Nano Focus

Crystalline oxides sculpted at the nanoscale

From the hammer and chisel to three-dimensional (3D) printing, tools used for sculpting, building, and creating artwork have evolved to fit the medium in which they work. In science, just as in the art world, the tools of creation must be specifically designed for each purpose. Today, as electronics and modern technology become smaller and smaller, materials science demands more powerful tools for the manipulation of matter at the scale of single atoms.

Control over materials at the nanoscale has been demonstrated through techniques such as scanning tunneling microscopy and noncontact atomic force microscopy (NC-AFM); however, these techniques suffer from tedious processing times and are limited to interactions with just surface atoms. While they offer alternative pathways to fabricating small-scale structures, they fail to achieve the versatility and level of precision necessary for many modern devices.

In a recent issue of *Small* (DOI: 10.1002/smll.201502048), a group of researchers at Oak Ridge National Laboratory demonstrate a novel method of building 3D structures at the atomic level. Stephen Jesse and colleagues describe *in situ* arrangement of atomic-scale surface/subsurface crystalline oxide structures using scanning transmission electron microscopy (STEM). The researchers begin with a layer of oxide material (such as $SrTiO_3$), rendered amorphous by ion bombardment. This amorphous layer of material then facilitates the study.

The researchers found that they were able to induce an amorphous-to-crystalline phase transformation simply by using the high-energy electron probe in STEM. Starting from a seed area of crystalline material and drawing outward, the researchers were able to promote directed epitaxial crystallization (having the same crystallographic orientation as the seed crystal) of the amorphous material. Through control of the speed and direction of the electron beam, the researchers succeeded in selectively crystallizing areas of the material with a precision of up to 1–2 nm, effectively sculpting new crystalline material from the starting amorphous layer.

This new technique allows for a high degree of control in the fabrication and manipulation of arbitrarily shaped atomic-scale crystalline structures. “This method gives you a unique platform for experiments that would be very difficult to otherwise perform,” says Qian He, a co-author of the article. “If you want to compare a 2-nm-film with a 2-nm-plus-one unit cell film, then you have to grow a whole bunch of films, then study them independently. Here we can just grow a film *in situ* while controlling the thickness, then study [it], then grow a little bit more and study [it] again,” He says.

The researchers believe that more complex 3D structures are possible through control of the electron-beam focus depth accompanied by selective removal of the remaining amorphous material by targeted e-beam milling or wet chemical etching. The researchers have also demonstrated that this fabrication method may be automated through a custom-built electron-beam path control program to vastly increase fabrication throughput. Even more significant is the possibility for older generation electron beam instruments (e.g., scanning transmission electron microscopes and e-beam patterning systems) to be repurposed to employ this fabrication technique, making it accessible for more widespread use. Analogous to modern-day 3D printing, this innovative fabrication technique achieves precise yet versatile manipulation of materials at the atomic level.

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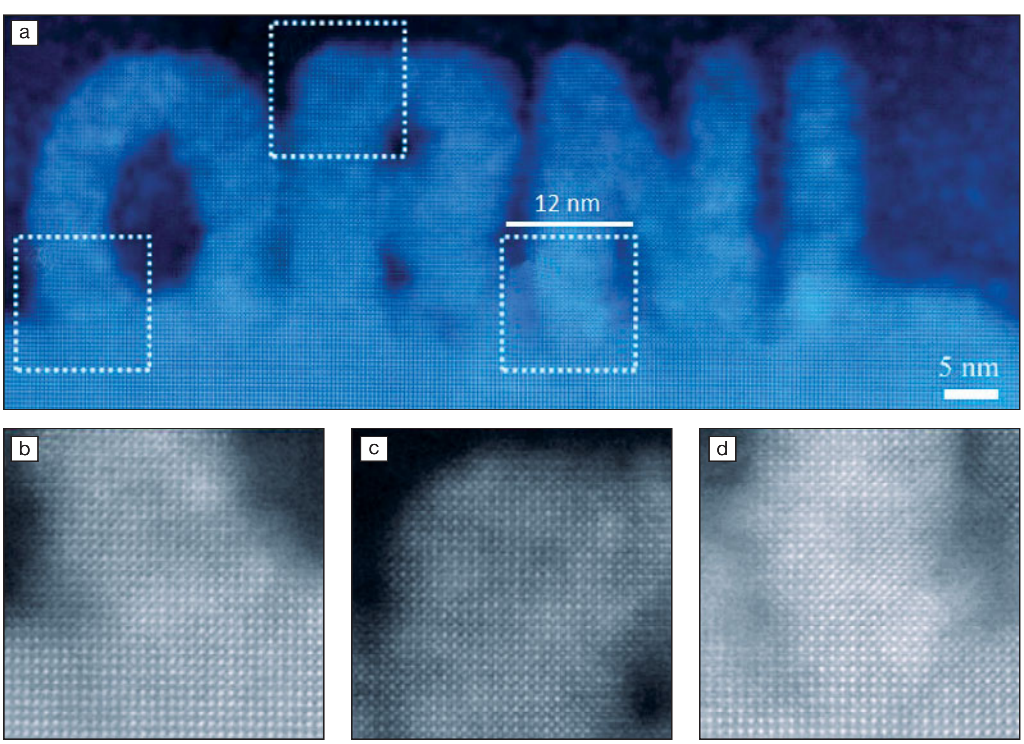

(a) Fourier–filtered high-angle annular dark-field images of crystalline regions of $SrTiO_3$. The square insets show magnified raw images of regions on the patterned letters. (b–d) In magnified images, note the same crystallographic orientation, which highlights the epitaxial character. Credit: *Small*/Wiley.

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