Look at Thermal Process Scale-Up Requirements Early When

Commercializing a New Material

Tom Mroz
Harper International
Lancaster, N.Y.

The success of process development can often be influenced by results and decisions made very early in the evaluation process.

A common question by materials developers is: "When is the right time during materials development projects to consider commercial scale issues?" Is it as early as initial conceptual experiments? Is it during initial scale-up of promising results? Is it during process refinement and capability determination efforts? Or is it when market potential confirms the need to begin continuous manufacturing? As the market for new materials development expands, and time-to-market can make the difference between success and failure, the answer is all of the above.

The concept of planning for success is not new, but often in materials process development projects, the application is delayed, or forgotten altogether. To neglect this lesson can lead to delays in project completion, excess costs associated with late process changes, and even elimination of a candidate process after significant investment. Even if a project successfully launches, late adoption of scaling considerations can require significant reengineering of the process, or missed opportunities for product quality, cost, and throughput.

Consider the case of early conceptual experiments. One might start down the path of curiosity, seeking to determine if a particular set of raw materials, process conditions, or energy inputs can lead to a particular result. Assuming the experiment is not theoretical in nature, but rather directed toward an eventual product offering, it is necessary even at these early stages to consider variables that might be affected by scaling. If commercialization is the goal, even the earliest experiments can be screened based on their potential for scalability. For instance, considerations regarding raw material cost, quality, physical characteristics, and other variables that are often neglected at this stage can play an important part in project success in later stages. Additionally, in cases where solid-gas exchange is required for the process, such as in reduction processes to form refractory metals, consideration for maximizing the interface between the solid and gas phases, as well as appropriate removal of reaction products to maximize reaction kinetics and minimize potential for back reactions or contamination, must also be provided.

Involvement with key vendors at an early stage can be a critical factor. The focus at Harper is on thermal process solutions for commercial materials processing applications. Historically, the company's involvement with the customer often occurs well into their development process, where many variables have already been established. In some cases, those decisions limit opportunities for designing the most effective thermal processing tool for the application. This can mean missed opportunities for cost savings, product quality, and throughput. In the most extreme cases, it can make the difference between success and failure for the entire project.

Designing the most effective thermal processing solution for production scale applications must include early consideration for energy efficiency, product quality, and throughput.

Scaling thermal processes is rarely a simple matter of linear extrapolation. At experimental rates, the conversion rate of many solid-solid and solid-gas reactions is primarily a function of the size of the reactants, and the level of mixing. As the reaction is scaled to larger sizes, the ability to heat or cool the mass of material and the ability to introduce or remove gasses from the solids plays an increasingly important role in reaction efficiency, often to the point of controlling the conversion rate.

Fig. 1 — *Continuous carbon fiber processing in a series of thermal steps; the process is engineered to apply heating and cooling to the most minimal load possible without need for material containment.*

Careful consideration of raw material forms and process steps can have a big impact on the range of processing options available for later thermal processing methods.

The most efficient thermal processes are engineered to apply heating and cooling to the most minimal load possible. In a perfect situation, this means processing the reactants in a continuous manner without need for material containment. In a nearly perfect example of such a process, carbon fiber is produced by drawing the material continuously and directly through various thermal process steps. Energy inputs are required only to heat the fiber and the process cover gas as it enters each thermal step. No secondary containers are required; the fiber supports itself throughout the process (Fig. 1).

For bulk particulate materials, other means of manipulating the material in the thermal step are required. Options such as rotary furnaces, fluidized beds, and vertical furnaces provide efficient processing options. In each case, it is necessary to consider the form of the reactants so they not only provide reaction kinetics, but also that their physical form is compatible with the process method.

Ignoring consideration of continuous processing until the basic process and materials are fully established can make converting the process to suit desired equipment long and expensive. Earlier consideration and even testing of commercially relevant process methods can minimize scale-up costs. This provides better control over the project schedule, better understanding of costs and facility requirements for the commercial process, and a quicker path to producing process-relevant samples for internal assessment and customer sampling.

Harper's Technology Center offers clients the opportunity to work with specialists using commercially relevant processing tools under production-scale conditions to test a process. This could involve initial feasibility testing of a concept, scaling of a concept to a continuous operation, or engineering studies to determine the most suitable manner in which to efficiently conduct a specific thermal process. Later in the development process, it might also include evaluation of pilot scale operations, preparation of test materials for sampling, or construction of pilot equipment for a customer to use at its own production floor (Fig. 2).

The benefits of using commercially relevant processing tools include addressing immediate experimental goals and providing an opportunity to access equipment design and scaling. Availability of experimental data from laboratory testing provides a basis to design full-scale equipment that closely matches customer requirements. Risks associated with under-engineered solutions or high costs are minimized by the ability to use actual process data to design a thermal system.

Examples of carefully planned scale-up

Production of uniform, fine molybdenum powder. Molybdenum reduction from the oxide to metal occurs in several steps, where the initial reduction from trioxide to dioxide is exothermic. The ability to obtain the proper particle morphology requires precise temperature control in the first step, and careful atmosphere control in the second step. A two-step thermal process involving the use of a rotary furnace followed by a pusher furnace resulted in production of a superior product. Significant experimental effort at the laboratory scale was necessary to identify the proper process conditions, and allowed for scaling to a successful production process. A full report on this process development can be found at www.harperintl.com/resources.

Production of high purity activated carbon for supercapacitors. A major university client previously prepared laboratory-scale samples of a suitable carbon material that tested successfully for use in supercapacitor applications. Harper performed process optimization and scaling work using a variety of techniques to ensure atmosphere integrity and product purity. Ultimately, a multistep rotary furnace process was engineered and built to achieve the production throughput goal, while meeting product specifications. By separating the process into several rotary furnaces, different materials of construction could be used in each furnace to provide the most suitable and efficient environment for each step.

Production of uniform, fine tungsten powder. Reduction of tungsten oxide to metal in a hydrogen environment theoretically is a straightforward thermal process. However, precise control of reaction parameters and environmental conditions are required to obtain a product that has a controlled particle size. The reduction process creates significant quantities of water vapor that change the chemical nature of the reaction environment. Tungsten vapor pressure increases in this environment, and can lead to rapid coarsening of the product. To minimizing coarsening, the process and associated equipment must be designed to remove the water vapor product and maintain a controlled dew point above the product at certain points in the reaction process. Simple experimental methods might provide this condition by maximizing gas flows to dilute the water vapor to acceptable levels.

However, for commercial production, diluting gas flows become prohibitively expensive. Alternative methods, involving furnace vent designs and careful gas flow patterns over a continuous reaction bed with controlled geometry has been demonstrated to provide the desired result in an economical fashion.

Conclusions

The success of process development can often be influenced by results and decisions made very early in the evaluation process. These decisions may later preclude certain equipment or process options that would otherwise make the difference in the quality, throughput, or efficiencies of the commercial scale product. The opportunity to evaluate new processes using commercially relevant equipment can assist in ensuring that raw material selection; initial processing steps; and assumptions regarding utilities consumption, product quality, and process yield will meet market requirements. Additional opportunities including obtaining equipment sizing data, matching process to equipment for maximized efficiency, and producing sufficient material for customer testing are realized when testing is performed in conjunction with the commercial scale equipment manufacturer. Ultimately, such a working arrangement maximizes the opportunity for success, often in a reduced timeframe. ❍

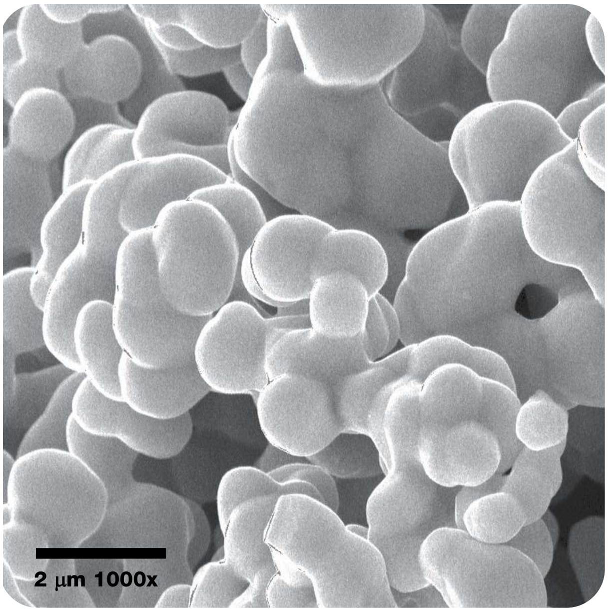

Fig. 2 — *Testing and sampling of materials is critical, especially for those requiring high levels of uniformity and purity.*

For more information: Diana Robbins, Marketing Manager, Harper International,11 W. Drullard Ave., Lancaster, NY 14086; tel: 716/684-7400, ext. 1116; fax: 716/684-7405; email: drobbins@harperintl.com; www.harperintl.com.