



Moving from Batch to Continuous
Production – Challenges & Risk Mitigation
for High Temperature Ceramics Processing

Dr. Prasad Apte, Harper International
Ceramics Expo, April 2015

Today's Agenda

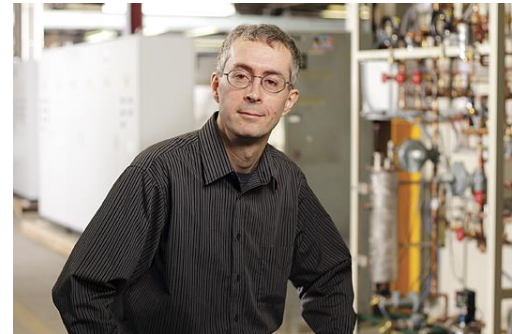
"Moving from Batch to Continuous Production – Challenges & Risk Mitigation for High Temperature Ceramics Processing"

- About Harper
- Challenges & Risks
- Scale Up Objectives
- Scale Up Success Factors
- Case Study
- Conclusion



About Harper

- Headquartered in Buffalo, NY
- An Employee-Owned Company
- Technology & Piloting Facilities
 - Harper / Buffalo, NY - particulate processing
 - Partner Facilities – Carbon Fiber line (ORNL), UHT Pusher furnace (UDRI)
- Manufacturing Facility
 - Equipment assembly and testing
- Multi-Disciplined Engineering Talent
 - Chemical
 - Ceramic
 - Mechanical
 - Electrical
 - Industrial
 - Process & Integration



About Harper

- > Established Leader in Thermochemical Processing Systems
- > Key Partner in Carbon Fiber Scale Up and Manufacturing

Primary Technical Focus:

- New / Challenging / Advanced Material Processing
 - 200°C – 3000°C
 - Batch and Continuous processing
 - Precise atmospheric controls
 - High purity requirements
 - Complex gas-solid interactions



Challenges in New Material Process & Product Development

Technical Success

- Product quality
- Handling of byproducts and wastes
- Large scale processing
- Continuous operation

Development Costs

- Cost of pilot scale equipment
- Resources for trials
- Analytical costs

Time Required for Development



Objectives in Thermal Processing Scale Up

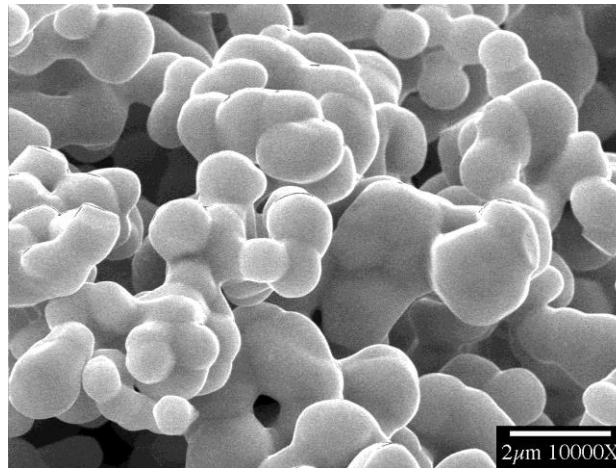
- ✓ Determine the feasibility under industrial conditions
- ✓ Reduce the development risk and the associated cost
- ✓ Generate data for scale up
- ✓ Develop a safe process that will meet environmental regulations



Scale Up Success - Test Facilities

Proper Sizing of Scale-Up Equipment

- Small enough to enable testing without using large amounts of raw material
- Large enough to provide adequate product for sampling
- Large enough for data generation to be used for commercial scale up
- Large enough to utilize readily available feeders, gas regulators, collection set up



Scale Up Success - Starting Status

- **Gram scale product**
 - Batch process
 - Generally static operation
 - Low gas flows
 - Well analyzed product
- **Data available**
 - Effect of temperature
 - Reaction time for static conditions
- **Data not available**
 - Effect of gas flow rate
 - Effect of bed depth
 - Gas solid contact



Scale Up Success - Desired Final Information

- Defined processing window
- Optimum values for -
 - feed rates
 - residence times
 - gas flow
- Design for -
 - Material feeding systems
 - Product removal systems
- Material entrainment in gas
- Effluents in the exhaust chemistry, quantity



Cascading system with multiple atmospheres

Scale Up Success - Information Generated in the Trials

- Rate limiting reaction kinetics
- Gas solid contact and methods to optimize it
- Techniques for -
 - suppressing entrainment
 - minimizing contamination
 - avoiding condensation/recycling from exhaust gases
 - raw material feeding and product discharge and collection
- Handling of exhaust gases
- Benefits of co-current or countercurrent flow

Scale Up Success - Data Analysis and Equipment Design

Data from laboratory tests will:

- ✓ Define the processing window
- ✓ Help develop process flow diagrams
- ✓ Provide scale up information for commercial production

Follow up engineering analysis:

- ✓ Equipment size scale up
- ✓ Thermal and stress modeling
- ✓ Define feed and product collection systems
- ✓ Determine gas handling systems
- ✓ Determine OPEX and CAPEX

Scale Up Success - Data Analysis and Equipment Design

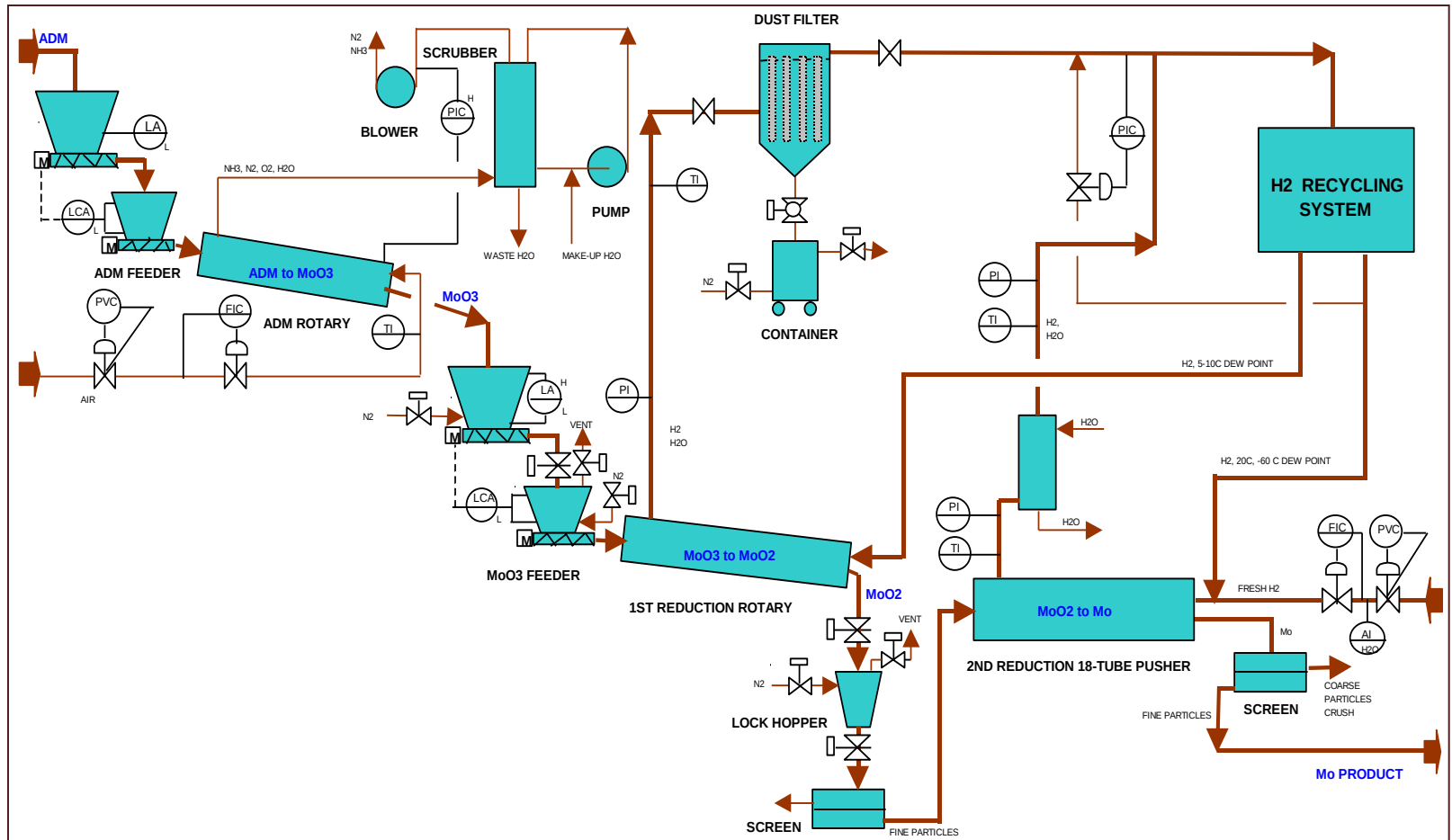
- **OPEX Estimation**

- ~80-90% of total cost over lifetime
- Process cost models
- Economics of increased production capacity with current and future technologies
- Analysis of best-suited thermal process technology system
- Identification of opportunities for improved product quality and cost reduction

- **CAPEX Definition**

- ~10-20% of total cost over lifetime
- Based on scale-up from trial data collected
- Scale up of processing equipment to meet desired production targets
- Design integration to handle feeding, product handling, gas systems and effluent mgmt
- Prepare a capital cost estimate

Case Study: Molybdenum Production





The Ignite™ program aims to help the progression of a discovery, an invention or a concept from a small, batch scale to a commercial stage.

- Helping customers turn the next generation of material innovations into profitable new markets
 - Utilizes our depth and breadth of experience in thermal processing
-
- ✓ Reduce Risk
 - ✓ Control Cost
 - ✓ Parallel Development
 - ✓ Controlled Scale-up
 - ✓ Successful Commercialization



Stage 1 - Prospecting

Gate I

Estimate Budget: \$ & Time

- Idea Generation
- Thought Experiments
- Confirm Thermodynamics
- Confirm Novelty (IP)
- Why is our idea better?

Technical Activities

Initiate
technical trials
for POC

- Industry Need
- Potential Customers
- Assuming complete success, what is the potential market?
- Anyone else doing it?

Commercial Activities

Best Done by Inventors

Stage 1 - Prospecting

Stage 2 – Initial Testing

Gate I

Gate II

Estimate Budget: \$ & Time

Estimate Budget: \$ & Time

- Idea Generation
- Thought Experiments
- Confirm Thermodynamics
- Confirm Novelty (IP)
- Why is our idea better?

Technical Activities

- Define simple Proof of Concept (POC) experiment
- Perform POC experiment
- Data Analysis
- Iterate POC experiment
- Draft scale up ideas

Initiate technical trials for POC

Initiate scale up discussion with Harper

- Industry Need
- Potential Customers
- Assuming complete success, what is the potential market?
- Anyone else doing it?

Commercial Activities

- Preliminary cost calculation
- Preliminary market size est.
- List potential customers
- Sketch business opportunity
- Define inventors IP

Best Done by Inventors

Stage 1 - Prospecting

Stage 2 – Initial Testing

Stage 3 – Applied Research 1

Gate I

Estimate Budget: \$ & Time

- Idea Generation
- Thought Experiments
- Confirm Thermodynamics
- Confirm Novelty (IP)
- Why is our idea better?

Technical Activities

Initiate technical trials for POC

- Industry Need
- Potential Customers
- Assuming complete success, what is the potential market?
- Anyone else doing it?

Commercial Activities

Best Done by Inventors

Estimate Budget:\$ &Time

- Define simple Proof of Concept (POC) experiment
- Perform POC experiment
- Data Analysis
- Iterate POC experiment
- Draft scale up ideas

- Preliminary cost calculation
- Preliminary market size est.
- List potential customers
- Sketch business opportunity
- Define inventors IP

Gate II

Initiate scale up discussion with Harper

Gate III

Estimate Budget: \$ & Time

- Grams → Kilograms
- Engage with Harper
- Identify OSHA concerns
- Design scale up #1-larger batch
- Schedule interaction test 1

- Refine market analysis
- Confirm supply chain
- Refine customer list
- Define path to market
- Preliminary interest level customers
- Outline inventors IP

Initiate scale up G to Kg

Collaboration with Harper

Stage 4 – Applied Research 2

Stage 5 – Trial Manufacturing

Estimate Budget: \$ & Time

- Perform tests at Harper
- Evaluate results
- Iterate tests as needed
- Confirm results/vs. objectives
- Evaluate product quality
- Design potential scale up
- Obtain input for process improvement from Harper

Technical Activities

Gate IV

Proceed to trial manuf.

Estimate Budget: \$ & Time

- Perform trials in equipment similar to commercial
- Product evaluation
- Iteration to optimize product quality
- Establish a manufacturing plan
- Define feed→process→product
- Define safety requirements and procedures

Commercial Activities

- NDA with selected customers
- Provide samples to customer
- Preliminary price/cost estimates
- Business concept
- Define final scale up trials and process
- Identify possible business partners
- Define IP ownership for Harper's process improvement ideas

- Larger scale evaluation at potential customers
- Define pathways to market
- Refined price/cost analysis
- Preliminary financial analysis
- Identify source for capital

Commercial Scale Unit

Collaboration with Harper

Thank you for your time!



Visit us at harperintl.com