U.S. DEPARTMENT OF ENERGY

Introduction to Graphite

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United States Department of Energy

DOE Alaska Workshop on Critical Materials

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Graphite - Background

- Major Crystalline Forms of Carbon
- Diamond, Graphite
- Natural Deposits, Synthetic Graphite
- No Domestic Production Natural Graphite
- Critical Material – USGS, DOE

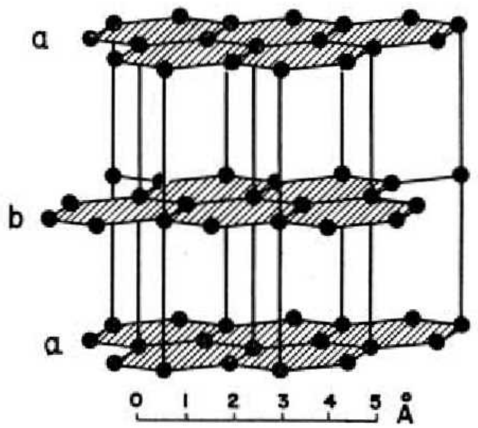

Figure 1. Layer structure of graphite.

Man-Made Graphite: Thermal Methods

- Acheson Process - Heating Solid Carbon Precursors with SiO_2 to 4,000^{o}C
- Pyrolysis of Methane, Ethane, Benzene 2,000^{o}C (Coatings)
- Heating Powdered Petroleum Coke above 3,000^{o}C
- Catalyst Aided Thermal Graphitization

Graphite: Uses

- Brake Linings
- Lubricants
- Powdered Metals
- Refractory Applications/Brick/Linings
- Steelmaking
- Anodes – Lithium Batteries
- Composites
- Electronics
- Foils
- Courtesy "MINERAL COMMODITY SUMMARIES 2019", Amy C. Tolcin editor, US Geological Survey, https://pubs.er.usgs.gov/publication/70202434

"Dynamic Dozen" Critical Materials

GOALS

● 100% clean electricity by 2035: 30 GW offshore wind by 2030 ●

● Zero-emission transportation: 50% EV adoption by 2030 ●

- Neodymium, Praseodymium and Dysprosium for magnets → Magnets enable efficient electric machines including wind generators, electric and fuel cell vehicle motors, industrial motors
- Lithium, Cobalt, Nickel, Graphite, and Manganese for energy storage → Batteries are needed for electric vehicles and grid storage to enable high penetration of zero-emission transportation and intermittent clean power generation
- Iridium & Platinum for electrolyzers; Platinum for fuel cells → Iridium and platinum for electrolyzers are needed for green hydrogen production and platinum for fuel cells used in transportation and stationary energy storage.
- Gallium for wide bandgap semiconductors, LEDs → Wide bandgap power electronics enable high voltage power generation (like wind) to connect to the grid
- Germanium for microchips (semiconductors) → Microchips for sensors, data, and control play an important role in SMART manufacturing, which will be needed to increase efficiency and minimize waste (inclusion GHGs); Fiber and infrared optics

America's Strategy to Secure the Supply Chain for a Robust Clean Energy Transition

Introducing The Electric Eighteen

Critical Materials for Energy

- Aluminum
- Cobalt
- Copper
- Dysprosium
- Electrical Steel* (grain-oriented steel, non-grain-oriented steel, and amorphous steel)
- Fluorine
- Gallium
- Iridium
- Lithium
- Magnesium
- Natural Graphite

Introducing The Electric Eighteen

Critical Materials for Energy

- Neodymium
- Nickel
- Platinum
- Praseodymium
- Silicon
- Silicon Carbide
- Terbium

- "Critical Materials Assessment", USDOE, July 31, 2023, available on-line https://www.energy.gov/sites/default/files/2023-07/doe-critical-material-assessment_07312023.pdf

Net Import Reliance for Energy Critical Materials

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Net Import Reliance for Selected Energy Critical Materials

Material	Net % Import Reliance in 2023	Uses
Aluminum	44	Aircraft, Packaging, Construction, almost all sectors of the economy and future clean energy
Cobalt	67	Batteries, Alloys
Copper	46	Electric Conductor, Vehicles, Wind
Dysprosium	> 95	Magnets
Gallium	100	Wide Bandgap Semiconductors
Iridium	100 (estimated)	Electrolyzers, Catalysts
Lithium	> 25	Batteries
Magnesium	> 50	Alloys
Natural Graphite	100	Batteries, Steelmaking
Neodymium	> 95	Magnets
Nickel	57	Batteries, Alloys, Steels, Catalysts
Platinum	83	Electrolyzers, Fuel Cells, Catalysts
Praseodymium	> 95	Magnets
Silicon	< 50	Microchips, Photovoltaics
Terbium	> 95	Wind Turbines, Electric Vehicles
Uranium	> 90	Nuclear Power

Import Sources for Energy Critical Materials

Figure 5 Countries Exporting Selected Energy Critical Materials to the United States

Material	Import Sources (2019-2022)
Aluminum	Canada, 52%; United Arab Emirates, 8%; Bahrain, 4%; Russia, 4%; other, 32%.
Cobalt	Norway, 25%; Canada, 15%; Finland, 13%; Japan, 12%; other, 35%.
Copper	Refined copper: Chile, 64%; Canada, 18%; Mexico, 11%; other, 7%.
Dysprosium	China 72%; Malaysia, 11%; Japan, 6%; Estonia, 5%; other, 6%. (estimate)
Gallium	Japan, 26%; China, 21%; Germany, 19%; Canada, 9%; other, 25%.
Iridium	
Lithium	Argentina, 51%; Chile, 43%; China, 3%; Russia, 2%; other, 1%.
Magnesium	Canada, 18%; China,5 9%; Israel, 9%; Taiwan, 9%; other, 55%. (all forms)
Natural Graphite	China 42%; Mexico, 16%; Canada, 15%; Madagascar, 12%; other, 15%.
Neodymium	China 72%; Malaysia, 11%; Japan, 6%; Estonia, 5%; other, 6%. (estimate)
Nickel	Canada, 46%; Norway, 9%; Finland, 7%; Russia, 7%; other, 31%. (primary)
Platinum	South Africa, 33%; Switzerland, 15%; Germany, 14%; Belgium, 9%; other, 29%.
Praseodymium	China 72%; Malaysia, 11%; Japan, 6%; Estonia, 5%; other, 6%. (estimate)
Silicon	Brazil, 23%; Russia, 21%; Canada, 18%; Norway, 7%; other, 31%. (all forms)
Terbium	China 72%; Malaysia, 11%; Japan, 6%; Estonia, 5%; other, 6%. (estimate)
Uranium	Canada 27%; Kazakhstan 25%; Russia 12%; Uzbekistan 11%; Australia 9%; other 16% (2022 imports, data from EIA)

Data from 2024 Mineral Commodities Summaries

Graphite

- **Critical Mineral (USGS) and a Critical Energy Material (USDOE)**

Natural Graphite

- US Imported 84,000 Metric Tons in 2023
- **100% Import Reliance**
- Import Sources: (2019-2022) China 42%; Mexico, 16%; Canada, 15%; Madagascar, 12%; other, 15%.
- The major uses of natural graphite were batteries, brake linings, lubricants, powdered metals, refractory applications, and steelmaking.
- The number of lithium-ion battery manufacturing facilities in the United States increased to 10 in 2023 from 3 in operation during 2019.
- An additional 28 facilities were under development.
- U.S. Geological Survey, 2024, Mineral commodity summaries 2024: U.S. Geological Survey, 212 p., https://doi.org/10.3133/mcs2024.

Domestic Demand for Graphite Will Grow

- "Very crude back of the envelope estimates for US EV market" – the current domestic annual automobile/SUV demand is around 15.5 million cars/year (2023). **The DOE has a goal of 50% EV adoption by 2030. Assuming this DOE goal is met,**

- EV car batteries contain approximately 150 lbs. of graphite. Assuming 3% annual increase in annual automobile demand (23% increase in automobile demand for 2030, 19 million cars/year in 2030):

- 19,000,000 cars sold in 2030/year * 0.5 EV * 150 lbs. graphite/electric vehicle * 1 ton/2,000 lb. = **713,000 tons graphite required for 2030 US EV demand**
- Versus 92,000 tons (84,000 metric tons) in 2023
- Not accounting for Electric Trucks, Consumer Batteries,……

Energy Critical Materials – Import Reliance

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- Many energy critical materials such as the DOE Electric Eighteen have high import reliance, at current levels of metal consumption, as shown in the USGS 2024 Commodity Minerals Summaries.

- The current materials vulnerability is highlighted by recent Graphite, Gallium, Germanium, and RE Trade Restrictions announced by China in the summer and fall of 2023.

- The Clean Energy Transition, including DOE EV Deployments and Clean Electricity Goals, will **lead to vastly higher energy critical material consumption/demand, both in the United States, and around the World, by 2030 and 2035.**

What Can We Do to Meet Burgeoning Demand?

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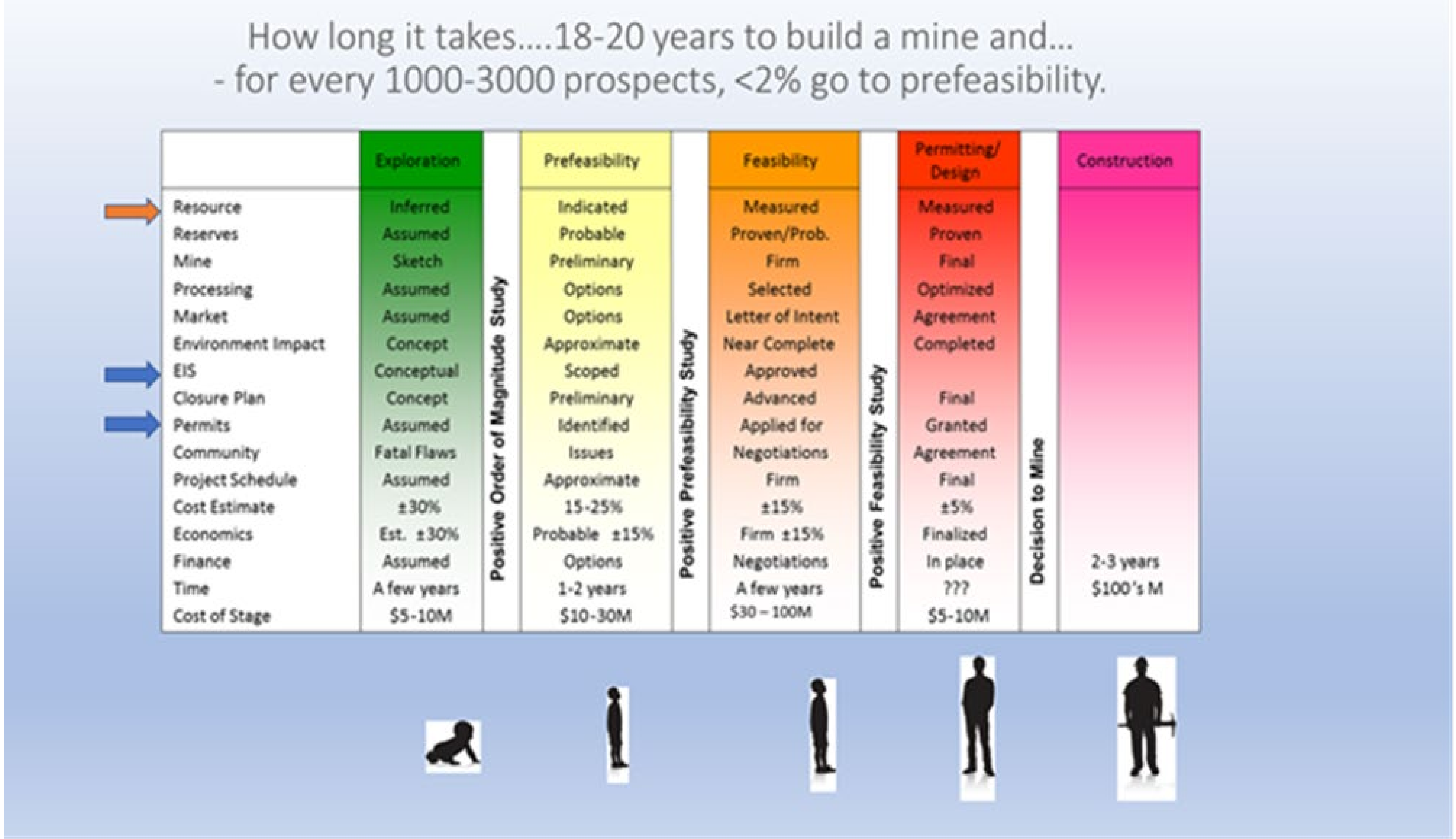
- **New Domestic Mines (Alabama, Alaska, Montana)**
- **New Domestic Processing Facilities (Cleaning the Natural Graphite)**
- This Takes Time
- On Order of 10 - 20 Years
- Resource Characterization – Extensive Sampling Program to Quantify
- Engineering Designs – Mining and Processing of Ore
- Financing – Many Millions of Dollars
- Permits – For Both Mining and Processing of Ore
- Construction and Shakedown

Typical Mine Project Timeline



How long it takes....18-20 years to build a mine and...
- for every 1000-3000 prospects, <2% go to prefeasibility.

	Exploration	Positive Order of Magnitude Study	Prefeasibility	Positive Prefeasibility Study	Feasibility	Positive Feasibility Study	Permitting/ Design	Decision to Mine	Construction
Resource	Inferred		Indicated		Measured		Measured		
Reserves	Assumed		Probable		Proven/Prob.		Proven		
Mine	Sketch		Preliminary		Firm		Final		
Processing	Assumed		Options		Selected		Optimized		
Market	Assumed		Options		Letter of Intent		Agreement		
Environment Impact	Concept		Approximate		Near Complete		Completed		
EIS	Conceptual		Scoped		Approved				
Closure Plan	Concept		Preliminary		Advanced		Final		
Permits	Assumed		Identified		Applied for		Granted		
Community	Fatal Flaws		Issues		Negotiations		Agreement		
Project Schedule	Assumed		Approximate		Firm		Final		
Cost Estimate	±30%		15-25%		±15%		±5%		
Economics	Est. ±30%		Probable ±15%		Firm ±15%		Finalized		
Finance	Assumed		Options		Negotiations		In place		2-3 years
Time	A few years		1-2 years		A few years		???		$100's M
Cost of Stage	$5-10M		$10-30M		$30 – 100M		$5-10M		

- Figure is courtesy of Lance Miller, Vice President, Natural Resources, NANA, from his presentation “Alaska Critical Mineral Resources”, DOE-DOD Seminar Series on Critical Materials, February 8, 2024.

What Can We Do to Meet Burgeoning Demand?

- Diversify and Secure International Sources
- Recycling
- Synthetic Graphite

Technology Disruptions

- Quench Demand for Battery Graphite
- Non-Graphitic Carbon Electrodes (Carbon-Silicon)
- Other Battery Chemistries

DOE Carbon Ore Portfolio

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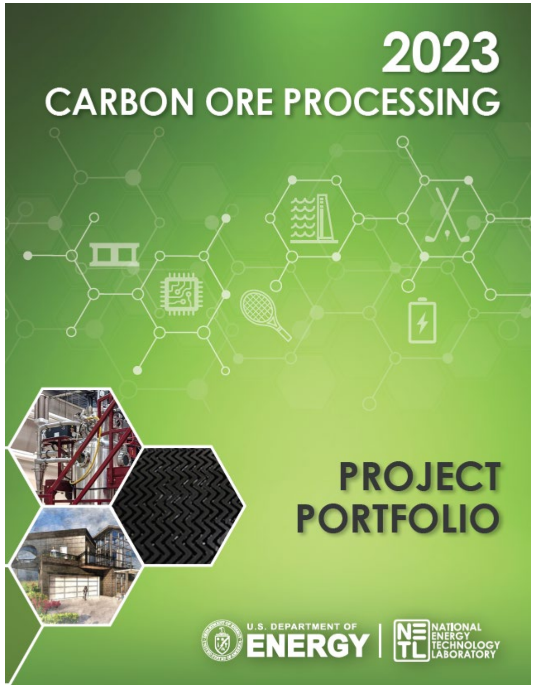
- Approximately 40 projects
- 23 active in 2024 and 2025
- https://netl.doe.gov/node/2476?list=Carbon%20Ore%20Processing
- Graphite
- Nanocarbons
- Supercapacitors
- Activated Carbons
- Silicon Carbide
- Building Materials
- Composites
- **Moving Towards High Value Products**
- Many Projects Ending



Carbons from Coal

Numerous Possibilities

- Activated Carbons and Supercapacitors
- Coke
- Chars
- Graphite and Carbon Electrodes
- Graphene
- Nanocarbons
- Composites and Alloys
- Carbon Fibers, Blocks, Roof Shingles, Deck Boards, Pipes
- Carborundum (Silicon Carbide), Diamond

Motivation for the Program

- Develop Clean Energy & Novel High Value Carbon Products to Incentivize and Facilitate Clean-Up of Waste Coal and Coal Byproduct Impoundments
- Use of Byproduct Carbons from Critical Material Recovery
- Focus on Clean Energy & Highest Value Products Such as Graphene, Nanocarbons, Graphite, Battery Electrodes, Specialty High Surface Area Activated Carbons, Novel Alloys, Fibers
- Develop High Volume Products Such as Building Materials
- Bricks, Blocks, Roof Shingles, Pipes, Deck Boards

Palette with Many Possibilities

Lignite Subbituminous Bituminous Anthracite

Low-ranking → High-ranking

Classic Analysis – Moisture, Volatile Matter, Fixed Carbon, Ash

Sequentially Dry, Pyrolyze and Burn Coal

Weight Loss From Each Step Yields – Moisture, VM, FC, and Ash (balance)

Source: Zazzeri, G. et al. (2016*). Atmos. Chem. Phys.* (16), 13669-13680. 10.5194/acp-16-13669-2016., Adapted from https://creativecommons.org

Graphitization in Nature – Coal and Graphite

Table 2. Variations of Physical and Chemical Properties with Rank and Their Useful Range as Rank Parameters[c]

Classification	% C (daf)[a] of Vitrinite	Vol. Matter % daf[a]	Moisture % in Situ	Cal. Value BTU/LB (af)[b]	Reflectance % (Vitrinite)	Important Characteristics
Peat						1. Free Cellulose 2. Plant Detail Recognizable
	60		75			
Soft Brown Coal						1. No Free Cellulose 2. Plant Structure Recognizable
		53	35	7,200	~0.3	
Lignite						1. Plant Structure Still Partly Recognizable 2. Vitrinite Formed
	~71	49	25	9,900		
Subbituminous						
	77	42	8–10	12,600	~0.5	
High Volatile Bituminous						Low-Reflecting Exinite
	87	29				
				15,500	1.1	
Med Vol. Bit. Low Vol. Bit. Semi-Anthracite						Exinite Lighter in color Exinite Vitrinite Indistinguishable
	91	8		15,000	2.5	
Anthracite Meta Anthracite Graphite						Anisotropic Reflectance
	100	0				

Applicability of Properties as a Rank Parameter: % Moisture in Situ; Calorific Value (af)[b]; % C daf,[a] Reflectance of Vitrinite; Volatile Matter daf[a]; X-Ray Diffraction; % H daf[a]

Increasing Rank

[a] daf—Dry Ash Free
[b] af—Ash Free
[c] Adapted from: "Coal and Coal Bearing Strata," (*Editors:* D. Murchison and T. S. Westall), and "The International Handbook of Coal Petrography," International Committee for Coal Petrology

Graphitization in Nature – Coal and Graphite

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Table 1. Coal and Coal-Related Carbonaceous Materials in Nature (from Schobert 1989)

Muck

Peat

Lignite ↑ closer to surface ↓ increasing age, fixed carbon & graphitization

Subbituminous Coal

Bituminous Coal

Anthracite

Graphitization in Nature – Coal and Graphite

Graphite Formed in Nature

- Elevated Temperatures/Pressures
- Or Contact with Hot Magmatic Fluids
- Typically Over Eons ("Coalification/Graphitization")
- Muck – Peat - Lignite – Subbituminous – Bituminous – Anthracite – Meta Anthracite – Graphite
- "The Geochemistry of Coal – Part I. The Classification and Origin of Coal", Harold H. Schobert, Journal of Chemical Education, 242-244, 1989.
- "The Geochemistry of Coal – Part II. The Components of Coal", Harold H. Schobert, Journal of Chemical Education, 290-293, 1989.

Graphitization in Nature – Coal and Graphite

NATIONAL ENERGY TECHNOLOGY LABORATORY

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→ Similar to Acheson Process

Albeit Much Slower!

Synthetic Graphite

- Produced by Heating Carbonaceous Materials to 3,000 – 4,000^{o}C
- For Long Periods of Time
- Acheson Process - 1890
- Others (Petroleum Coke)
- Energy Intensive
- Polluting

- DOE is Seeking Alternative (Lower Temperature) Methods

Value-Added Products from Coal

Part 1. Graphite (and Carborandum)

- Multi-Billion Dollar Annual Industry
- Various Precursors
- Thermal Formation
- Edward Acheson 1890
- Making Silicon Carbide by Heating Coke and Sand to 3000^{o}C
- Form Graphite at Even Higher Temperatures (4000^{o}C)
- By Thermal Decomposition of Silicon Carbide
- Carbon + Sand → Silicon Carbide (Carborandum SiC) → Graphite
- Anthracite an Optimum Feed for His Process

Edward Acheson (1856-1931)

- Prolific Inventor (70 Patents)
- Western Pennsylvania
- Worked for Edison
- Interested in Making Diamonds
- Industrial Abrasive
- Through Heating Carbon at High Temperatures
- Ended Up Making Silicon Carbide
- And Graphite
- Processes Used to this Day

Catalysts for Thermal Graphitization

- Abundant Literature
- Review – Rongyan Wang, "Catalytic Graphitization of Coal-Based Carbon Materials with Light Rare Earth Elements", Langmuir 2016, 32, 8583−8592
- La, Ce, Pr , Fe, Co, Ni, Si (Acheson), Ti, W, Mo, Cr
- Still Require High (Typically 2700^{o}C) Temperatures
- One of the Best CeO_2 – $CaCO_3$ or MnO_2 US Patent 3,615,209
- 2200^{o}C – 88 - 100% Graphitization
- Fe catalysts - 1300^{o}C
- "Fe based catalysts for petroleum coke graphitization for Lithium Ion battery application", Agung Nugroho, Materials Letters, 303, 130557, 2021
- Mechanisms: Carbide Formation (Acheson), Orientation, Nucleation

Low Temperature Production of Graphite

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Graphite can be synthesized from either coal or petroleum coke at high temperatures of 3000 – 4000 ^{o}C. These high temperatures make the production of synthetic graphite an expensive and environmentally damaging endeavor. The United States has over four billion tons of waste coals, scattered in over one thousand impoundments.

The DOE is investigating concepts for production of graphite from abundant waste coals, at temperatures below 1800^{o}C. This would facilitate clean-up of the waste coal sites and spur production of domestic graphite in a more environmentally friendly and economical manner.

Key Challenges in the Technology Area

- The impact of impurities in the waste coals on the formation of the graphite product.
- Disposition and mitigation of release of toxic elements within the coal such as mercury, arsenic, selenium, cadmium, phosphorus, antimony, sulfur, nitrogen, and halogens.
- Disposition of uranium and thorium present in the feed coal
- Mitigation of organic pollutants
- Economics of the lower temperature processes versus the commercial processes for production of graphite
- Verification of the final synthetic graphite product being highly suitable for use in batteries.
- Producing a brief techno-economic analysis showing creation of a significant number of stable domestic jobs.
- Demonstration of enhancing environmental justice for communities negatively impacted by the waste sites.

Recent Technical Accomplishments – Carbon Ore

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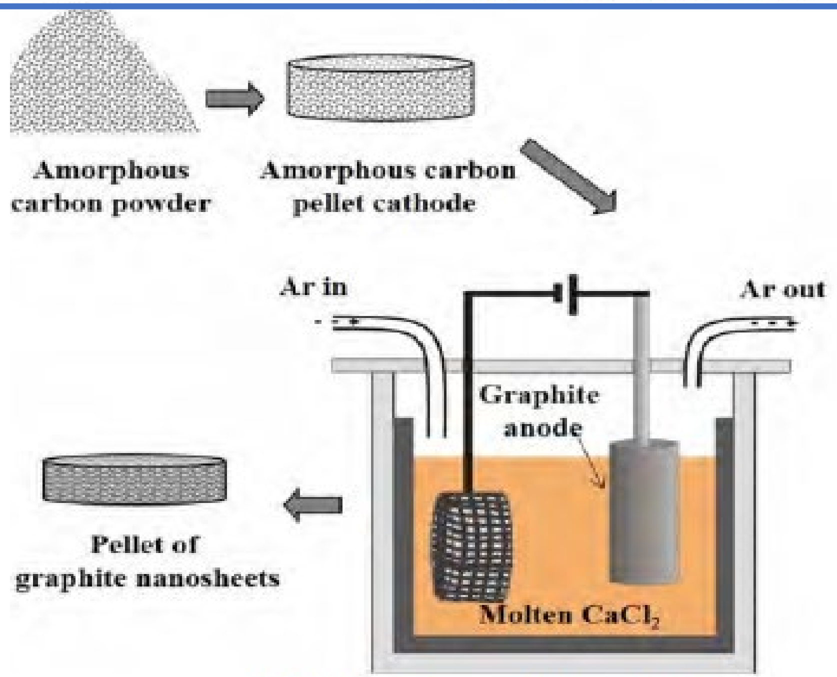
Low Temperature Production of Graphite

Graphite can be synthesized from either coal or petroleum coke at very high temperatures of 3000 – 4000°C. These high temperatures make the production of synthetic graphite an expensive and environmentally damaging endeavor. NETL discovered that iron is a surprisingly effective **catalyst for converting coal to graphite at much lower temperatures of around 1500°C.** Additionally, both the catalyst & acid used to recover it can be recycled and reused to produce graphite that performs well in lithium-ion battery tests. This effort has significance for utilizing abundant domestic waste coals for fabricating battery anodes, at large-scales. https://www.netl.doe.gov/node/12897 September 19, 2023, NETL website

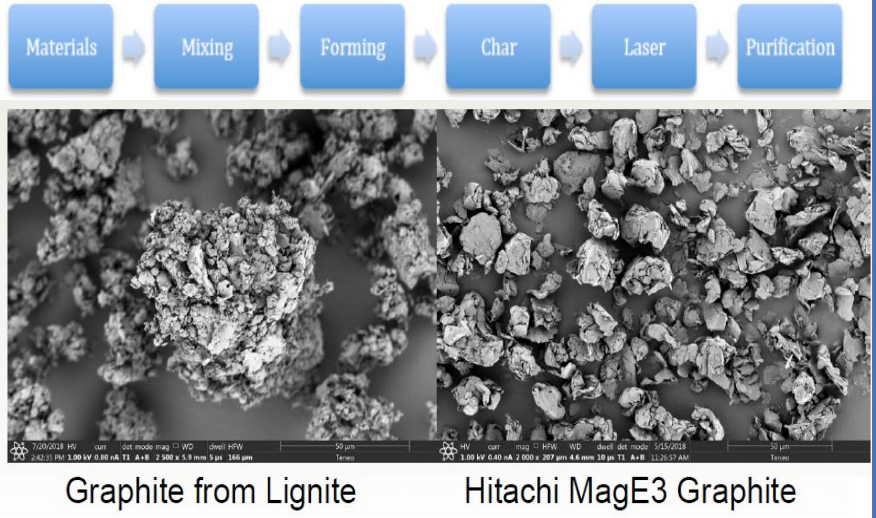
- **Other Significant Breakthroughs by George Washington University and ORNL**
- **Laser Method, Electrochemical Cell**

Transformation of Carbon Ore to Graphite

To address anticipated increase in demand, funding research on synthetic graphite



ORNL is investigating the conversion of coal char into graphite using a low-temperature electrochemical graphitization process



George Washington University is researching coal to Li-ion battery grade (potato) graphite

***FOA 2405:* "Advanced Coal Waste Processing**

Technology Disruptions

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New Battery Electrodes

- **Carbon, but not Graphite**
- Such as Carbon Silicon Materials

- **Adoption of Non-Graphite Carbons Would Disrupt Projections for Graphite Demand**
- **DOE Research**

SBIR Topic

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Direct Use of Coal or Carbonaceous Coal Wastes as Lithium-Ion Battery Anodes

- Potential SBIR Topic
- Maximum Phase I Award Amount: $250,000
- Maximum Phase II Award Amount: $1,600,000
- Less Expensive Alternative to Graphite
- 50% Coal or Waste Coal Content
- May Use Silicon or Other Materials
- Projected Explosive Demand for Carbon Electrodes in Lithium Batteries
- FY24-Phase-I-Release-2-TopicsV612082023.pdf (osti.gov)
- **Posted January 18, 2024; Applications Due in March 2024; Awards in June**
- Accepting SBIR Phase I Applications: YES
- Accepting STTR Phase I Applications: YES

Recent Technical Accomplishments – Carbon Ore

U.S. DEPARTMENT OF ENERGY

Coal-Derived Battery Anodes Show Great Commercial Promise

https://netl.doe.gov/node/12705 July 19, 2023, NETL website news item

DOE supported R&D of Semplastics' technology to utilize domestic, abundant, and inexpensive coal-derived lithium-ion battery anodes as an alternative to graphite. This technology received the Voltage Award from the Battery Innovation Center, which recognizes an emerging company and/or technology with the highest potential to make a difference in batteries and electrification. Under this agreement, coal-derived lithium-ion battery anodes have been tested extensively in 18650 cells, an industry standard size used in BEVs such as the Tesla Model S & X.

Technology Disruptions

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New Battery Chemistries and Materials

- **Nearly Infinite Number of Possibilities/Combinations**
- EMF Series – Thermodynamics of Oxidation/Reduction - Suggests Possible **Electrodes**
- **Electrolytes** – Aqueous, Solid, Ions,……
- Issues – Energy Density, Lifetime, Charge/Recharge Cycles and Kinetics, Safety, Cost, Material Availability, Fabrication, Geometry, Electrode Morphologies, …..
- **Typically Takes Years of Testing to Perfect**
- Microsoft and PNNL AI Initiative to Speed Up Battery Development
- https://quantum.microsoft.com/en-us/our-story/quantum-elements-overview
- https://cloudblogs.microsoft.com/quantum/2023/08/09/accelerating-materials-discovery-with-ai-and-azure-quantum-elements/
- **Adoption of New Batteries Would Disrupt Future Need for Graphite**
- **DOE Research**

Conclusions/Future Work

- Acheson Process and Recent Variations - Great Success
- Carbon + Sand → Silicon Carbide (Carborundum SiC) → Graphite

Challenges

- Energy Intensive (3000-4000^{o}C)
- Slow (Heating in Two Steps: 1000^{o}C, 3000-4000^{o}C)
- Impurities in Coal (and Cokes)

Future Work (Synthetic Graphite)

- Reduce Required Temperatures and Reaction Times
- Produce Other Value-Added Carbons
- Routes: Catalysts, Electrochemical (ORNL), Laser Heating (GWU)

Future Research/Work

- **Domestic Mining and Processing of Natural Graphite**
- **Recycling**
- **International Sources**
- **Alternative Battery Electrodes (Carbon – Silicon)**
- **New Battery Chemistries**

Annual Review Meetings

- Downtown Pittsburgh
- October 2022 and April 2024
- 67 Presentations on Carbon Ore Research
- Presentations are Available On-line
- https://netl.doe.gov/22RS-proceedings
- https://netl.doe.gov/24RS-COP-proceedings

Additional Information

- Much additional information is available on the NETL Carbon Ore website:
- https://netl.doe.gov/Carbon-Ore-Processing

- A factsheet is also available:
- https://netl.doe.gov/sites/default/files/2022-11/Program-151_0.pdf

Additional Information

- “Graphite Flows in the U.S.: Insights into a Key Ingredient of Energy Transition”, Jinrui Zhang, Chao Liang, and Jennifer B. Dunn, Environmental Science & Technology, 2023, 57, 3402−3414.
- “Catalytic Graphitization of Coal-Based Carbon Materials with Light Rare Earth Elements”, Rongyan Wang, Langmuir 2016, 32, 8583−8592
- USGS 2018 Minerals Yearbook, Donald W. Olson, May 2023, available on-line https://pubs.usgs.gov/myb/vol1/2018/myb1-2018-graphite.pdf
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Fossil Energy and
Carbon Management

Questions

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