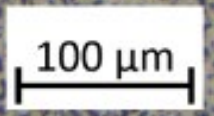
Molded Graphite Products Synthesized from Waste-Coal

DE-FE0032141

Dwayne Morgan

Touchstone Research Laboratory, Ltd.

U.S. Department of Energy
National Energy Technology Laboratory
Resource Sustainability Project Review Meeting
October 25 - 27, 2022



Project Overview

TOUCHSTONE® RESEARCH LABORATORY, LTD.

- Team
  - DOE Project Manager: Mark Render
  - Principal Investigator: Dwayne Morgan
  - Recipient: Touchstone Research Laboratory
  - Subrecipient: Virginia Tech
  - Industry Partner: Arq
- Period of Performance
  - Start June 1, 2022
  - End May 31, 2024
- Project Budget
  - Total: $1,250,000
  - DOE Share: $1,000,000
  - Cost Share: $250,000

Who We Are

TOUCHSTONE® RESEARCH LABORATORY, LTD.

- 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 Objectives

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- Develop low-ash high-density graphite from cleaned domestic waste-coal feedstock.
  - Impurity ≤ 1wt%
  - Moisture ≤ 1wt%
  - Coal feed particle size < 40μm
- Reduce graphite production costs and dependance on petroleum coke.
  - Identify waste-coal fines that yield graphitizable carbon
  - Determine feasibility in achieving low-ash and low-moisture content in waste-coal precursor
  - Define optimal coal rank, particle size, binder levels and pressure for molding processes
- Transition the technology from laboratory scale proof of concept (TRL-3) to pilot system validation in a relevant environment (TRL-5).

Technology Background

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Current SOA Coal Cleaning

- Coal preparation requires carefully engineered processing plants which employ low-cost solid-solid and solid-liquid separation processes
- Coal preparation is readily defined by five distinct unit operations:
  1) Coal size reduction (crushing/milling)
  2) Size separation - Jigging and dense medium separation (DMS)
  3) Solid separation
  4) Solid-Liquid separation
  5) Waste disposal

Technology Background

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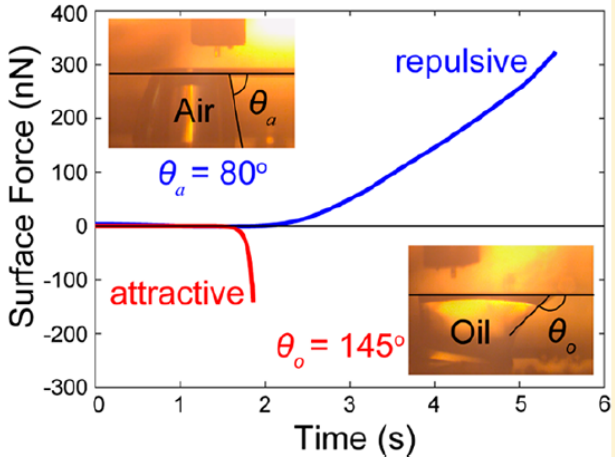
Hydrophobic-Hydrophilic Separation (HHS)

- 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

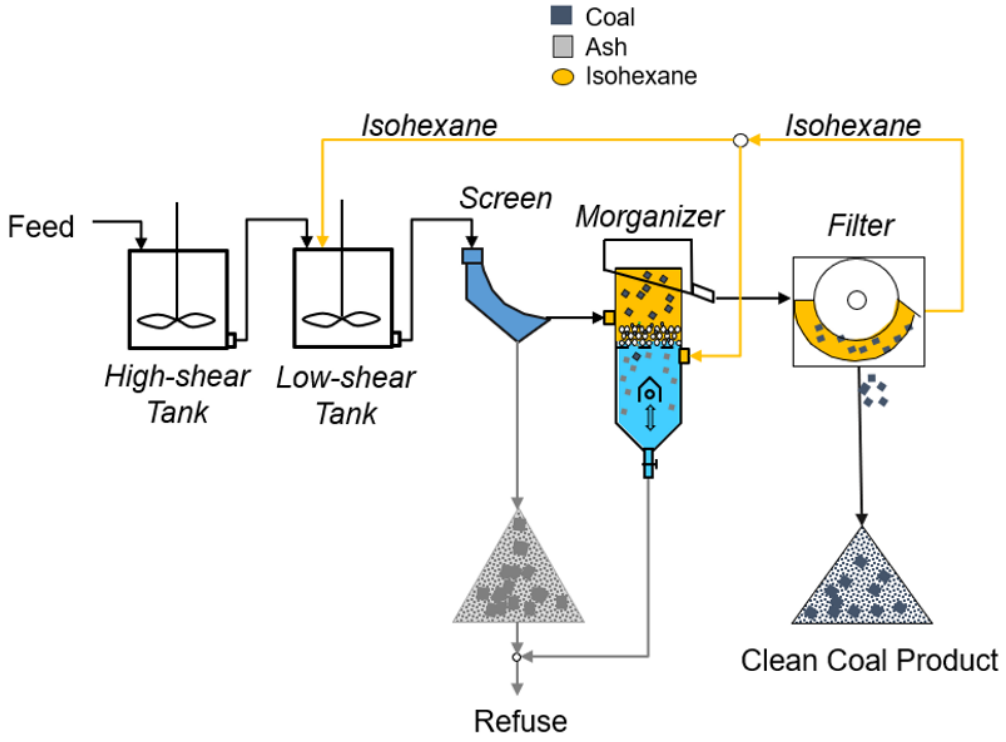


Huang and Yoon, Langmuir (2009)

  - Morganizer
    - Removed entrained impurities

Arq 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



Virginia Tech HHS

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Prior Work Obtained on Bituminous and Anthracite

- 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.
- More recently, the HHS process has been tested on 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 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

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

Touchstone's Prior Work

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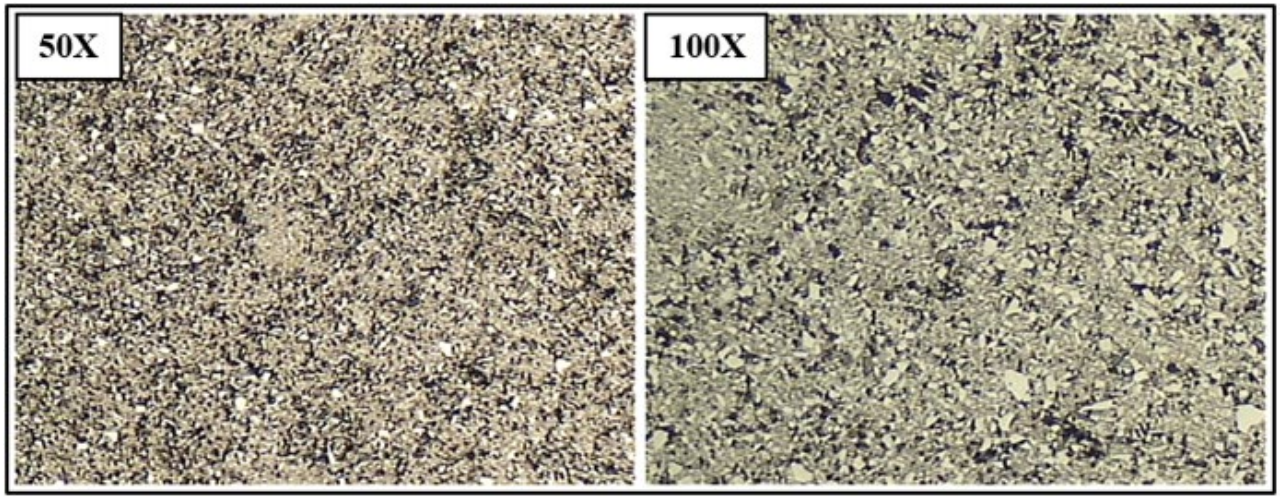
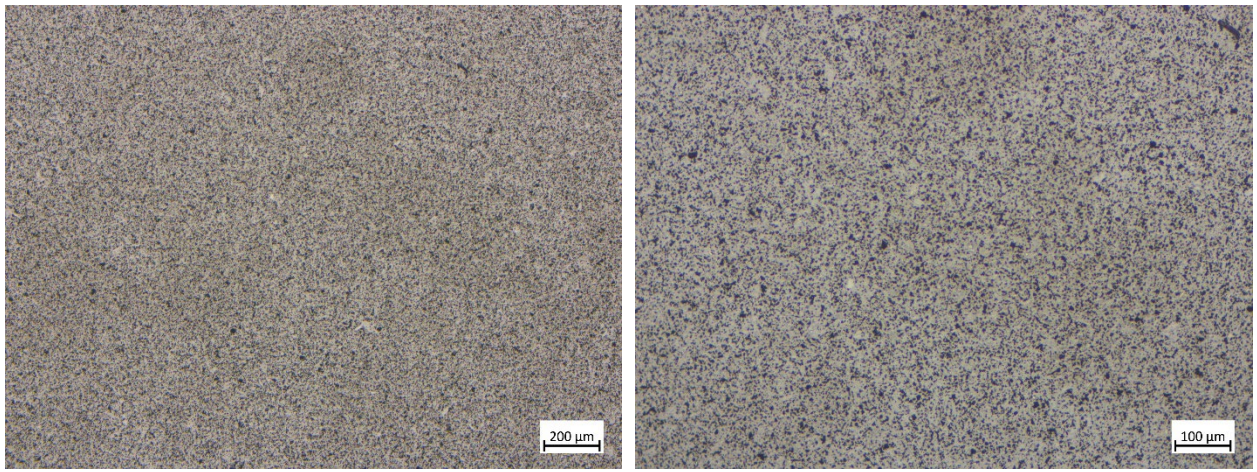
Coal-based Isomold Graphite (74 µm grain Max), No Binder



Pet coke-based Isomold Graphite (5 µm grain), With Binder



Touchstone's *Target

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Property	Unit	Touchstone		Molded	Isomolded		
		Carbon	Graphite*	*CeraMaterials M1*	*Amsted ATR*	*POCO AXM5Q*	*POCO ZXF5Q*
Density	g/cm^3	1.52	1.70	1.78	1.63	1.73	1.78
Grain Size	µm	35	5	20 (43 Max)	25	5	1
Porosity	%	33	25	21	28	23	21
Compressive Strength	MPa	138	110	50	55	124	175
Flexural Strength	MPa	NR	60	30	25	69	112
CTE	ppm/°C	3.5	3.5	4.5	2.5	7.9	8.1
Thermal Conductivity	W/m·K	5	85	NR	100	95	70
**** Target***							

Project Scope

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- Laboratory Trials
  - Virginia Tech - HHS
    - Carbon recovery from waste streams HHS
    - Bituminous coal and anthracite
  - Touchstone – cleaned product screening
    - Characterize and thermally treat to yield graphite powders.
    - Down-select products based on the degree of graphitizability
    - Initiate preliminary mix trials with ultrafine coal filler and binders
- Pilot Process Development (Prototyping)
  - Virginia Tech provide ultrafine clean-coal for billet prototyping
  - Touchstone produce prototype billets using defined formulations and processes from the laboratory trial work.
- Graphite Test and Validation
  - Touchstone materials characterization lab
  - Voice of the customer (VOC)
- Commercialization Plan (HHS & Graphite Process)

Work Plan

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Task and Subtask For This Effort

1.0 - Project Management and Planning

2.0 – Coal Screening – Deg. of Graphitization

- 2.1 – Coal Procurement
- 2.2 Thermal Treatment
- 2.3 Graphite Powder Characterization

♦ ***A – Coal Screening via Graphitization Trials - <u>Complete</u>***

3.0 – Laboratory Scale Process Optimization

- *3.1 HHS Batch and Continuous Tests*
- *3.2 Particle Size Classification (Sizing)*
- *3.3 Mix and Pressure Molding*
- *3.4 Thermal Treatment*
- *3.5 Materials Characterization*

♦ ***B – Mix Recipe Defined (5/31/2023)***

4.0 Graphite Pilot Processing Trials

- *4.1 Coal Cleaning & Drying via HHS*
- *4.2 Particle Sizing*
- *4.3 Mix and Pressure Molding*
- *4.4 Thermal Treatment*
- *4.5 Batch Qualification (Test & Verification)*

♦ **C – *Graphite Process Defined, Batch* Processes Qualified (01/02/2024)**

5.0 Commercialization

- *5.1 Market Analysis & Planning*
- *5.2 Customer Product Evaluation*
- *5.3 Technical-Economic Analysis*
- *5.4 Voice of the Customer (VOC)*
- *5.5 Commercialization Plan for Product Launch*

Project Success Criteria

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- 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.

Perceived Risks and Mitigation Strategies

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Perceived Risk	Risk Rating			Mitigation/Response Strategy
	Probability	Impact	Overall	
	(Low, Med, High)			
Financial Risks:				
Equipment Cost	Low	Low	Low	Touchstone has conducted mix trials of various coals and has determine it will need a high shear mixer for blending carbon filler (coal) with binder. Accordingly, Touchstone is in process of acquiring a sigma mixer.
Technical/Scope Risks:				
Coal impurity	High	Med	Low	Touchstone's mitigation strategy is to subcontract Virginia Tech and use their HHS coal cleaning technology to low ash impurity to acceptable levels (~1%).
Molding coal feed	Low	Med	Low	Touchstone has demonstrated the ability to mold coal feed in this effort although mix and molding trials are ongoing. An objective is to yield high carbon and graphite density without pre-calcining the coal feed. If densities are low, then the mitigation will be to pitch impregnate the molded carbon.
ES&H Risks:				
Volatiles in Graphitization Process	High	High	High	Reduce mineral deposit in coal feed via HHS process, thereby lowering subsequent residual ash in carbon products to be graphitized.
External Factor Risks: None currently				
Cost/Schedule Risks: None currently				
Management, Planning, and Oversight Risks: None currently				

Status - Coal Screening

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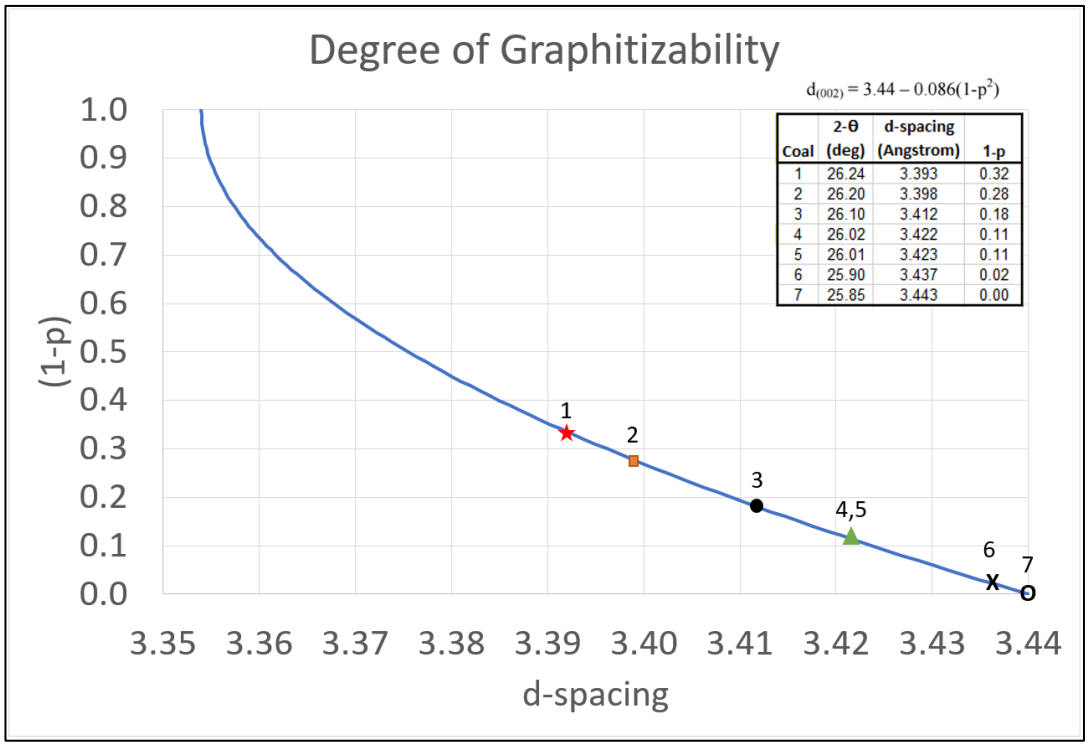
Cleaned Waste (Disposal) Coal → Graphite

Theoretical Density:
2.26 g/cc at 1-atm and 300K



Coal	2-θ (deg)	d-spacing (Angstrom)	1-p
1	26.24	3.393	0.32
2	26.20	3.398	0.28
3	26.10	3.412	0.18
4	26.02	3.422	0.11
5	26.01	3.423	0.11
6	25.90	3.437	0.02
7	25.85	3.443	0.00

Highly Graphitic

Turbostratic

Pilot Process Graphitzer
3000 °C Max

X-Ray Diffractometer

Status - Materials Characterization

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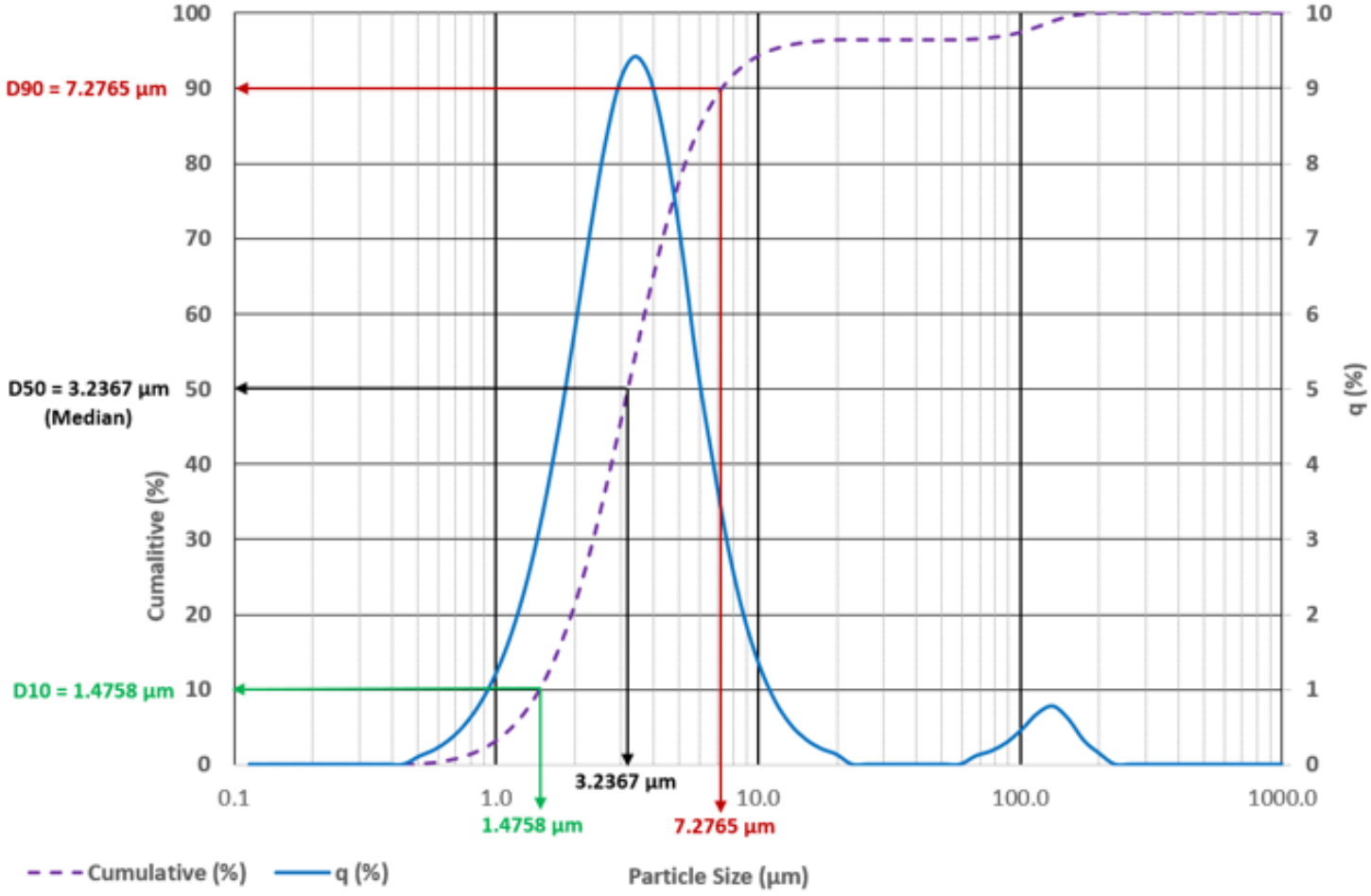
Particle Size Analysis



Status - Materials Characterization

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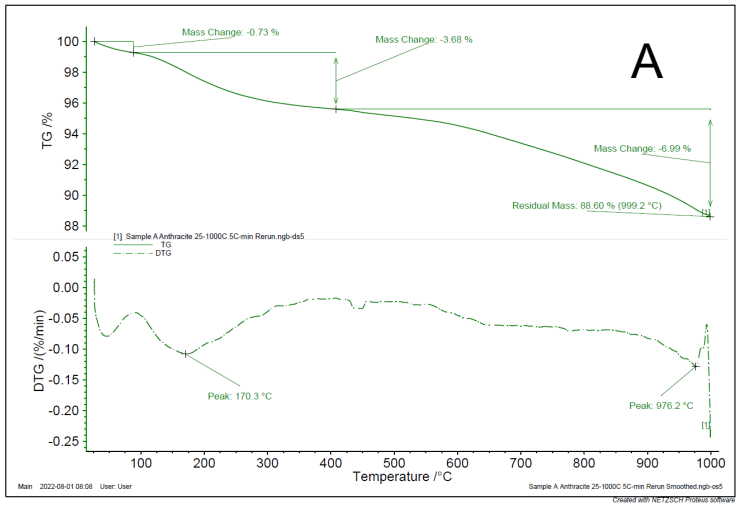
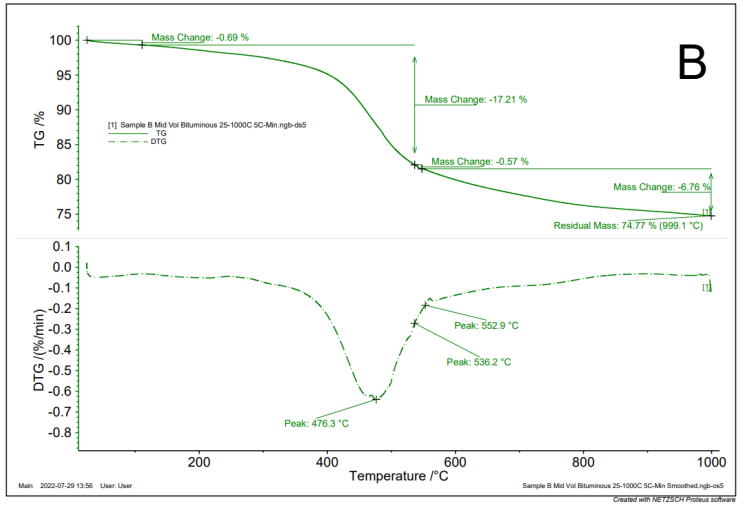
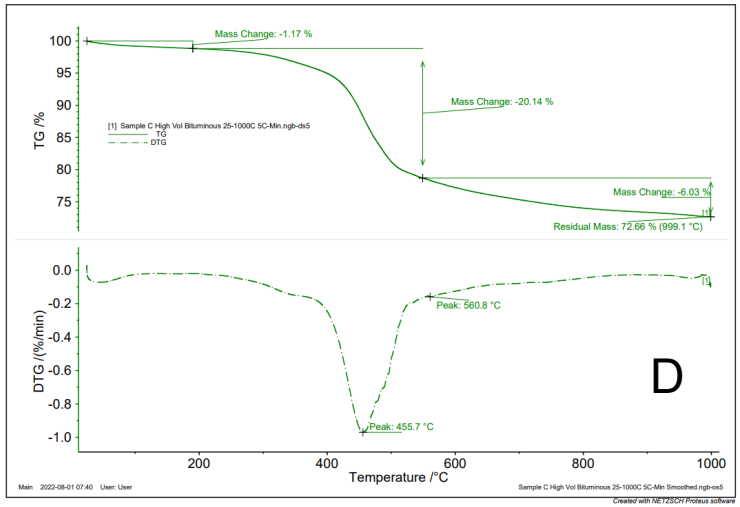
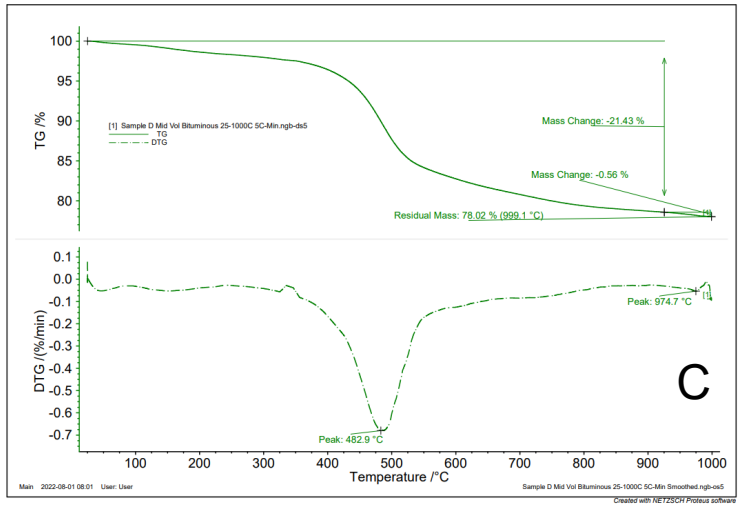
Thermogravimetric Analysis (TGA)









ID	CY (%)	DTG (°C)
A	88.6	170
B	74.8	473
C	78.0	483
D	72.7	456

Status - Materials Characterization

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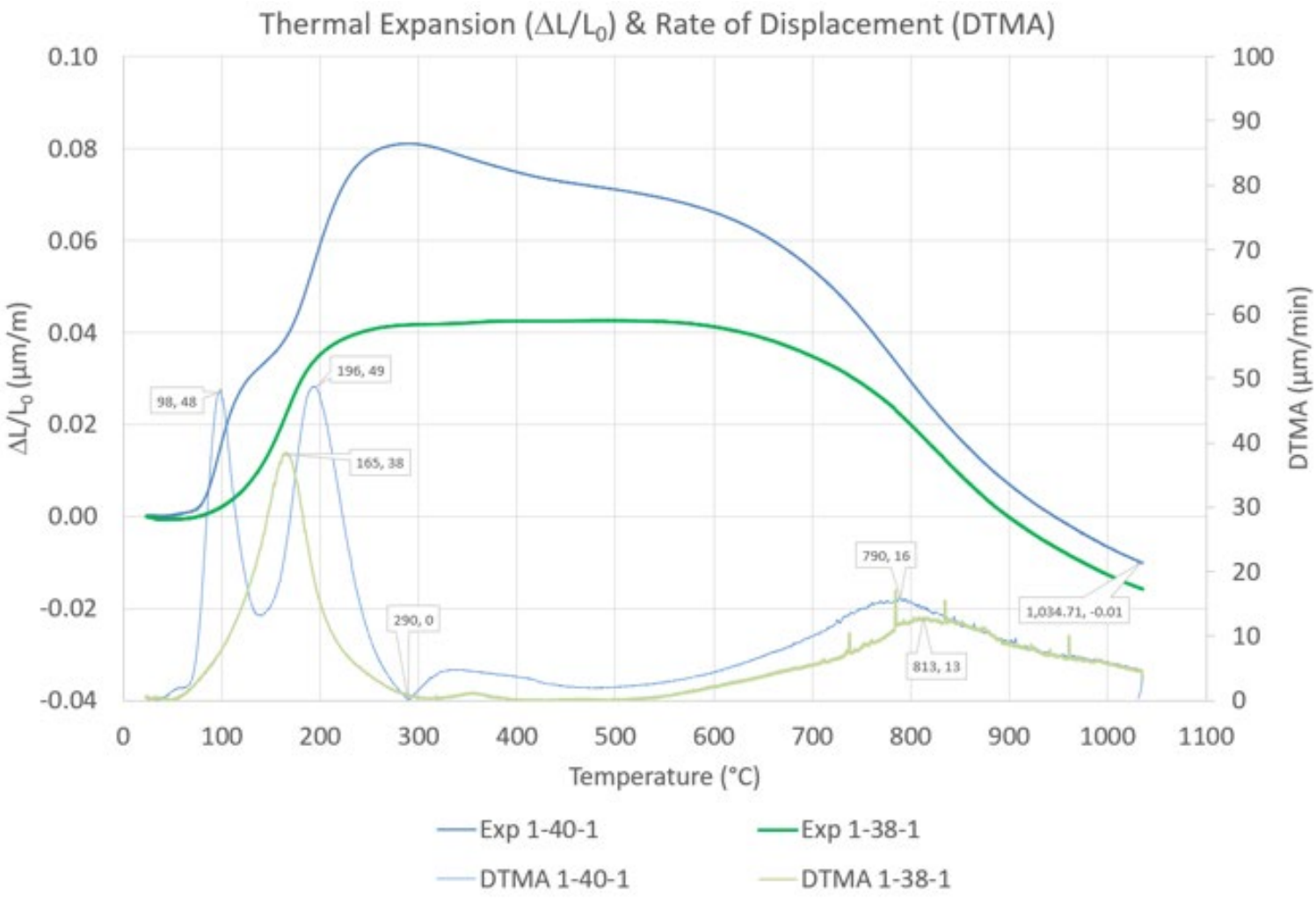
Thermo-Mechanical Analysis (TMA)



Status - Process Development

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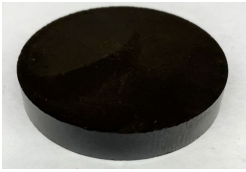
Laboratory Mix Trials - Work In Process

Process Factors	Description
A	Filler (coal type, sizing, ash)
B	Binder (type, amount)
C	Additives (type, amount)
D	Molding (method, pressure, temperature)
E	Carbonization (heating rate, temperature, soak)
F	Graphitization (heating rate, temperature, soak)

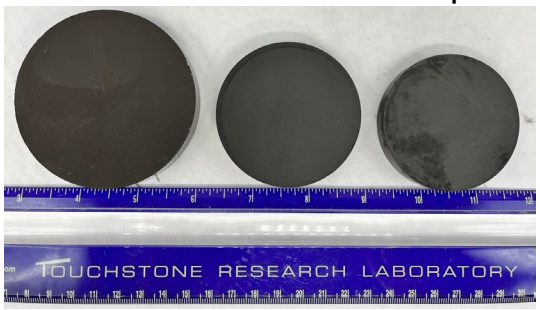
Molded – 3”ϕ

H=0.91”

H=0.52”



Molded Carbon Graphite



Mixing

Uniaxial Press

Isomold Press

Pyrolysis (Carbonization)

Pitch Impregnation

Graphitization

Accomplishments

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- HHS bituminous impoundment coal fines:
  - As-received superfine ash reduced from 4.97% to ~2%, 97-99% carbon recovery
  - Ash in ultrafine particle size reduced from 5.69% to <1%, 99% carbon recovery
  - Sampling increased to kilogram levels for product evaluation
  - Significant reduction in anthracite ash content levels, investigations ongoing
- Graphite processing Milestone “A” achieved for identifying suitable HHS cleaned carbon feedstock for molding graphite
- Initiated preliminary molding trials using cleaned ultrafine coal/binder
- Developed characterization methods for coal selection and process optimization:
  - XRD - Rank coal graphitizability by fraction of disordered graphite
  - TGA - Carbon yields of coal feed and binder materials
  - TMA - Critical process temperatures and rates of volumetric expansion and shrinkage during pyrolysis (carbonization)

Synergy

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Innovation Cycle

Fundamental Research	Applied Research	Product Development	Commercialization
Virginia Tech	Touchstone Arq & Virginia Tech	Touchstone Arq & Virginia Tech	Touchstone Arq

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

- Arq will 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
- Arq motivated in implementing an alternative "more efficient" waste-coal cleaning technology

**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.*

Path Forward

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Virginia Tech HHS

- Various coal samples are being tested to identify the best feedstocks for graphitization studies:
  - Different ranks of coal
  - Mineral matter liberation from the carbon matrix
  - Optimal operating conditions for the HHS process
  - Lower ash products with <<1% ash
- Improving unit operation to reduce the footprint
  - Recycling spent oil *via* steam stripping
- Developing a small modular unit that can be tested on a mine site.
  - To produce enough materials for pilot-scale graphitization studies
- Explore methods of reducing cost
  - Capital
  - Operational

Path Forward

TOUCHSTONE® RESEARCH LABORATORY, LTD.

Touchstone Graphite Processing

- Graphite process development
  - Characterize coal feedstock (ongoing)
  - Define mix recipe and process
    - Filler and binder level
    - Mixer operating conditions (time and temperature)
  - Define molding requirements
  - Optimize bake and graphitization schedules
- Qualify product through testing and customer evaluations
- Develop product data sheets and market research
- Determine critical needs for expansion and product launch:
  - HHS forecast and raw material suppliers
  - Resources
  - Location
  - Capital procurement

Summary

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- The HHS process shows that reducing ash to <1% in impoundment coal fines is achievable.
- Ultrafine coal cleaned via HHS can be molded.
- Characterization methods have been developed to predict yielding higher quality graphite products and reduce costly scrap.

Questions & Comments

TOUCHSTONE® RESEARCH LABORATORY, LTD.

- Dwayne Morgan
- Phone: 304-547-5800, ext. 231
- Email: drm@trl.com

Backup Slides

TOUCHSTONE® RESEARCH LABORATORY, LTD.

25

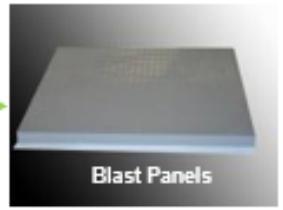
Touchstone: Innovation

TOUCHSTONE® RESEARCH LABORATORY, LTD.

Touchstone's Applications in Coal-based Carbon and Graphite

Carbon foam used in hot structures for thermal protection

Carbon foam and polyurea energy absorbing panels

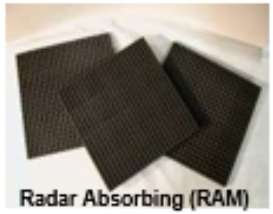


Carbon foam provides electromagnetic shielding

Composite Tooling made from carbon foam and graphite foam may be for making carbon composite aerospace components

High density carbon panels for weapons bays or plume management

Fire resistant carbon foam panels for bulkheads and decks



High density graphite for heat exchangers

Carbon foam can absorb radar

Appendix

TOUCHSTONE® RESEARCH LABORATORY, LTD.

- These slides will not be discussed during the presentation but are mandatory.

Organizations

TOUCHSTONE® RESEARCH LABORATORY, LTD.

- Project Team
  - DOE Project Manager: Mark Render
  - Principal Investigator: Dwayne Morgan
  - Recipient: Touchstone Research Laboratory (Brian Gordon, Dwayne Morgan)
  - Subrecipient: Virginia Tech (Dr. Roe-Hoan Yoon and Dr. Aaron Noble)
  - Industry Partner: Arq (Kenny Schmitt and Tim Blackburn)
- HHS Development
  - Virginia Tech
    - HHS process is being transitioned from laboratory research to pilot process.
    - Virginia Tech and Arq will evaluate effectiveness in reclaiming high volumes of carbon product via HHS.
    - The cleaned products from the pilot process will be made available to Touchstone as feedstock for molded graphite processing.
  - Arq
    - Provides Touchstone with their cleaned waste-coal ultrafine product for evaluation to be used as baseline against HHS.
    - Assisting Virginia Tech with identifying mine locations for sampling waste-coal fines.
    - Arq is key in transitioning the HHS from pilot scale to commercial scale.
      - Arq is providing Virginia Tech and Touchstone with desired HHS capacity requirements and recommendations on how to make the process available at the mine location, i.e. it is not feasible to truck waste-stream to a central HHS cleaning facility.
      - Arq will be evaluating the risk, cost, and benefits in commercializing HHS.

Organizations (cont.)

TOUCHSTONE® RESEARCH LABORATORY, LTD.

- Touchstone
  - Graphite product development
  - Characterize the cleaned coal products received from Virginia Tech and Arq.
  - The various cleaned products are used in molding trials to evaluate the processability and quality of graphite synthesized from waste-coal reclaimed via HHS.
  - Touchstone will summarize facility and equipment requirements for implementation.
  - Responsible for the risk, cost, and benefits analysis as it relates to utilization of reclaimed carbon via HHS for molded graphite products.

Gantt Chart

TOUCHSTONE® RESEARCH LABORATORY, LTD.

ID	Task Name
1	Start Project
2	**1.0 Project Management**
3	1.1 Project Management Plan
4	1.2 Technology Maturation Plan
5	1.3 Summary of Environmental Justice Document
6	1.4 Summary of Economic Revitalization and Job Creation
7	1.5 Reporting
8	**2.0 Coal Screening - Degree of Graphitization**
9	2.1 Coal Procurement
10	**2.2 Thermal Treatment**
13	2.3 Degree of Graphitization (XRD)
14	2.4 Milestone A - Low, Med, High Rank Coal Feedstock Selections
15	**3.0 Laboratory Scale Process Optimzation**
16	3.1 HHS Batch and Continuous Tests
17	3.2 Particle Size Classification (Sizing)
18	3.3 Mix & Pressure Molding
19	**3.3 Thermal Treatment**
20	3.3.1 Carbonization
21	3.3.2 Graphitization
22	3.4 Materials Characterization
23	3.5 Milestone B - Mix Recipe Defined
24	**4.0 Graphite Pilot Processing Trials**
25	4.1 Coal Cleaning & Drying HHS
26	4.2 Particle Sizing
27	4.3 Mix and Pressure Molding
28	**4.4 Thermal Treatment**
29	4.4.1 Carbonization
30	4.4.2 Graphitization
31	4.5 Batch Qualification (Test & Verification)
32	4.6 Milestone C - Graphite Process Defined/Batch Processes Qualified
33	**5.0 Commercialization**
34	5.1 Market Analysis & Planning
35	5.2 Customer Product Evaluation
36	5.3 Technical-Economic Analysis
37	5.4 Voice of the Customer
38	5.5 Commercialization Plan for Product Launch
39	End of Project