## surface area.

In the two-step process developed in this study, tin fluoride was first dissolved in water at 50°C for 20 min. and the solution was then left undisturbed at 28°C for three days. Particles precipitated and settled at the bottom, and were collected and dried at 60°C for 12 h.

Structural characterization using xray diffraction and transmission elec-

## **Energy Focus**

Modified SMP allows high resolution mapping of lithium-ion diffusion

ithium-ion batteries are one of the most ubiquitous power sources for portable electronics, but the design of new batteries is currently limited by a poor understanding of the nanoscale mechanisms of lithium ion transport and how such mechanisms are affected by microstructure and defects. In the August 29th online edition of Nature Materials (DOI: 10.1038/nnano.2010.174), N. Balke of Oak Ridge National Laboratory (ORNL), A.N. Morozovska of the National Academy of Science of Ukraine, D.W. Chung of Purdue University, and their colleagues report on a novel method of mapping ion diffusion in LiCoO<sub>2</sub> battery electrodes.

Using a modified scanning probe microscope (SPM) tip, the researchers applied a periodic electric field to induce the motion of lithium ions. The flux of ions into or out of the electrode is actron microscopy revealed that the precipitates were nanosheets composed of a mixture of  $\text{SnO}_2$  main phase and SnO additional phase. Brunauer– Emmett–Teller (BET) surface area measured from nitrogen isotherms revealed a value of 194 m<sup>2</sup>/g, more than double the highest value reported in previous studies whereas pore sizes were measured at 1–2 nm.

companied by local structural changes that are detected by the deflection of the same tip. A combination of high frequency imaging and low frequency spectroscopy allows mapping of local ionic dynamics with nanoscale precision. The researchers estimate that this method offers a strain detection limit 6–8 orders of magnitude better than classical electrochemical techniques.

The team first applied a constant voltage to the SPM tip, causing a change in lithium ion concentration and an associated strain of the electrode. They observed changes in the electrode's morphology after biasing, as well as diffusion of ions across (001) planes.

They then applied a high-frequency periodic voltage, inducing changes in the resonance frequency resulting from cathode structure changes. The team mapped variations in Young's modulus and related them to fluctuations in lithium diffusion and intercalation behavior.

In their final experiment they varied both biasing pulse frequency and amplitude, allowing them to measure ion The nanocrystalline sheets with large specific surface area produced in this technique are expected to be better suited for use in sensors. The researchers believe that this new fabrication method provides additional benefits as an eco-friendly alternative with reduced production costs through lower energy consumption and CO, emission.

Kaushik Chatterjee

diffusion across large time scales in battery-like conditions with <100 nm spatial resolution.

The use of the band excitation technology previously developed at ORNL has allowed the team to separate the electrochemical strain signal from the variations in surface topography that plague conventional SPM measurements. Dynamic hysteresis loop mapping has also helped the team to probe changes in lithium ion dynamics across a sample's surface and enhance its response at grain boundaries.

According to the researchers, this is the first time electrochemical reactivity has been observed on the level of a single structural defect. The researchers said that this novel combination of temporal and spatial resolution will help them study the local factors controlling electrochemical reactions, ultimately permitting the design of more efficient and reliable batteries.

**Steven Spurgeon** 



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