ash contents would have been reduced to < 1% for both samples.

HHS Test Results Obtained on Anthracite samples

Sample	HHS Yield (%)	HHS Product		HHS Organic Carbon Recovery (%)
		Ash (%)	Moisture (%)	
Silt Pond	75.69	2.80	1.23	87.20
Clean Coal	95.12	1.49	0.97	97.96

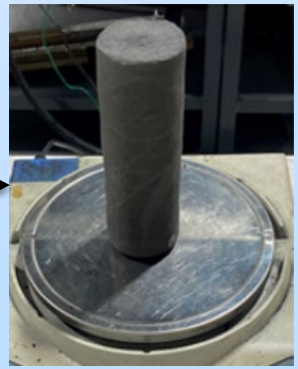
Milling, Mixing, Molding (MMM)

Ball Mill

Mixer

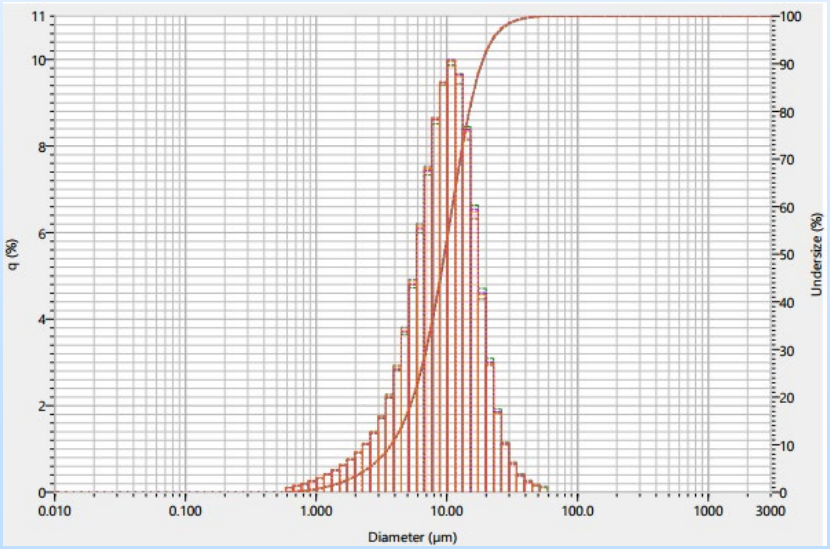
Filler + Binder

Isostatic Press



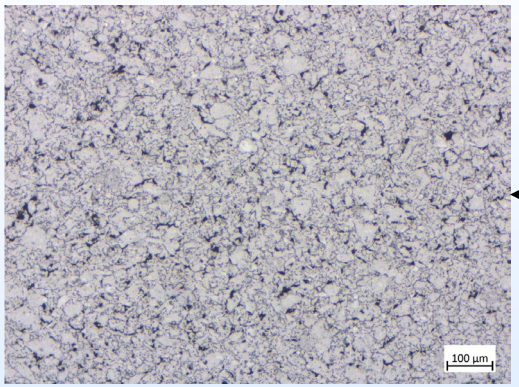
Part

Class	Isomold Graphite (Average Particle Size)
Medium	21 µm – 100 µm
Fine	11 µm – 20 µm
Superfine	***6 µm - 10 µm***
Ultrafine	1 µm - 5 µm
Angstrofine	< 1 µm

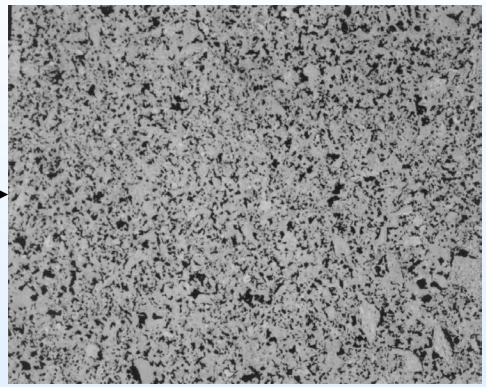


Isomold Graphite Microstructure 100X Magnification

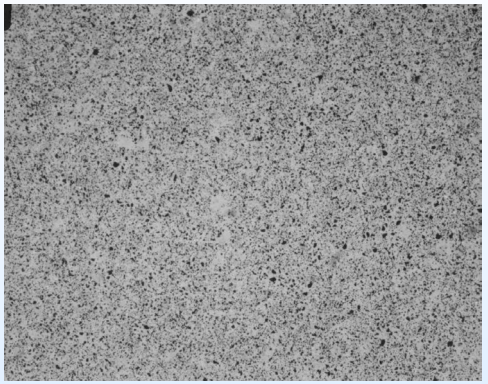
Touchstone | **Industrial**



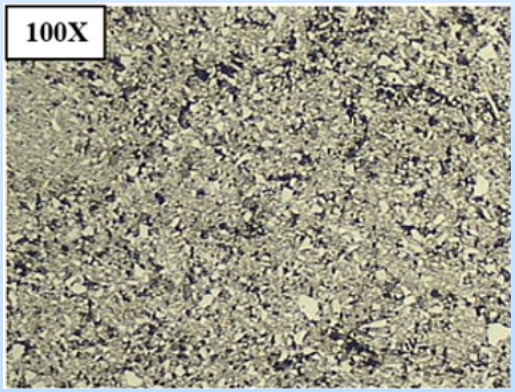
Superfine
Refuse-Coal (HHS)



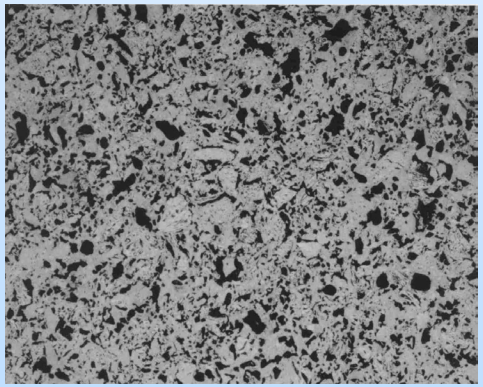
Superfine [1]



Ultrafine [1]



Medium
Coal (no binder)



Fine [1]

Particle size range [1]

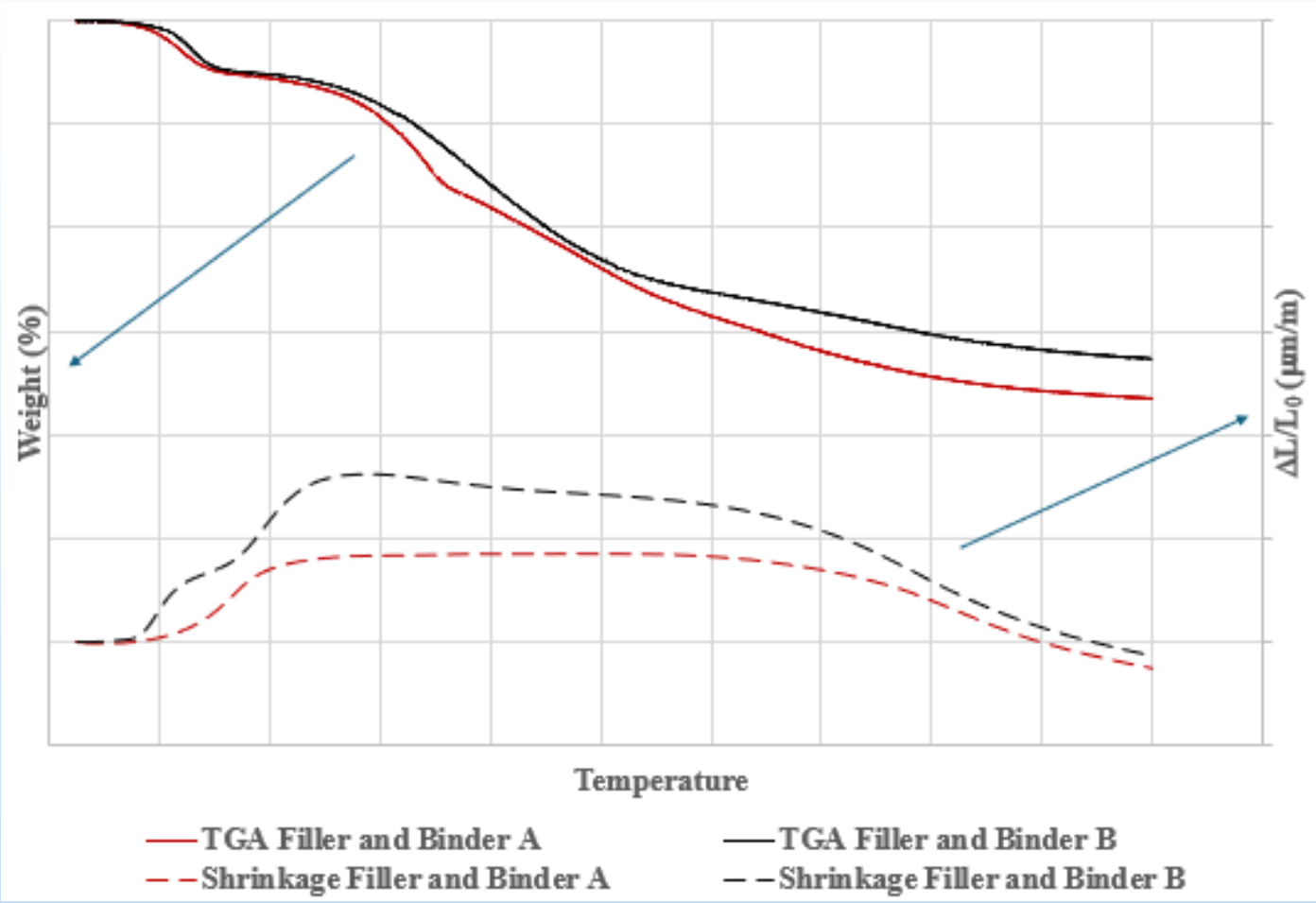
Class	(µm)
Ultrafine	1-5
Superfine	*6-10*
Fine	11-20
Medium	21-100

Reference [1]: Morgan, D., *Properties and Characteristics of POCO EDM Graphite,* International Symposium of Electro Machining (ISEM XIII) · May 1, 2001

Carbonization

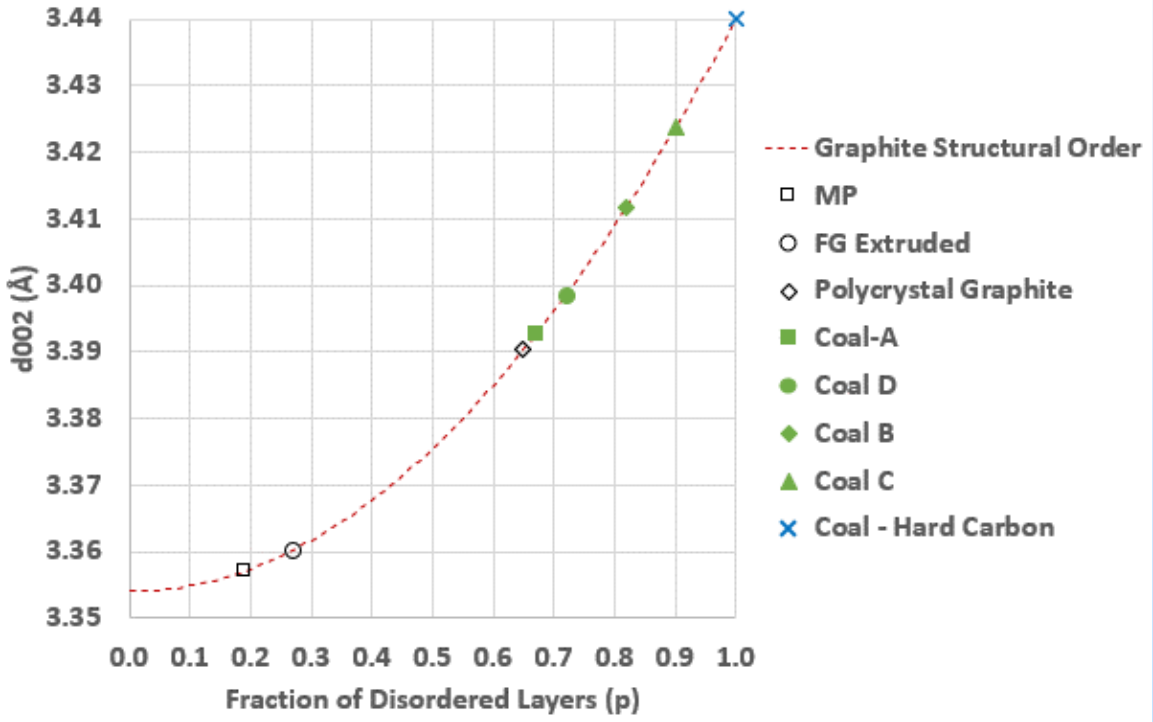
Pyrolysis

Densification

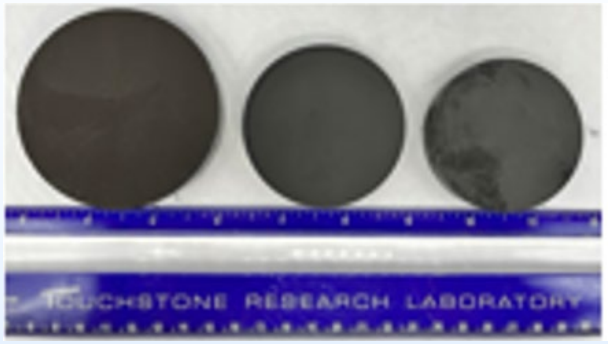


Graphite

Mean Grain Size	Density	Ash	d_{002}
μm	g/cm^3	ppm	Å
10	1.52	1,010	3.398



Green Carbon Graphite



Graphitizing Furnace

Properties

Property	Units	*Touchstone Isomold*		Molded	Isomold	
		Target	*Actual**	Fine Grain	Medium Grain	Superfine Grain
Density	g/cm^3	1.70	***1.52***	1.78	1.63	1.75
Grain Size	µm	10	***10***	20 (43 Max)	25	10
Porosity	%	25	***33***	21	28	22
Ash	**ppm**	**< 3000**	***1,010***	**< 3000**		
Compressive Strength	MPa	100	***TBD***	50	55	96
Flexural Strength	MPa	50	***TBD***	30	25	56
CTE	ppm/°C	3.5	***TBD***	4.5	2.5	7.9
Thermal Conductivity	W/m·K	85	***TBD***	NR	100	95

**** Superfine graphite produced from HHS cleaned refuse coal.***
***** Will require densification via pitch impregnation.***

Path Forward

- Virginia Tech
  - Upgrading pilot facility
    - Improving unit operation to reduce the footprint
    - Explore methods of reducing capital and operational costs
  - Continue analysis/optimization efforts with cleaning coals via HHS
- Touchstone
  - Mechanical tests and product verification
  - Update business case
    - Market analysis and planning
    - Develop commercialization plan
    - Technical-Economic Analysis

Outreach

- Local community/non-profit involvements list:
  - EdVenture Group
  - Leadership Wheeling
  - Marshall Advanced Manufacturing Center
  - Pittsburgh Technology Council
  - Schrader Center/Oglebay Institute
  - St. Clairsville Chamber of Commerce
  - WV Department of Commerce
  - WV Entrepreneurship Ecosystem
  - WV Manufacturing Extension Partnership
- Regular meetings with local economic development groups
  - Regional Economic Development (RED)
  - Ohio County Development Authority (OCDA)
  - West Virginia Economic Development
  - West Virginia Small Business Development Center (WVSBDC)
- Keynote addresses by President/CEO Brian Joseph
  - Federal Laboratory Consortium Annual Meeting
  - Federal Laboratory Consortium Mid-Atlantic Annual Meeting
  - Technology and Investor Forum, Arlington, VA.
  - Emerging Minority Business Leader's Forum, West Liberty University.
  - Milliken Innovation Forum, Spartanburg, SC.
  - St. Clairsville Area Chamber of Commerce

Workforce Development

Touchstone seeks to make science and engineering a more desirable career path for students in Appalachia, where it is currently under-represented.

- Job Fairs
  - Attends job fairs across the region
  - Utilizes human resources services
  - Job listings on social media
    - Indeed
    - LinkedIn
    - Facebook
- Summer Internship Program
  - 5-10 interns are accepted each year
  - Provided with shadowing opportunities on SBIR/STTR projects with high level engineers and technicians
  - Has partnered with local universities and groups, i.e., Community Foundation for the Ohio Valley (CFOV), to seek interns

Summary

- Ash impurity for superfine grain graphite exceeds expectations indicating 1,010 ppm (0.10%)
- Higher ash content may be liberated from refuse coal resulting in higher carbon recovery via HHS:
  - Ultrafine (5μm)
    - Ash impurity ≤1.0 %
    - Carbon recovery 99%
  - Superfine (10μm)
    - Ash impurity ≤ 2%
    - Carbon recovery 97-99%
- Laboratory and pilot processes defined, i.e., MMM, bake, and graphitization.
  - Microstructure, physical and thermophysical properties test methods defined, characterization complete.
  - Mechanical properties to be determined.
  - Pitch impregnation will be required to achieve densities >1.60 g/cm^3.

Questions & Comments

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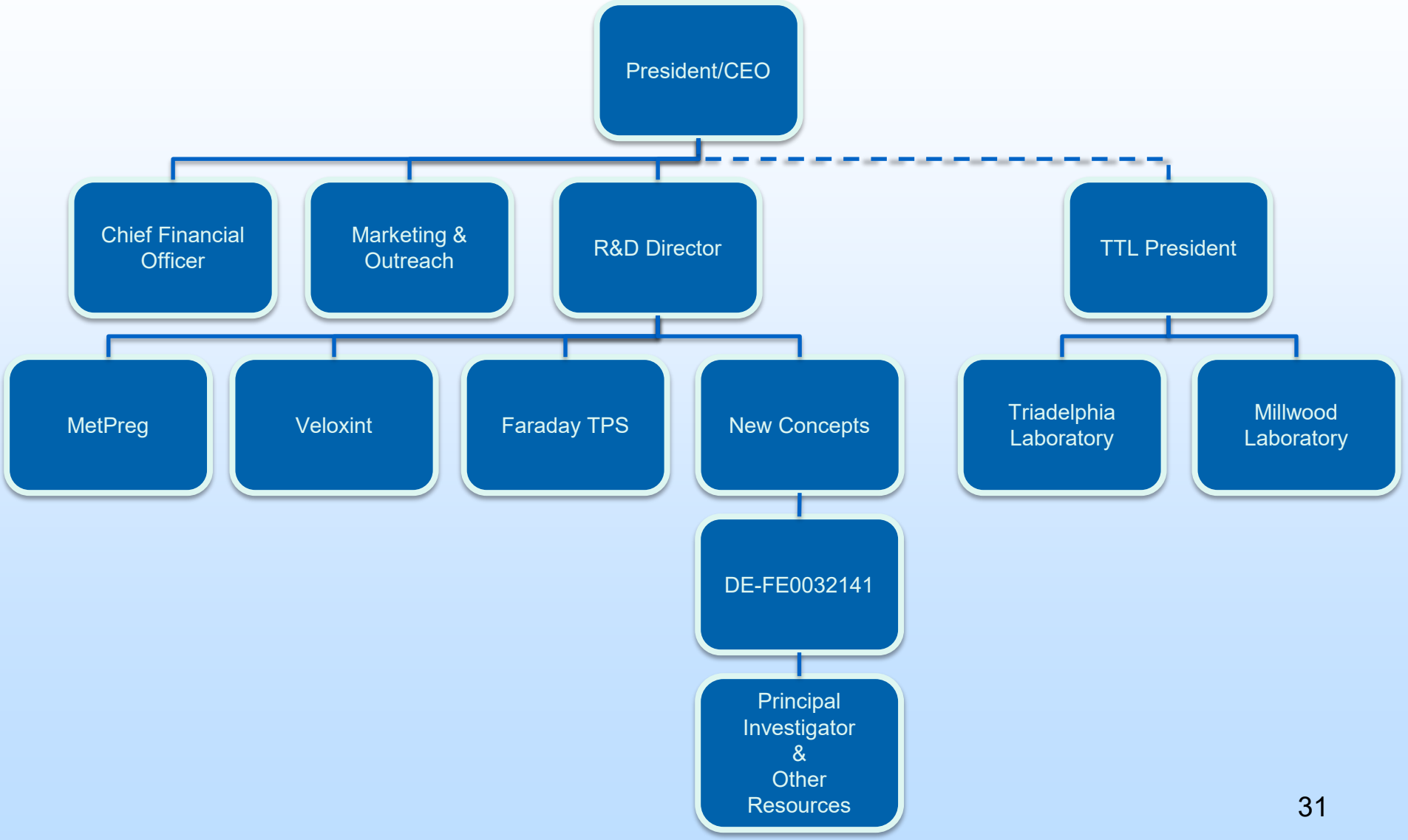
Appendix

Touchstone Research Laboratory Organizational Chart



Gantt Chart

Task Name
Start Project
1.0 Project Management
2.0 Coal Screening - Degree of Graphitization
2.1 Coal Procurement
2.2 Thermal Treatment
2.3 Degree of Graphitization (XRD)
2.4 Milestone A - Low, Med, High Rank Coal Feedstock Selections
3.0 Laboratory Scale Process Optimzation
3.1 HHS Batch and Continuous Tests
3.2 Particle Size Classification (Sizing)
3.3 Mix & Pressure Molding
3.4 Milestone B - Bench Scale Mix Formulations Characterized
3.5 Thermal Treatment
3.6 Materials Characterization
3.7 Milestone C - Mix Recipe Defined
4.0 Graphite Pilot Processing Trials
4.1 Coal Cleaning & Drying HHS
4.2 Particle Sizing
4.3 Mix and Pressure Molding
4.4 Heat Treatment, Bake and Graphitization
4.5 Milestone D - Green Billet Molded Shape and Density Verified
4.6 Year 1 Project Extension
4.7 Graphite Production & Qualification
4.8 Batch Qualification (Test & Verification)
4.9 Voice of the Customer
4.10 Commercialization Plan for Product Launch
4.11 Milestone E - Process Defined-Product Qualified
5.0 Commercialization
5.1 Market Analysis & Planning
5.3 Technical-Economic Analysis
End of Project