Developing Flame Spray Production Level Process for Active Materials

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Project ID: ES315

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Washington, D.C.

Overview

Timeline

- Project start date: Jan. 2016
- Project end date: Sept. 2017
- Percent complete: 95%

Budget

- Total project funding:
  - $500K in FY16

Barriers

- 2.1.1 A: Cost of Li-ion batteries
- 2.1.1 C: Performance of Li-ion batteries

Industrial Partners

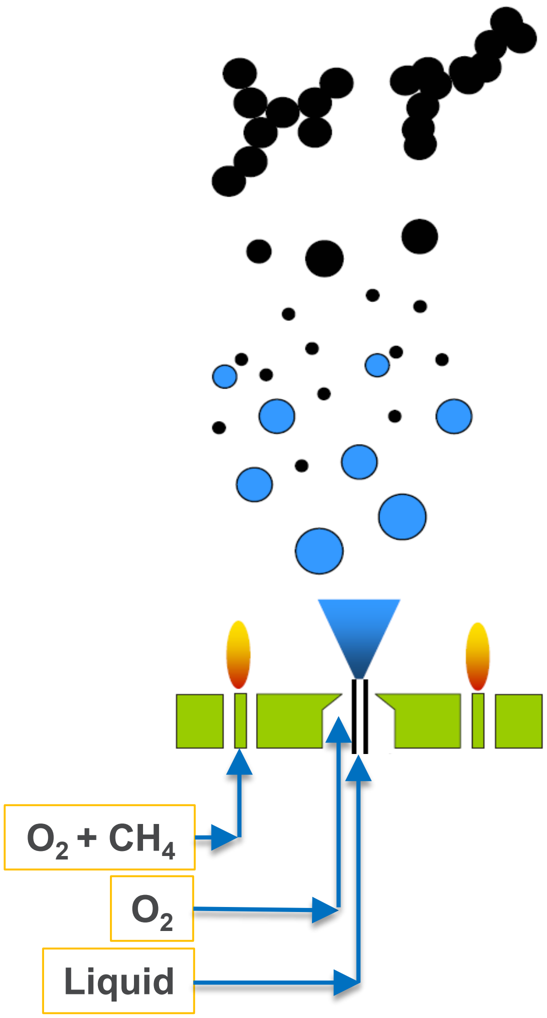
- Cabot Corporation
- Praxair Specialty Ceramics

Supporting battery research for:

- DOE Battery Research Community

Approach and Strategy

- Flame Spray (combustion synthesis) is a proven industrial technology for commodity scale production of numerous simple compounds (TiO_2, C black, SiO_2).
- R&D using flame spray for battery materials is ongoing around the world in academic and industrial settings showing the potential promise of this approach.
- This effort will specifically follow the guidance of our industrial partners to assure sensible approaches to achieve scalability and low cost.



Approach and Strategy

- Establish a flexible flame spray pyrolysis R&D synthesis facility for evaluation scale materials production:
  - Daily production of 5 hours x 20 g/hr = 100 g.
  - High temperature residence time section up to 900 deg C.
  - Clean-in-place for rapid clean-up and minimization of run-to-run drag out.
  - Modular engineering for exchangeable burner designs.
    - First deployment: Cabot Spray Pyrolysis (aqueous solvent) or Flame Spray Pyrolysis (organic solvent).
  - Robust engineering controls for safe facility operation and nanomaterial control.

- Future integration with Argonne basic science capability:
  - Chemical analytics for particle formation in flames.
  - CFD simulation with Multiphysics of the solution combustion and particle formation.
  - Advanced Photon Source (APS) in-situ beamline for particle genesis diagnostics.

Approach - Milestones

FY15	Project start	*Completed*	Feb-16
	Facility Specification	*Completed*	May-16
	Facility Design	*Completed*	Aug-16
	Place major Equipment orders	*Completed*	Aug-16
	Burner Design	*Completed*	Sep-16
	Control System Design	*Completed*	Sep-16
	NFPA 86 Burner Control Panel recvd	*Completed*	Oct-16
	Spencer Blower recvd	*Completed*	Nov-16
FY16	Installation of fume hoods and ventilation	*Completed*	Jan-17
	Construction of facility	*Completed*	Apr-17
	Burner Tests	*Target*	May-17
	Materials production	*Target*	Jun-17

Objectives - Relevance

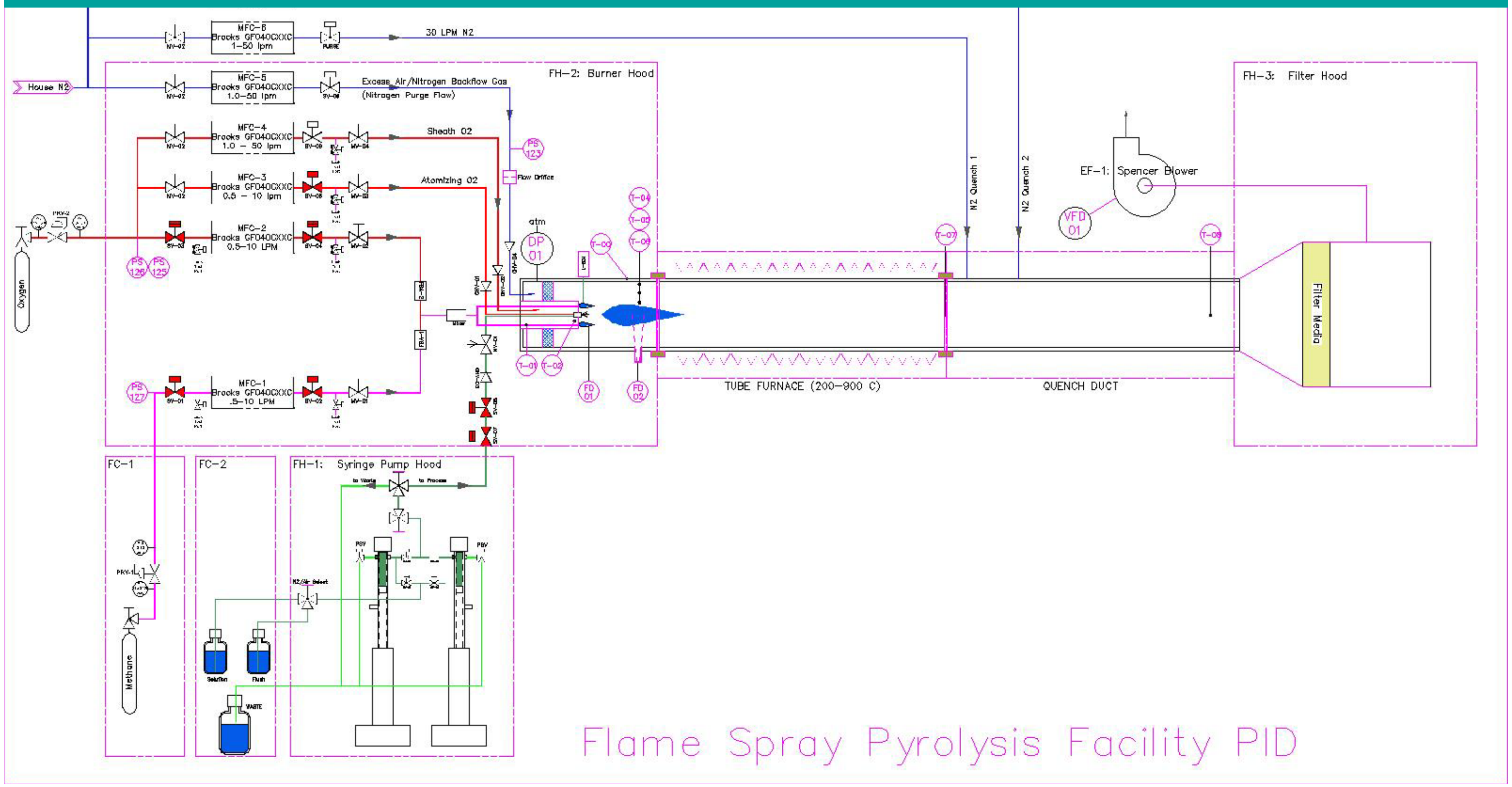
- The objective of this task is to establish a flexible R&D capability in the MERF for developing Flame Spray Pyrolysis (FSP) as a synthesis method for battery cathode materials
  - Develop FSP battery materials with industrial partner guidance to assure relevance to sensible scale-up strategies.
  - Provide quantities of high quality materials sufficient for industrial evaluation.
  - Explore simplification of battery manufacturing by eliminating of calcination, combining cathode powder and carbon matrix, direct deposition of cathode/carbon material onto electrode substrates (roll-to-roll schemes)
- The relevance of this task to the DOE Vehicle Technologies Program is:
  - This synthesis technique has the potential to provide large cost reduction through continuous high-volume production methods.
  - The high purity and crystallinity of FSP materials has the potential to improve performance for the same materials synthesized by other means.

Technical Accomplishments And Progress Overview

- System Specifications
  - Fully enclosed and atmosphere controlled process path (no induced draft air flow)
  - Pilot flame 25,000 Btu/hr
  - Spray flame 25,00 Btu/hr
  - Liquid feedrate1-10 mL/min
  - Oxygen atomizing spray nozzle
  - 20 g/hour solids production rate
  - 72” x 5” ID quartz tube furnace high-T (< 900 C) residence time section
  - Gas quench cooling section
  - Up to 200^{o}C filtration section
  - 40 in-W.C @100 cfm Spencer blower to support hot-gas filtration/material-collection down to nanometer particle size.
  - Mass flow controllers for all gas streams
  - ISCO syringe pumps for liquid feed.
  - Labview based system integration and process control
  - NFPA 86 compliant flame supervision controls

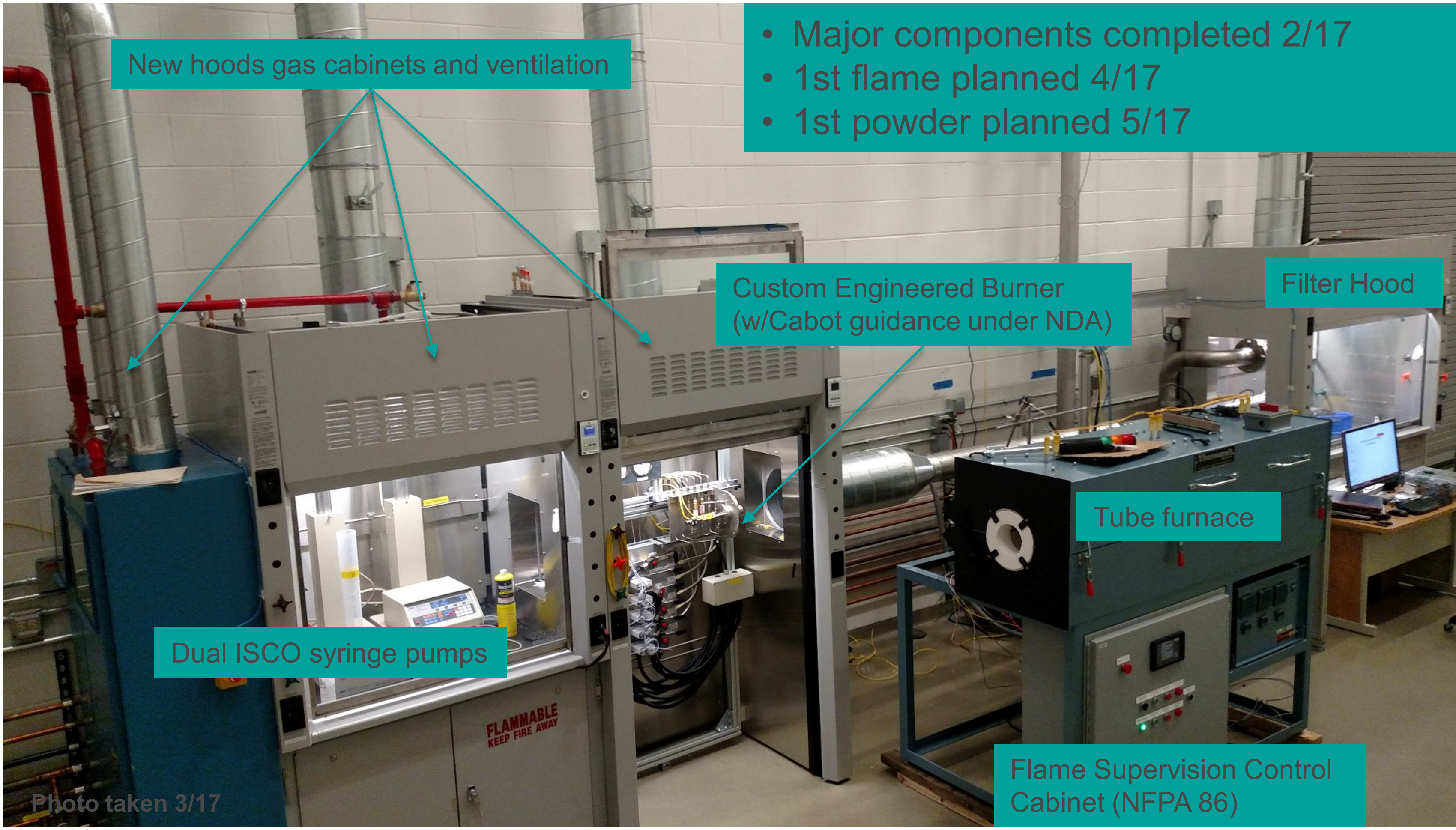
Technical Accomplishments And Progress Overview

- System Engineering completed 7/16
  - Cabot burner design
  - New Ventilation system for combustion gas/particle separation, nanoparticle control.
  - Dual ISCO syringe pumps for liquid feed
  - Commercial NFPA 86 burner controls panel
- Major components ordered 7/16



Technical Accomplishments And Progress Overview

Facility Construction



- Major components completed 2/17
- 1st flame planned 4/17
- 1st powder planned 5/17

Technical Accomplishments And Progress Overview

Flame Supervision and Control Panel (FSCP)

Flame Supervision Control Cabinet (NFPA 86)

- NFPA-86 Compliant Burner Safety Control
  - Honeywell SLATE hardware
  - Separate controllers for Pilot and Spray flames
  - Flame rod detection of pilot flame
  - UV scanner detection of Spray Flame
  - N2 purge period enforced before any light-off attempt/re-attempt.
  - Interlock chain includes: E-stop, Loss-of-flame, Positive pressure in exhaust duct, Over-temperature in tube furnace, quench duct, filter box and blower, gas pressure in-range
  - Optional pilot operation when Spray Flame is on (optional combustion support)
  - Full integration with the Labview based experiment control and following NFPA-86 rules

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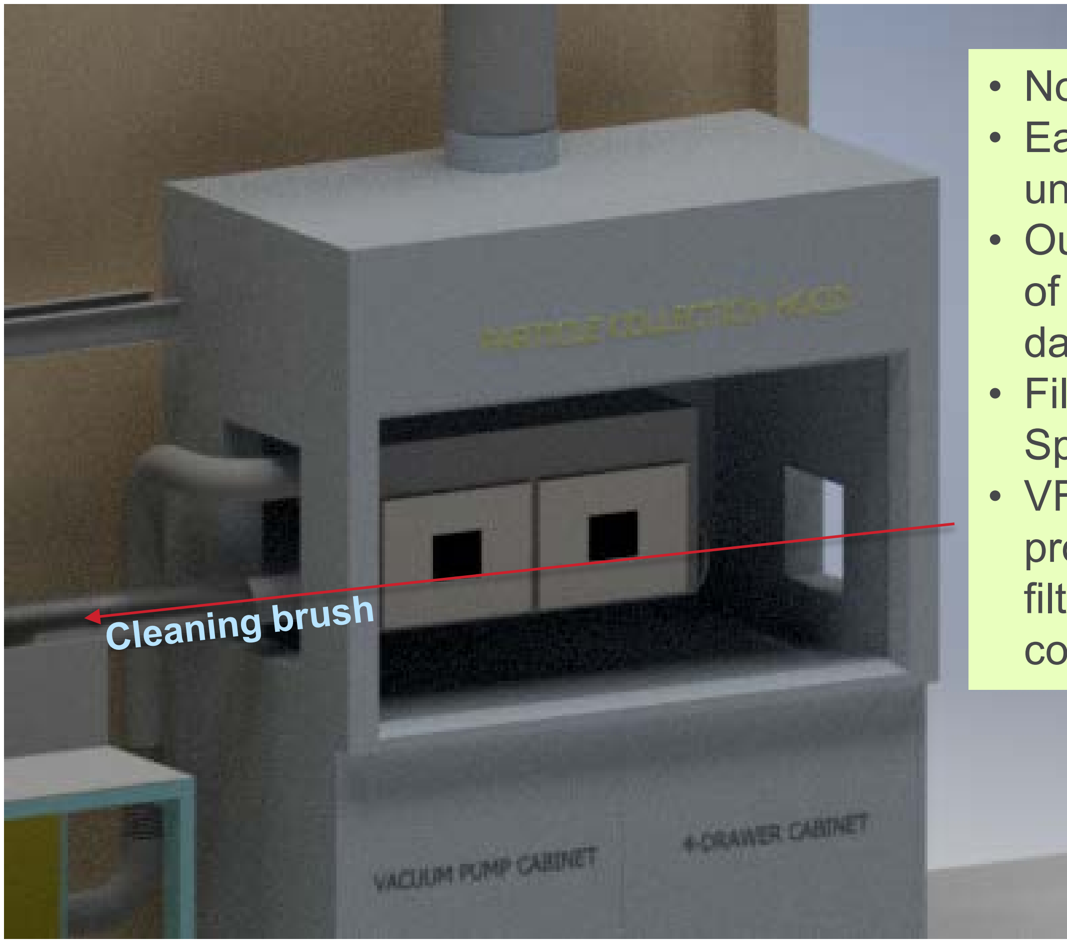
Technical Accomplishments And Progress Overview

Particle Collection



- Normal filters - 3 ft^2 (3 12”x12” pads)
- Easy access doors to collect material under fume hood sash
- Outboard access port for deployment of a clean in place power brush for day-to day cleanout.
- Filter powered by a 40 inch W.C Spencer Blower
- VFD on blower with a PID loop provides automatic adjustment of filter pressure drop to maintain constant combustion flowrate.

Collaboration and Coordination with Other Institutions

- Cabot Corp. has providing engineering guidance for burner technology and will provide guidance for synthesizing NCM materials.

CABOT

- Praxair (Specialty Ceramics and Surface Technologies groups) have expressed interest in developing cooperative research based on their combustion spray technology.

PRAXAIR
SURFACE TECHNOLOGIES

- Professor S. Pratsinis

ETH zürich

- Future Argonne collaborations being established:
  - CSE Analytical Chemistry screening of combustion reaction chemistry using shock tube studies.
  - Advanced Computing Facility (ACF) combustion and particle formation modelling
  - Advanced Photon Source: Ex-situ diagnostics: (a)Pair Distribution Function, (b) X-ray Diffraction, (c) Small Angle Scattering, (d) EXAFS. In-situ beam-line facility for particle formation diagnostics in flames.

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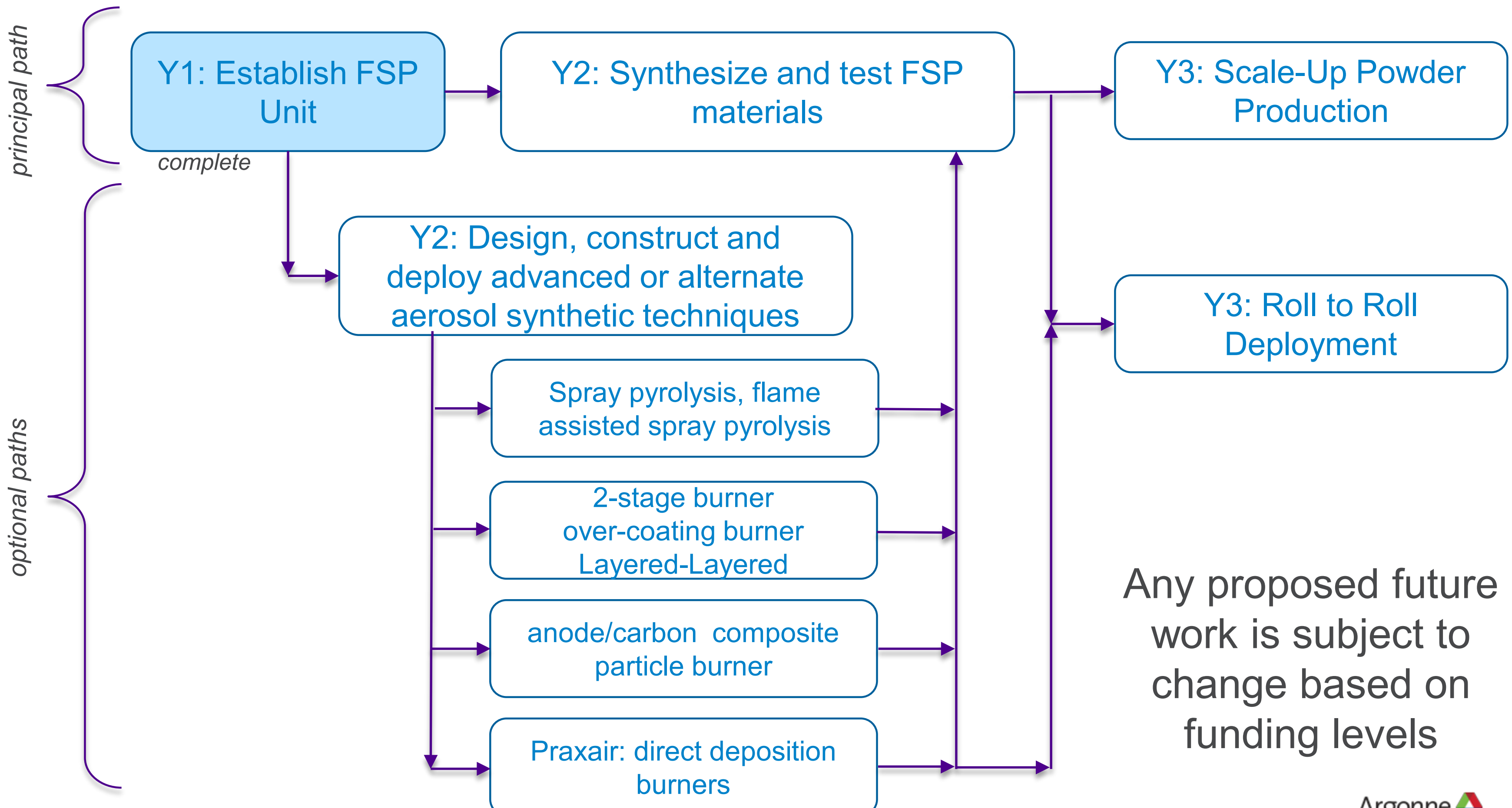
Remaining Challenges and Barriers

- Optimize the new MERF FSP facility for effective day to day operation to attain a high throughput R&D sample production.
- Discover performance and cost competitive NCM and other battery materials

Proposed Future Research

- The modular design of the MERF FSP Facility will permit exchanging the burner components to permit exploration of the different variations of aerosol synthesis while maintaining a high sample throughput of the principal path.



Any proposed future work is subject to change based on funding levels

Summary Slide

- The principle goal of establishing an FSP capability at the Argonne MERF facility has been completed.
- Industrial collaborations have been established for battery materials development moving forward. Plans are in place to begin NCM development with Cabot. Negotiations are in progress with Praxair to do the same.
- The modular design of the facility facilitates flexibility to explore all forms of aerosol synthesis including using equipment loans from our industrial partners.