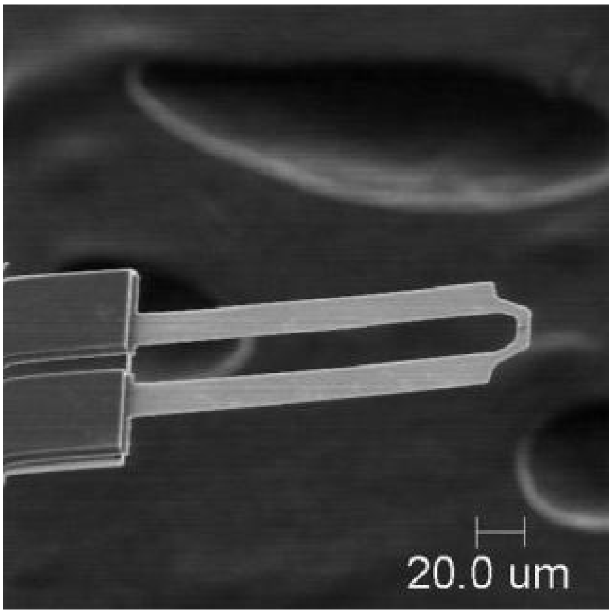
'World's smallest controlled heat source' studies explosives at the nanoscale

Georgia Tech Assistant Professor William King displays an experimental apparatus for nanoscale thermal analysis. Credit: Georgia Tech Photo: Gary Meek

Using nanometer scale analysis techniques and quantities too small to explode, researchers have mapped the temperature and length-sale factors that make energetic materials – otherwise known as explosives – behave the way they do.



Close-up of silicon heater-cantilever fabricated at Georgia Insitute of Technology. Image courtesy of William King.

Using the "world's smallest controlled heat source" – a tiny atomic-force microscope (AFM) cantilever – scientists from the Georgia Institute of Technology and Texas Tech University have developed a new way to study explosives that have nanometer-scale features. The technique provides new information about such phenomena as melting, evaporation and decomposition of explosives at the smallest length scales. Because the performance of these materials depends heavily on nanometer-scale factors such as crystal size and voids between crystals, the research could ultimately lead to safer explosives and better control over how they work.

Dubbed "nanodectonics," the research was described in the August 29 online issue of the American Chemical Society journal *Nano Letters*.

"Scientists would like to design energetic materials with specific responses, with a given temperature producing a given burn rate, for example," explained William King, an assistant professor in the Georgia

Institute of Technology's School of Mechanical Engineering. "Before our measurements, no one was able to interrogate these properties at the nanometer scale. With the data we have generated, it is possible to build physics-based models of how these materials behave rather than relying on empirical relationships seen at the macro scale."

Using an AFM tip capable of heating spots as small as a few nanometers in diameter, the researchers performed nanometer-scale thermal analysis on thin films of a polycrystalline energetic material known as Pentaerythritol Tetranitrate (PETN). They melted, evaporated and decomposed the PETN at length scales ranging from 100 nanometers to a few micrometers.

"We have shown that we can control the morphology of energetic materials on the nanoscale, and also measure nanoscale properties of these materials," said Brandon Weeks, an assistant professor in the Department of Chemical Engineering at Texas Tech University. "The hope is that since very small amounts of the material are needed for study, we can measure the properties in a very safe manner and extrapolate the information to bulk properties. Thus far, there has been very little research into the nanoscale properties of energetic materials outside of military applications."

For instance, voids between crystals of energetic materials are believed to play an important role in the rapid decomposition – or explosion – of the materials. When exposed to an initiation stimulus, these voids become "hot spots" and act as ignition sites that grow in temperature, size and pressure, leading to the detonation processes that make explosives useful in construction, mining and other commercial activities.

The formation of these voids is not directly controlled during materials synthesis. However, a better understanding of explosives at the nanoscale could lead to better control of the synthesis process – and better explosive materials, Weeks said.

"Ideally, we want to control the nanoscale properties of energetic materials to understand the physics at short length scales and make the materials safer," he added. "Perhaps we could engineer features into a material like PETN that would make it sensitive to a certain initiation stimulus. If the correct stimulus were not used, then the material would no longer behave like an explosive."

Experimentally, the researchers used their heated AFM cantilever to apply heat to a thin film of PETN. By varying the temperature as the cantilever was scanned across the film, the researchers were able to map the melting, evaporation and decomposition rates as a function of temperature, and observe their effects.

"By controlling the way we scan the tip over the surface, we can cause the material to re-condense into its solid form," King said. "When it re-condenses, it has fundamentally different crystalline structure. That gives us control over the crystal structure on the nanometer functional length scale."

The crystalline structure of energetic materials changes over time, and the researchers measured those changes during their study of the PETN films. For instance, the crystals become larger over time, which changes the materials properties and can make explosives less effective as they age.

PETN is a high explosive used in mining, construction and the defense industries, but because the researchers worked with such small quantities of it, there was no danger of an explosion in their lab. Weeks said the samples they studied were just a thousandth of the amount necessary to support an explosion. He estimated that the amount of material removed by the cantilever during the tests amounted to about 400 zeptograms. (A zeptogram is one-sextillionth of a gram).

The silicon-based cantilevers, which are fabricated in King's research group, include a built-in electrical resistance heater than can produce temperatures of up to 1,000 degrees Celsius. The temperature of the probe can be controlled to within approximately one degree Celsius.

Beyond energetic materials, the analytical technique made possible by the heated AFM cantilever could be used to study and improve other materials.

"We would expect other crystalline or polycrystalline materials to generally behave in a similar fashion, although the specifics would be unique for each material," King added. "We could use this same technique to study small-scale thermal properties of a whole suite of materials that we haven't been able to measure before. If we can get to know these materials at the nanometer scale, that would allow us to design them at larger scales."

Source: Georgia Institute of Technology