Researcher's cryo-imaging continues to drive quantum discoveries

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Lena Kourkoutis, M.S. '06, Ph.D. '09 and Judy Cha, Ph.D. '09, pictured in 2023, were in the first cohort of doctoral students in the research lab of David Muller in 2003. Two years ago, they finally had a chance to collaborate, but just as the project was beginning, Kourkoutis died. She was 44. Credit: Cornell University

It wasn't nostalgia that brought Judy Cha, Ph.D. '09, back to Cornell. Among the reasons she left Yale University and joined the Department of Materials Science and Engineering two years ago was the prospect of working with an old friend: Lena Kourkoutis, M.S. '06, Ph.D. '09.

In 2003, they were in the first cohort of doctoral students in the research lab of David Muller, the Samuel B. Eckert Professor of Engineering in Cornell Engineering, and Cha and Kourkoutis shared an office for six years. Now they were both established academics with research labs of their own.

Kourkoutis had pioneered a form of scanning transmission electron microscopy (STEM) that could be conducted in situ at cryogenic temperatures, and Cha looked forward to using the imaging technique for her work, exploring novel electronic properties and phase transformations of quantum nanomaterials.

"I was so excited to work with Lena," Cha said. "It was like, finally, we are both now established, our identity is secure, let's work together."

But just as their collaboration was beginning, it was over. Kourkoutis died in June 2023 at the age of 44.

However, their brief collaboration generated enough findings for a series of papers, the latest of which was published August 6 in *Proceedings of the National Academy of Sciences*.

"I consider it her parting gift for me," Cha said. "I hope that she's proud of the work. I truly mean that."

Cornell Engineering recently held a memorial symposium that honored Kourkoutis and the impact of her scientific research, professional service and community leadership. Approximately a hundred mentees, collaborators and colleagues gathered for the weekend-long event.

'She debugged everything'

Cha's team set out to study the microstructure of tantalum disulfide (TaS_2), a nanomaterial that features a phenomenon known as charge density wave (CDW), in which atoms distort in a periodic order, changing the material's electronic properties. CDW has been known to transition TaS_2 from a conductor to an insulator. But when the material is thinned out, CDW disappears. Nobody knows why.

"Our original idea was to directly locate it inside the electron microscope as a function of thickness and try to see if we can add to this puzzle," Cha said.

To make CDW appear in tantalum disulfide, the material needs to be cooled to -190°C—liquid nitrogen temperature. That meant the researchers needed an electron microscope that could function at -200°C.

"Cornell is one of the few places in the world that has expertise in that," Cha said. "And this was pioneered by Lena."

Kourkoutis' cryo-imaging method has enabled scientists to study the structure of energy, quantum and even biological materials at picometer precision while varying the temperature. In recent years, this method has produced a wide range of discoveries, from revealing why lithium batteries were failing, to capturing the physical and chemical interactions that sequester carbon in soil, to exposing the tiny chemical flaws that lead to tooth decay.

"If you cannot control the temperature, you cannot repeat the experiment on the same sample. It's just a one-time observation," Cha said. "Lena made it so that we can control the temperature and still take amazing images. She debugged everything and solved all the technical challenges. Now it really allows you to do science."

Members of Cha's research group worked with Kourkoutis's team to learn their cryo-STEM techniques. Kourkoutis was also generous in letting the Cha group use her lab's experimental setup.

"I learned so much from Lena and her group through the course of this collaboration," said the paper's co-lead author James Hart, a former postdoctoral researcher who is now with the Naval Research Lab. "Her cryo-STEM expertise guided all of the experiments. Working with Lena was a true pleasure."

The imaging produced so much high-dimensional data, the researchers turned to Eun-Ah Kim, professor of physics in the College of Arts and Sciences, who used machine learning to sift through the team's terabyte-scale datasets to identify TaS_2's underlying behavior.

"Our machine-learning technique was developed for large volumes of X-ray diffraction data, but I always thought this approach is applicable to other types of probes," Kim said. "It was exciting to take a stab at cryo-STEM data as the first new experimental probe to be tested. We are enthralled that this first adventure was fruitful."

The researchers ultimately found that as TaS_2 is made thinner, its layers contain more and more stacking defects. The defects alter the CDW phase transition temperature by nearly 75 Kelvin, and the transition gets pinned at the specific defect site. This prevents conducting CDWs from transitioning into insulators.

By controlling these defects, the researchers anticipate being able to create potential memory devices.

On a larger scale, the findings demonstrate the important role that a material's microstructure—particularly defects—can play in affecting its properties. While introducing defects into metals is a common way to create a stronger, more ductile product (think of blacksmiths hammering away in their shop), for electronic and quantum materials, defects have been strenuously avoided. Cha hopes to change that.

"Traditionally, we always study perfect bulk crystals, as perfect as we can get them, and we do neutron or diffraction scattering or some sort of transport measurements that are a global property," she said. "This real-space information of microstructure is largely missing. We don't think about it. This is a ripe time to think about it, especially because we now have cryo-STEM that can go down to relevant temperatures."

Cha's group has several more papers in the pipeline with Kourkoutis listed as a co-author, as do a number of other engineering faculty.

"I think there are still many papers that will come out for the next at least two to three years, because science takes a long time," Cha said. "She was involved in many projects until about a month prior to her death. She was such a dedicated researcher, and her impact is far-reaching. I really want to pay homage to her."

More information: James L. Hart et al, Real-space visualization of a defect-mediated charge density wave transition, *Proceedings of the National Academy of Sciences* (2024). DOI: 10.1073/pnas.2402129121

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