

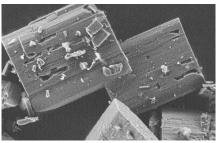
Novel process allows easy and precise control of nanoparticle structure and size distribution through real-time adjustments to reaction pressure, temperature, and other factors.

Argonne National Laboratory has a patented hydrothermal synthesis process that, for the first time, enables the scalable manufacturing of highly valuable nanoparticles, including thermochromic VO<sub>2</sub> for smarter windows. Argonne has also developed a novel, supercritical hydro/solvothermal synthesis process to produce single crystal materials for energy storage.

Solution-phase based hydrothermal synthesis uses a continuous flow micro-reactor—it's tabletop size and safer to use than traditional batch processing, because it does not require high temperatures or pressures for a large volume of reactant system over an extended period to attain optimal chemical reactions. The process enables adjustments to reaction pressure, temperature, residence time, reactant concentration, solvent type, and other factors to exactly control particle structure and size distribution.

The process cuts reaction time from two to three days down to minutes while yielding nanoparticles of uniform size. With output increases made possible by networking multiple microfluidic reactors together, the process can produce scalable volumes of  $VO_2$  particles and single crystal materials at kilo per day rates.

Argonne invites industry to de-risk materials scale up at the U.S. Department of Energy's leading collection of materials scale-up laboratories, the Materials Engineering Research Facility—home to the DOE's first pre-pilot scale supercritical hydro/solvothermal synthesis system.



Single crystal nickel-rich NMC811 battery material produced at supercritical hydro/ solvothermal reaction facility.

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#### **CASE STUDIES**

## **Smarter Windows**

Commercial production of VO<sub>2</sub> opens the door to widespread adoption of smart windows that use thermochromics technology — infrared energy automatically passes through windows in the winter for warmth and is blocked in the summer for cooling, while visible light always passes through.

Until now, smart window users have had to choose between expensive electrochromic windows, or low efficiency VO<sub>2</sub>-based thermochromic windows made in small, variable quality batch process. Argonne is advancing hydrothermal synthesis to reduce the VO<sub>2</sub> particle size from 100 nanometers to 50 nanometers or smaller to increase transparency and solar energy modulation efficiency of the composite smart film.

The new generation of low-haze windows can:

- Control more than twice the solar energy for high and low temperatures
- Switch from blocking to passing through infrared light in micro-or nano-seconds
- Block heat at 77 degrees
   Fahrenheit, or a specific
   temperature, instead of 154 degrees
   Fahrenheit
- ☐ Enhance energy efficiency without tinting

#### **Better Batteries**

Single crystal materials, in which the crystal lattice is continuous to the edges without grain boundaries, possess unique mechanical, optical and electrochemical properties.

Single crystal battery materials, with their lower surface area and exceptional material integrity, offer superior cycling performance and life compared to materials composed of many crystals.

Supercritical hydro/solvothermal synthesis enables manufacturers to affordably produce large volumes of highly crystalline single crystals — from the 200 nanometers ideal for nanomaterials up to 20 micrometers used in battery materials — with grain boundaries and particle crack formation eliminated.

Argonne scientists have used the technology to control the particle size of lithium cobalt oxide from 300 nanometers to 1 micrometer, to increase charge and discharge rates, to produce the first commercial-scale quantities of the battery material lithium iron phosphate.

Battery materials made with these single crystals have:

- □ Increased cycle life
- $\hfill\Box$  Improved performance
- □ Fewer defects
- □ Optimized catalytic activity

# AN ECONOMICAL, GREEN OPTION

Supercritical hydro/solvothermal synthesis does more than efficiently produce uniform, high-quality material. A green chemistry that uses water and pressure for extraction instead of organic solvents, along with heat recovery ability and a shorter processing time, means cost savings and less environmental impact.

#### **OTHER APPLICATIONS**

- ☐ Cathode battery material
- Nickel-rich cathode battery material
- Cobalt-free high energy lithiumand manganese-rich battery material
- ☐ Silicon nanoparticles and composites
- □ Battery electrolyte recycling
- □ Battery cathode recycling
- Supercritical carbon dioxide extraction
- ☐ Supercritical water oxidation



Multi-purpose Supercritical Hydro/Solvothermal Reaction Facility. The pre-pilot scale facility can produce approximately 100 grams of single crystal product per day in batch or continuous operation mode.