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Making big leaps in understanding nanoscale gaps

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The QPress facility at CFN Credit: Brookhaven National Laboratory

Creating novel materials by combining layers with unique, beneficial properties seems like a fairly intuitive process—stack up the materials and stack up the benefits. This isn't always the case, however. Not every material will allow energy to travel through it the same way, making the benefits of one material come at the cost of another.

Using cutting-edge tools, scientists at the Center for Functional Nanomaterials (CFN), a U.S. Department of Energy (DOE) User Facility at Brookhaven National Laboratory, and the Institute of Experimental Physics at the University of Warsaw have created a new layered structure with 2D materials that exhibits a unique transfer of energy and charge. Understanding its material properties may lead to advancements in technologies such as solar cells and other optoelectronic devices. The results were published in the journal *Nano Letters*.

2D materials: Tiny, but mighty

Transition metal dichalcogenides (TMDs) are a class of materials structured like sandwiches with atomically thin layers. The meat of a TMD is a transition metal, which can form chemical bonds with electrons on their outermost orbit or shell, like most elements, as well as the next shell. That metal is sandwiched between two layers of chalcogens, a category of elements that contains oxygen, sulfur, and selenium.

Chalcogens all have six electrons in their outermost shell, which makes their chemical behavior similar. Each of these material layers is only one atom thick—one-millionth the thickness of a strand of human hair—leading them to be referred to as two-dimensional (2D) materials.

"At the atomic level, you get to see these unique and tunable electronic properties," said Abdullah Al-Mahboob, a Brookhaven staff scientist in the CFN Interface Science and Catalysis group. "TMDs are like a playground of physics. We're moving energy around from one material to the other at an atomic level."

Some new properties start to emerge from materials at this scale. Graphene, for example, is the 2D version of graphite, the material that most pencils are made of. In a Nobel Prize-winning experiment, scientists used a piece of adhesive tape to pull flakes off of graphite to study a layer of graphene. The researchers found the graphene to be incredibly strong at the atomic level—200 times stronger than steel relative to its weight. In addition, graphene is a great thermal and electrical conductor and has a unique light absorption spectrum. This unlocked the door to studying the 2D forms of other materials and their properties.

2D materials are interesting on their own, but when combined, surprising things start to happen. Each material has its own superpower—protecting materials from the environment, controlling the transfer of energy, absorbing light in different frequencies—and when scientists start to stack them together, they create what is known as a heterostructure. These heterostructures are capable of some extraordinary things and could one day be integrated into future technologies, like smaller electronic components and more advanced light detectors.

QPress: A first-of-its-kind experimental tool

While the exploration of these materials may have started with something as simple as a piece of adhesive tape, the tools used to extract, isolate, catalog, and build 2D materials have become quite advanced. At CFN, an entire system has been dedicated to the study of these heterostructures and the techniques used to create them—the Quantum Material Press (QPress).

"It's hard to compare the QPress to anything," said Suji Park, a Brookhaven staff scientist specializing in electronic materials. "It builds a structure layer by layer, like a 3D printer, but 2D heterostructures are built by an entirely different approach. The QPress creates material layers that are an atom or two thick, analyzes them, catalogs them, and finally assembles them. Robotics is used to systematically fabricate these ultrathin layers to create novel heterostructures."

The QPress has three custom built modules—the exfoliator, cataloger, and stacker. To create 2D layers, scientists use the exfoliator. Similar to the manual adhesive tape technique, the exfoliator has a mechanized roller assembly that exfoliates thin layers from larger source crystals with controls that provide the kind of precision that can't be achieved by hand.

Once collected and distributed, the source crystals are pressed onto a silicone oxide wafer and peeled off. They are then passed along to the cataloger, an automated microscope combing several optical characterization techniques. The cataloger uses machine learning (ML) to identify flakes of interest that are then cataloged into a database. Currently, ML is trained with only graphene data, but researchers will keep adding different kinds of 2D materials. Scientists can use this database to find the material flakes they need for their research.

When the necessary materials are available, scientists can use the stacker to fabricate heterostructures from them. Using high-precision robotics, they take the sample flakes and arrange them in the order needed, at any necessary angle, and transfer substrates to create the final heterostructure, which can be stored long-term in a sample library for later use.

The climate is controlled to ensure the quality of the samples and the fabrication process from exfoliation to building heterostructures is conducted in an inert gas environment in a glovebox. The exfoliated flakes and the stacked samples are stored in vacuum, in the sample libraries of the QPress cluster.

Additionally, electron beam evaporation, annealing, and oxygen plasma tools are available in the vacuum side of the cluster. Robotics are used to pass samples from one area of the QPress to the next. Once these novel heterostructures are fabricated though, what do they actually do and how do they do it?

After the team at CFN fabricated these fascinating new materials with the QPress, they integrated the materials with a suite of advanced microscopy and spectroscopy tools that enabled them to explore optoelectronic properties without exposing the samples to air, which would degrade material structures. Some of the delicate, exotic quantum properties on 2D materials need ultra-low cryo-temperatures to be detected, down to just a few kelvins. Otherwise, they get perturbed by the slightest amount of heat or any chemicals present in the air.

This platform will include advanced microscopes, X-ray spectrometers, and ultrafast lasers that are able to investigate the quantum world at cryo-temperatures.

Building better structures

Using the advanced capabilities of these resources, the team was able to get a more detailed picture of how long-distance energy transfer works in TMDs.

Energy wants to move across materials, the way a person wants to climb a ladder, but it needs a place to hold on to. Bandgaps can be thought of as the space between the rungs of a ladder. The larger the gap, the harder and slower it is to climb. If the gap is too large, it might not even be possible to finish moving up. Using materials that already have great conducting properties, this specialized team of scientists was able to stack them in a way that leveraged their structure to create pathways that transfer the charge more efficiently.

One of the TMDs the team created was molybdenum disulfide (MoS_2), which was shown in previous studies to have strong photoluminescence. Photoluminescence is the phenomenon that makes certain materials glow in the dark after they are exposed to light. When a material absorbs light with more energy than that energy bandgap, it can emit light with photon energy equal to the bandgap energy.

If a second material with an equal or lower energy bandgap gets closer to the first, as close as a sub-nanometer to few nanometers, energy can transfer nonradiatively from the first material to the second. The second material can then emit light with photon energy equal to its energy bandgap.

With an insulating interlayer made of hexagonal boron nitride (hBN), which prevents electronic conductivity, scientists observed an unusual kind of long-distance energy transfer between this TMD and one made of tungsten diselenide (WSe_2), which conducts electricity very efficiently. The energy transfer process occurred from the lower-to-higher bandgap materials, which is not typical in TMD heterostructures, where the transfer usually occurs from the higher-to-lower bandgap 2D materials.

The thickness of the interlayer played a big role, but also appeared to defy expectations. "We were surprised by the behavior of this material," said Al-Mahboob. "The interaction between the two layers increases along with the increase in distance up to a certain degree, and then it begins to decrease. Variables like spacing, temperature, and angle played an important role."

By gaining a better understanding of how these materials absorb and emit energy at this scale, scientists can apply these properties to new types of technologies and improve current ones. These could include solar cells that absorb light more effectively and hold a better charge, photosensors with higher accuracy, and electronic components that can be scaled down to even smaller sizes for more compact devices.

More information: Arka Karmakar et al, Excitation-Dependent High-Lying Excitonic Exchange via Interlayer Energy Transfer from Lower-to-Higher Bandgap 2D Material, *Nano Letters* (2023). DOI: 10.1021/acs.nanolett.3c01127

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