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Gas to Carbon Crystals (G2CX)

DE-FE0031868

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

Brad Rupp
Xerox PARC

Agenda

1. Project Overview
2. Technology Background
3. Project Scope
4. Current Status
5. Scale-Up & Commercialization
6. Workforce Development
7. Wrap-Up

Project Overview

parc A Xerox Company	**PARC** project lead
UC RIVERSIDE	**UC Riverside** metallurgy
modular CHEMICAL	**Modular Chemical** consulting
Susteon	**Susteon** process design
PHILLIPS 66	**Phillips 66** downstream, carbon
CIVITAS	**Civitas** upstream, flare

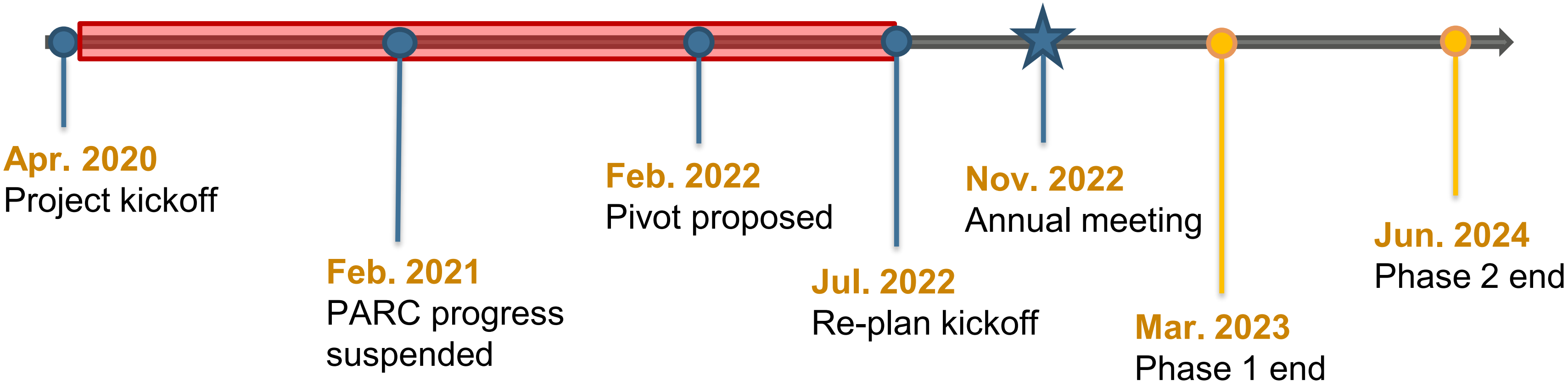
Concept

- Produce high-value carbon that meets market product specifications using carbon derived from the pyrolysis of flared gas

Objectives

- Identify high-value offtake market for pyrolysis carbon produced from PARC's proprietary methane pyrolysis process
- Develop process for upgrading pyrolysis carbon to high-value product that meets market specifications and requirements
- Scale the process and perform TEA to show commercial viability

Project Overview



- April 2020 to July 2022: progress severely slowed/hampered due to COVID-19 (~20% spent to date)
- July 2022 to now: working on pivot concept

	Federal ($MM)	Cost Share ($MM)
BP1	1.16	0.29
BP2	1.45	0.36
Total	2.61	0.65

Flare Gas in the US

Key Gaps

1. In the absence of a pre-existing pipeline, no existing technology **can monetize flared gas at the required small scale**

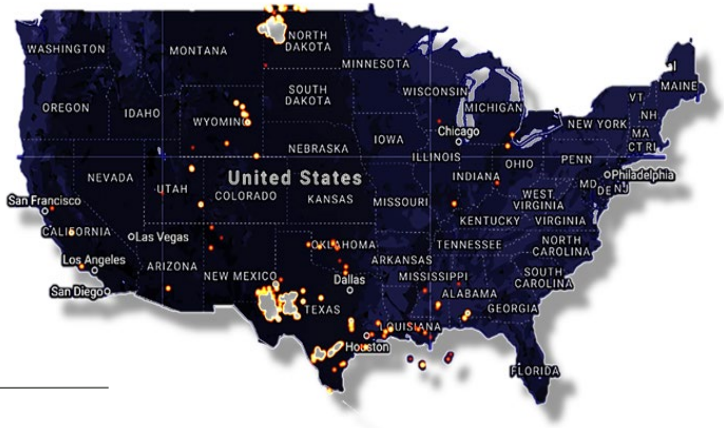
2. **Products from flared gas must have high value** to justify transportation and a market size equivalent to the flared gas problem

Flared Gas Problem

	Clustered Flares (sites)	Flared Volumes (MMcfd)	Ave Flare (Mcfd)
United States	70,749 (23%)	1,360 (10%)	8.1
N. America	96,968 (32%)	1,870 (13%)	8.1
Worldwide	303,590 (100%)	14,029 (100%)	19.3

- Ave flared site is small ~ 8 Mcf/d
- Nat gas price is low ~ 4.00 $/Mcf
- Ave value per site ~ $32 per day

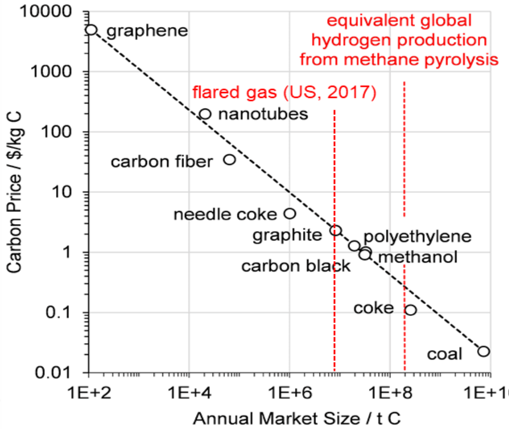
Flared Gas - United States, 2018



Market for Carbon Products

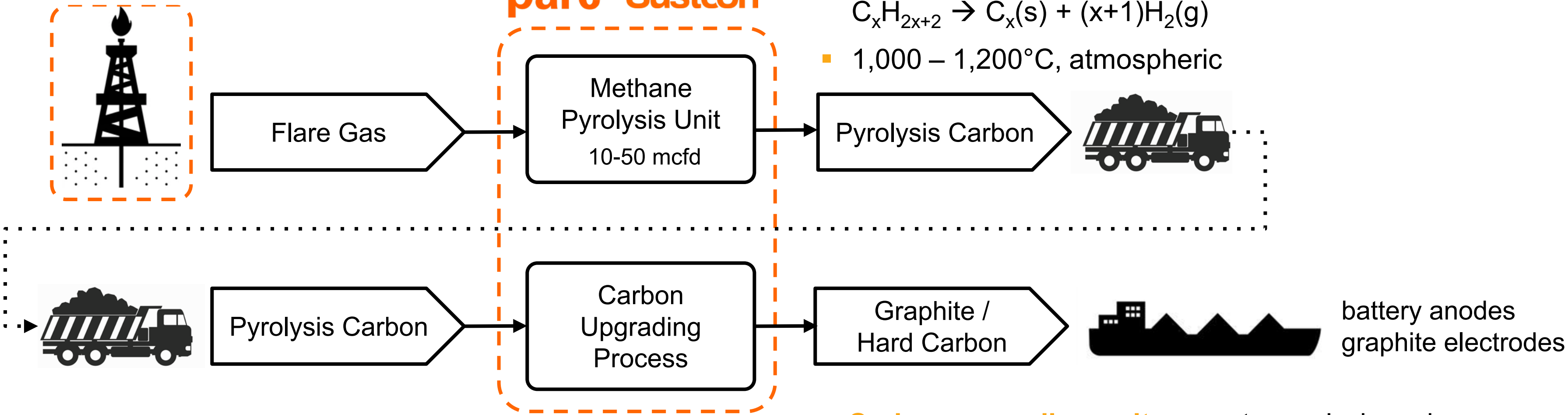
Product	Value	($/m³)	($/t-C)
Natural Gas	3.0 $/Mcf	0.11	105
LNG	6.5 $/Mcf	130	218
Methanol	380 $/t	300	142
Crude	50 $/bbl	310	289
Polyethylene	1.1 $/kg	1,034	942
Carbon Powder	2.0 $/kg	900	2,000
Carbon Fiber	30 $/kg	53,000	30,000

Carbon Price vs Market Size



Concept

- Natural gas **pyrolysis unit** to turn natural gas into solid carbon
  $C_xH_{2x+2} \rightarrow C_x(s) + (x+1)H_2(g)$
- 1,000 – 1,200°C, atmospheric



- **Carbon upgrading unit** converts pyrolysis carbon into higher value product
- 1,300 – 1,600°C, atmospheric

Properties of the pyrolysis carbon product will be modified through:

1. adjusting the methane pyrolysis unit's operating parameters, and
2. developing a standalone carbon upgrading process

Civitas Flare Gas Site Composition

Flare Composition

Component	Mol%
Nitrogen	5%
Carbon Dioxide	2%
Hydrocarbons	93%
Methane	29%
Ethane	19%
Propane	22%
C4+	23%

Certificate of analysis: 03/15/2022

Site Considerations

- Source of current flare gas is vapors from oil tanks
- Potential for small amounts of H_2S (0-10 ppm)
- Ability to handle N_2 and CO_2
- Wide range of volumes available based on location (5,000 SCF/Day up to 100,000 SCF/Day)
- Equates to ~ 300 – 6,000 kg/day carbon produced
- Pilot-scale pyrolysis module would need to be able to shut down instantly if the facility has a safety shut down

1 SCFD = 28.3 SLD

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Project Tasks

1. Project Management and Planning
2. Carbon Fiber Production
3. Materials and System Modeling
4. Materials Characterization
5. Experimental Reactor

Phase 1

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6. Bench-Scale Reactor
7. Bench-Scale Prototype
8. Pilot-Scale Reactor

Phase 2

Milestones & Success Criteria

Subtask	Milestone Title and Description	Anticipated Completion Date
4.2 Go/No-Go	Proof-of-concept demonstration of high-value carbon	03/31/2023
5.3	Batch production of high-value carbon	06/30/2023
1.3	Techno-economic model shows the feasibility of a process producing upgraded carbon which meets specs defined in Subtasks 2.1, 2.2, and 3.2	09/30/2023
6.3	Commission of bench-scale carbon upgrading prototype	12/31/2023

Phase 1

- Proof of concept (lab-scale) carbon upgrading process
- Preliminary TEA supporting economic process

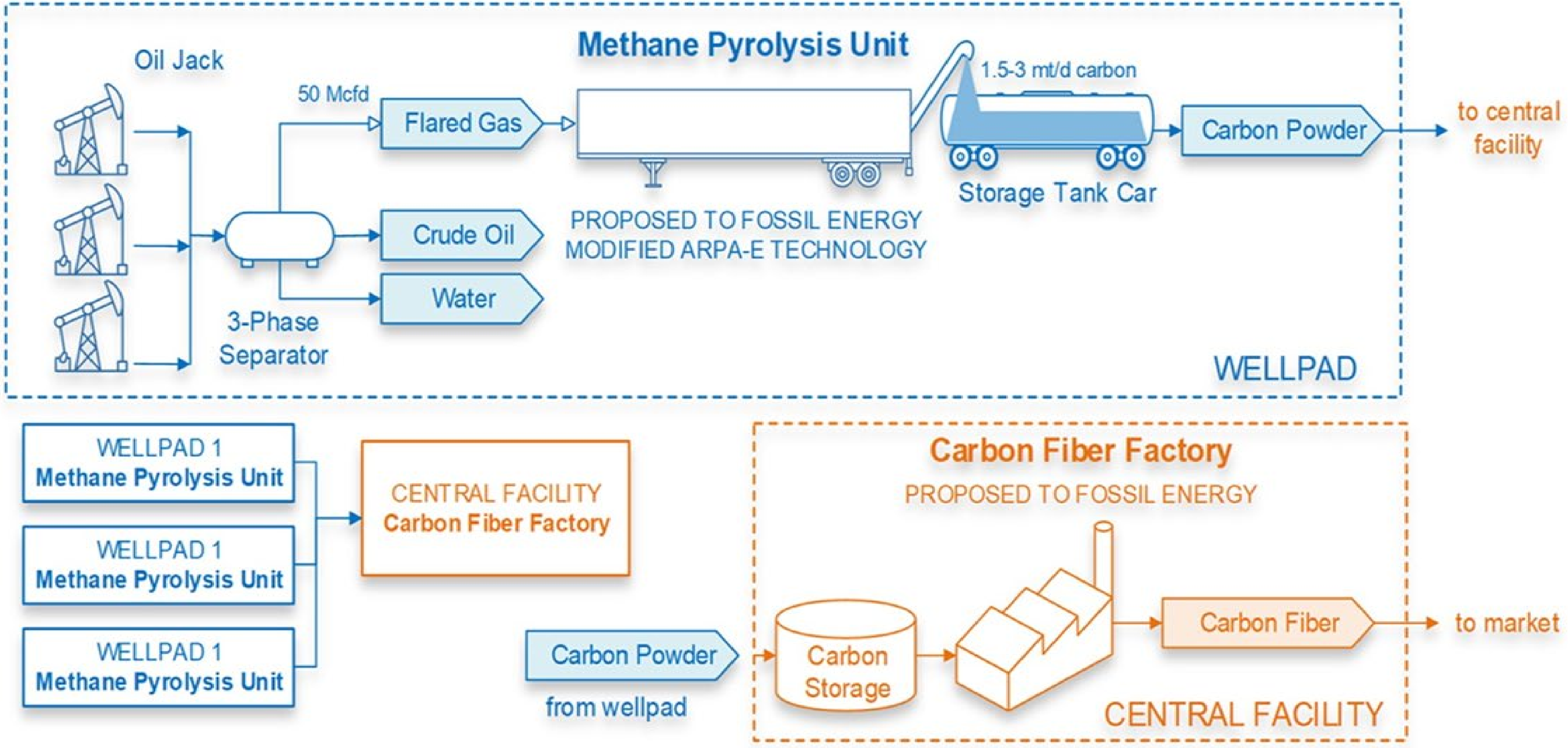
Phase 2

- Integrated bench-scale carbon upgrading prototype
- Design for scale-up prototype

Risks & Mitigation

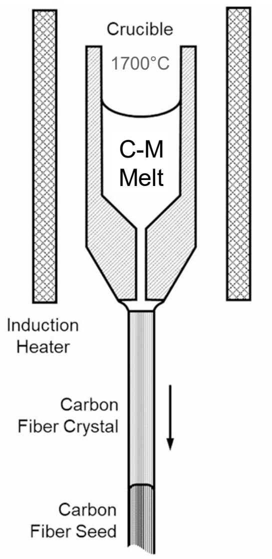
Risk	Potential Impact	Mitigation Strategy
Upgraded carbon quality insufficient to meet market spec	No offtake market for carbon product, unable to make business case	Expand market discovery options beyond graphite electrode and battery anodes
Laboratory activities delayed by COVID	Pace of laboratory work may be slow due limited laboratory access; parts procurement	Optimize the scheduling lab access to minimize our team's virus exposure; maintain larger materials/parts inventory
Pyrolysis reactor space time yield too low	Pyrolysis process becomes uneconomical	Evaluate other pyrolysis approaches. The project already includes exploring an upgrading method that is agnostic to type of carbon feedstock.
Flared gas contaminants could impact process performance	Process could become uneconomical	Evaluate pilot site gas compositions early in program
Equipment build delays due to supply chain issues	Due to the impact of COVID and other world events on supply chains, procurement of parts may be challenging and delay the timeline.	The team will identify components and parts needed early on and prioritize activities to minimize delays.
Environmental barriers for ultimate process	If carbon nano-particulates or NOx are produced during pyrolysis or carbon upgrading, public and environmental health concerns will arise.	Process equipment will be designed and operated to conform with OSHA standards. The cost of including ultra-low NOx H_2-fired burners will be explored as a part of the process modeling task.
Availability of project team members	If team members are unable to spend the time required to achieve assigned tasks, the project progress may be delayed.	We will review the Gantt chart, progress against targets, and time needed to complete required tasks with the team at least once per month in order to ensure sufficient time to plan ahead and distribute resources accordingly to accomplish the tasks at hand.
Cost-share	If cost-share commitments are not upheld, the cost-share requirements and project budget could be impacted.	Over 90% of the cost-share is provided by PARC and will be completed as costs are incurred. As such, risk of cost-share loss is expected to be low.

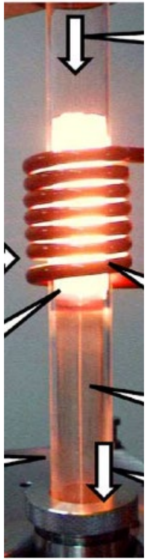
Previous Concept – Natural Gas to Carbon Fiber



Carbon Fiber Pulling

C-FIBER GROWTH





MICRO-PULLDOWN TECHNIQUE

- Developed in Japan over last 20 years for optics industry
- R&D led by Professor Tsuguo Fukuda
- Commercial systems available in Japan (exclusively)

Fukuda, Inventor

Commercial Reactor

PROJECT RISK

No	Description	Risk	Potential Impact	Mitigation Strategy
2.	Unable to acquire crystal growth equipment	Major (6)	Will have to build equipment in-house, resulting in a major project delay/setback	Make our first task to determine feasibility of leasing/buying μ-PD system

Combination of language barriers and COVID restrictions prevented establishing an effective collaboration with key Japanese researchers critical to developing and acquiring a commercial micro-pulldown reactor

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Carbon Fiber Summary

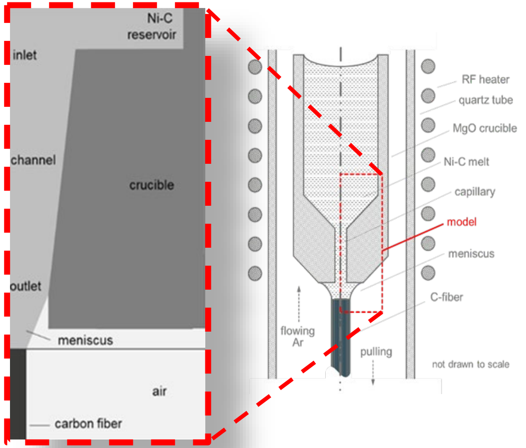
What did we accomplish?

- **Validated carbon fiber growth rates.** Multiphysics model demonstrated C-fiber growth rates over 1 mm/min and established a crucible design basis.
- **Identified thermochemically stable crucible materials.** Magnesia, yttria-coated tungsten, and yttria-coated graphite can meet aggressive reactor conditions (1800–2200 °C in carbon-saturated molten nickel).
- **Verified commercially viable process economics.** Estimated MSP of $1.28/kg C-powder for Methane Pyrolysis Module (1600 kg/d carbon, 200 Mcfd gas) and $15.6/kg C-fiber for Carbon Fiber Plant (8000 kg/d carbon). Today carbon fiber sells for over $30/kg C-fiber.

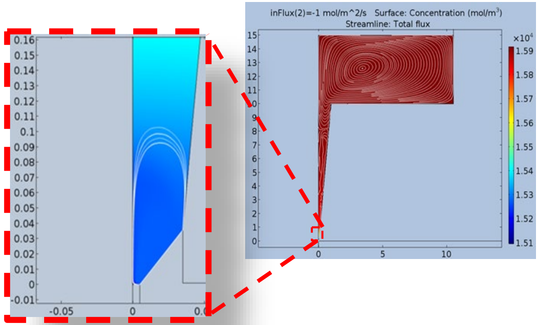
What is our current challenge (status)?

- **Unable to effectively communicate with key Japanese researchers.** Collaboration with Japanese leaders in micro-pulldown crystal growth proved too logistically challenging.
- **Impractical to build crystal growth system in-house.** Japan developed micro-pulldown crystal growth reactors over 20-year time period.

Reactor Model

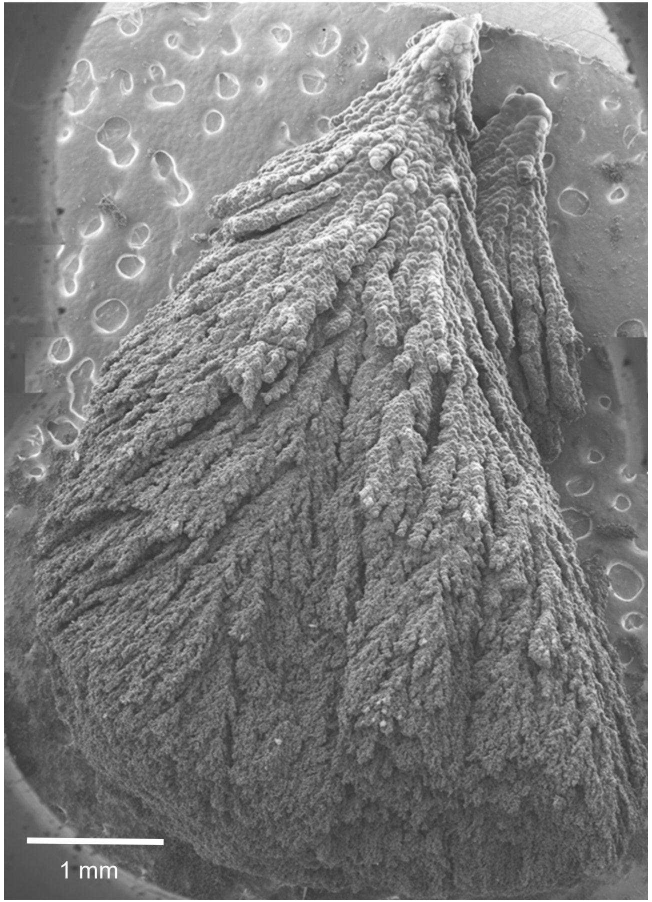


COMSOL Simulation



Graphite Production from Pyrolysis Carbon

	Graphite Price Drivers (low value → high value)
Chemical Purity	80% → 99.999%
Crystallinity	amorphous → large crystals
Particle Size	100’s nm → 10’s µm
Shape	irregular → spherical
Surface	rough → smooth
Porosity	high → low



Aim for high purity and crystallinity through carbon post-processing and/or pyrolysis reactor optimization

Amorphous Carbon to Graphite

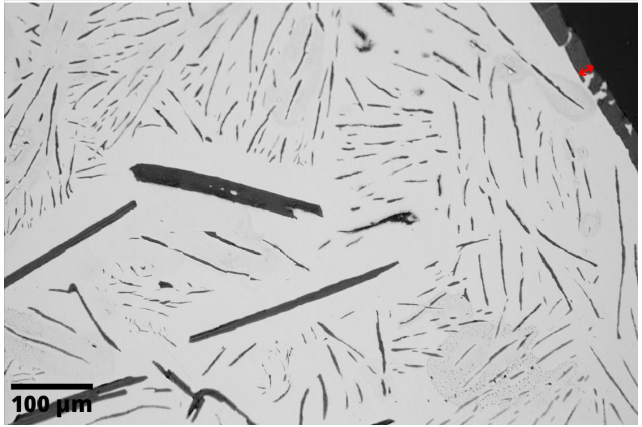
Powdered spherical glassy carbon

Reactor crucible packed with bulk carbon prior to operation

Apparatus during operation

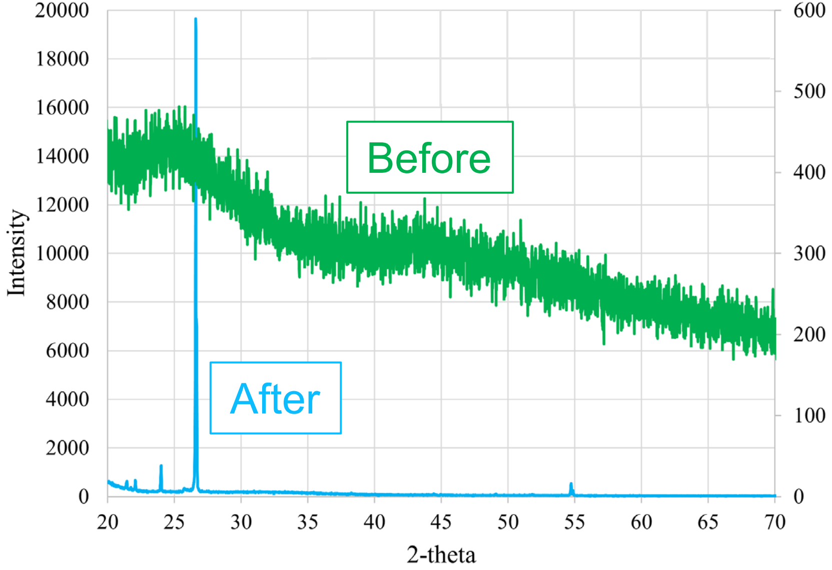
Amorphous Carbon to Graphite Results

- Amorphous glassy carbon precursor
- 2-theta peaks at 26.5° and 54.5° correspond to graphite
- Lack of peak at 52° means there is little nickel left
- Sharp peak at 26.5° means the graphite is highly crystalline



X-Ray Diffraction

Heat treatment in reactor to 1500°C

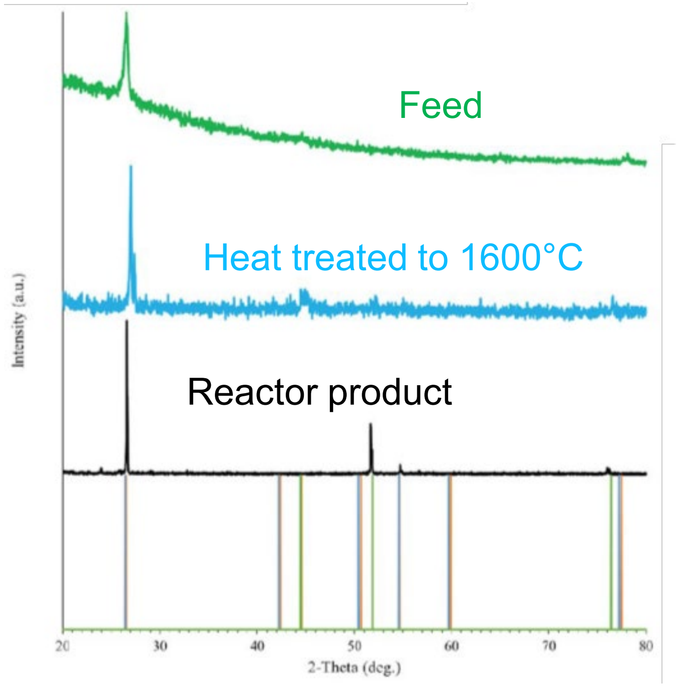


Graphitization reactor produces highly crystalline graphite at lower temperature

Experimental Status

- Have shown good preliminary results for carbon upgrading process
- Have shown crystalline product with a number of different precursor reactants
- Will test new material systems for reactor catalyst
- Batch reactor now (grams)
- Designing semi-batch / continuous process for graphite production

Advantage over current graphitization process, operates at ~1500°C lower



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Scale-Up & Commercialization

Scale-Up

- Grams → kilograms of graphite product
- Bench-scale reactor (1-10 kg/day)
- Techno-economic analysis to determine appropriate size scale
- Design for larger pilot plant

Commercialization

- Too early to tell
- Spin-off or joint development

Workforce Development Efforts

- Thus far graduated one PhD student and one post-doctoral student
  - Steven Herrera (UC Riverside)
  - Hooman Sabarou (UC Riverside)
- Onboarding one post-doctoral student
  - TBD (UC Riverside)

Summary

- Pivoted technology away from original concept of carbon fiber production
- Developed new concept around upgrading pyrolysis carbon to graphite
- Shown good preliminary results producing highly crystalline graphite in our experimental reactor at temperatures much lower than commercial graphitization process
- Working on scaling up process to test graphite for electrical properties

THANK YOU

Brad Rupp, PhD
brupp@parc.com
Work: (650) 812-4144

PARC, a Xerox Company
3333 Coyote Hill Road
Palo Alto, CA 94304

Appendix

Organization Chart

Organization	Contributors	Roles & Responsibilities	
parc A Xerox Company	Dr. Brad Rupp (PI) Dr. Jin Ki Hong Dr. Jessica Medrado Dr. Aravindh Rajan Dr. Daniel Bullard Mr. Ze He	▪ Project Management ▪ Pyrolysis Process Development	▪ Pyrolysis Carbon Production ▪ Bench-Scale Pyrolysis Process
PHILLIPS 66	Dr. David Ingram Dr. Neal McDaniel Dr. Nasim Haji Akbari Balou Dr. Jim Seaba	▪ Carbon Characterization ▪ Carbon Upgrading Process ▪ Carbon Market Evaluation	▪ Process Modeling & Design ▪ Techno-Economics ▪ Commercialization Partner
CIVITAS	Mr. Sheldon Mullet, PE	▪ Flared Gas Site Host (Future) ▪ OSBL Site Evaluation	▪ Business Case Development ▪ Commercialization Partner
UC RIVERSIDE	Prof. Reza Abbaschian Mr. Steven Herrera Ms. Raquel Jaime	▪ Graphitization Proof-of-Concept ▪ Carbon Characterization	▪ Bench-Scale Graphitization
Susteon	Dr. Raghubir Gupta Dr. Vasudev Haribal	▪ Process Modeling & Design ▪ Techno-Economics	▪ Commercialization Advisor
modular CHEMICAL	Dr. Dane Boysen Dr. Mary Louie	▪ Carbon Product Characterization	▪ Graphitization Process Design

Project Tasks

Process Feasibility

Scale-Up