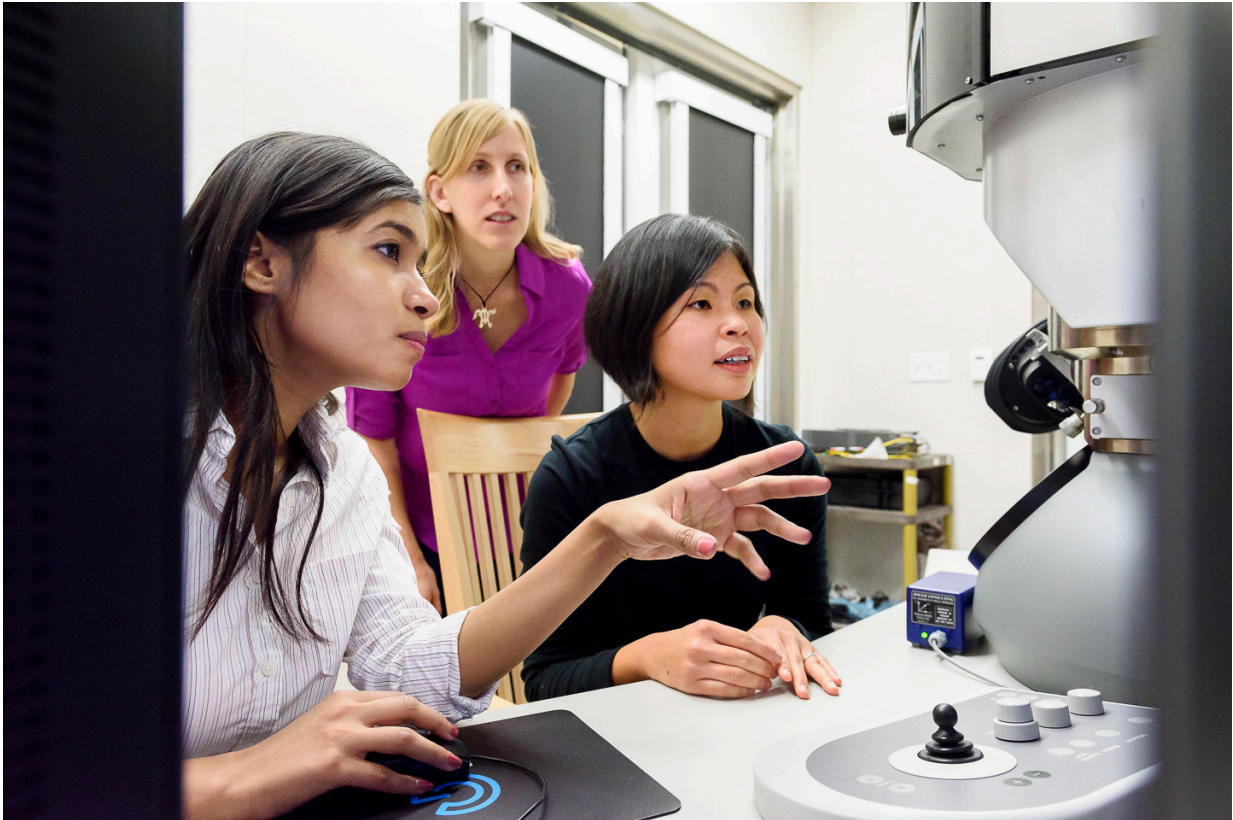


# Nanoscale view of energy storage

January 16 2017

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Using advanced electron microscope techniques, (left to right) graduate student Fariah Hayee, Professor Jen Dionne and senior research scientist Ai Leen Koh captured extremely high-resolution video of atoms moving in and out of nanoparticles. Credit: L.A. Cicero

In a lab 18 feet below the Engineering Quad of Stanford University, researchers in the Dionne lab camped out with one of the most advanced

microscopes in the world to capture an unimaginably small reaction.

The lab members conducted arduous experiments - sometimes requiring a continuous 30 hours of work—to capture [real-time](#), dynamic visualizations of atoms that could someday help our phone batteries last longer and our electric vehicles go farther on a single charge.

Toiling underground in the tunneled labs, they recorded atoms moving in and out of nanoparticles less than 100 nanometers in size, with a resolution approaching 1 nanometer.

"The ability to directly visualize reactions in real time with such high resolution will allow us to explore many unanswered questions in the chemical and physical sciences," said Jen Dionne, associate professor of materials science and engineering at Stanford and senior author of the paper detailing this work, published Jan. 16 in *Nature Communications*. "While the experiments are not easy, they would not be possible without the remarkable advances in [electron microscopy](#) from the past decade."

Their experiments focused on hydrogen moving into palladium, a class of reactions known as an intercalation-driven phase transition. This reaction is physically analogous to how ions flow through a battery or fuel cell during charging and discharging. Observing this process in real time provides insight into why nanoparticles make better electrodes than bulk materials and fits into Dionne's larger interest in [energy storage devices](#) that can charge faster, hold more energy and stave off permanent failure.

## Technical complexity and ghosts

For these experiments, the Dionne lab created palladium nanocubes, a form of nanoparticle, that ranged in size from about 15 to 80 nanometers, and then placed them in a hydrogen gas environment within

an electron microscope. The researchers knew that hydrogen would change both the dimensions of the lattice and the electronic properties of the nanoparticle. They thought that, with the appropriate microscope lens and aperture configuration, techniques called scanning [transmission electron microscopy](#) and electron energy loss spectroscopy might show hydrogen uptake in real time.

After months of trial and error, the results were extremely detailed, real-time videos of the changes in the particle as hydrogen was introduced. The entire process was so complicated and novel that the first time it worked, the lab didn't even have the video software running, leading them to capture their first movie success on a smartphone.

Following these videos, they examined the nanocubes during intermediate stages of hydrogenation using a second technique in the microscope, called dark-field imaging, which relies on scattered electrons. In order to pause the hydrogenation process, the researchers plunged the nanocubes into an ice bath of liquid nitrogen mid-reaction, dropping their temperature to 100 degrees Kelvin (-280 F). These dark-field images served as a way to check that the application of the electron beam hadn't influenced the previous observations and allowed the researchers to see detailed structural changes during the reaction.

"With the average experiment spanning about 24 hours at this low temperature, we faced many instrument problems and called Ai Leen Koh [co-author and research scientist at Stanford's Nano Shared Facilities] at the weirdest hours of the night," recalled Fariah Hayee, lead co-author of the study and graduate student in the Dionne lab. "We even encountered a 'ghost-of-the-joystick problem,' where the joystick seemed to move the sample uncontrollably for some time."

While most electron microscopes operate with the specimen held in a vacuum, the microscope used for this research has the advanced ability

to allow the researchers to introduce liquids or gases to their specimen.

"We benefit tremendously from having access to one of the best microscope facilities in the world," said Tarun Narayan, lead co-author of this study and recent doctoral graduate from the Dionne lab. "Without these specific tools, we wouldn't be able to introduce hydrogen gas or cool down our samples enough to see these processes take place."

## **Pushing out imperfections**

Aside from being a widely applicable proof of concept for this suite of visualization techniques, watching the atoms move provides greater validation for the high hopes many scientists have for nanoparticle energy storage technologies.

The researchers saw the atoms move in through the corners of the nanocube and observed the formation of various imperfections within the particle as hydrogen moved within it. This sounds like an argument against the promise of nanoparticles but that's because it's not the whole story.

"The nanoparticle has the ability to self-heal," said Dionne. "When you first introduce hydrogen, the particle deforms and loses its perfect crystallinity. But once the particle has absorbed as much hydrogen as it can, it transforms itself back to a perfect crystal again."

The researchers describe this as imperfections being "pushed out" of the nanoparticle. This ability of the nanocube to self-heal makes it more durable, a key property needed for energy storage materials that can sustain many charge and discharge cycles.

## **Looking toward the future**

As the efficiency of renewable energy generation increases, the need for higher quality [energy storage](#) is more pressing than ever. It's likely that the future of storage will rely on new chemistries and the findings of this research, including the microscopy techniques the researchers refined along the way, will apply to nearly any solution in those categories.

For its part, the Dionne lab has many directions it can go from here. The team could look at a variety of material compositions, or compare how the sizes and shapes of nanoparticles affect the way they work, and, soon, take advantage of new upgrades to their microscope to study light-driven reactions. At present, Hayee has moved on to experimenting with nanorods, which have more surface area for the ions to move through, promising potentially even faster kinetics.

**More information:** Tarun C. Narayan et al. Direct visualization of hydrogen absorption dynamics in individual palladium nanoparticles, *Nature Communications* (2017). DOI: 10.1038/ncomms14020 , [www.nature.com/articles/ncomms14020](http://www.nature.com/articles/ncomms14020)

Provided by Stanford University

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