

Materials Researchers Pick Up the Pace of Discovery

BOSTON—Speed was a common theme among some 4400 scientists who gathered here from 1 to 5 December for the fall meeting of the Materials Research Society (MRS). Among the highlights: a rapid-fire approach for creating and testing hundreds of slightly different electronic devices simultaneously, a faster way to make high-temperature superconducting wires, and a quick approach to durable coatings.

Shotgun Chemistry Takes Aim at Devices

Combinatorial chemistry, the shotgun approach to synthesizing and testing hundreds or even thousands of slightly different compounds at one time, is already the rage in drug discovery. Over the last couple of years, the technique has also started to catch on in the materials science community as a way to speed the discovery of everything from new catalysts to polymers. At the MRS meeting, a team of researchers from Lawrence Berkeley National Laboratory in California reported taking the approach one step further by creating the first-ever combinatorial library of electronic devices, each made with a slightly different mix of materials that influenced its performance.

For their library, the team—led by Berkeley Lab postdoc Ichiro Takeuchi, physicist Xiao-Dong Xiang, and chemist Peter Schultz—made simple charge-storing devices known as dielectric capacitors. By keeping the configuration of each capacitor the same while varying the materials used to build it, the researchers were able to compare the electronic performance of all the devices in the library and identify the most promising combination for further study. The new work “is looking very good,” says Randolph Treece, a chemist and device expert at Superconducting Core Technologies in Golden, Colorado. “Technologists will probably see this as a good screening approach” to evaluate a wide array of possible electronic materials quickly.

Dielectric capacitors, which are the basis

of a type of computer memory known as dynamic random access memory (DRAM), consist of a sandwich of two flat electrodes separated by a layer of insulating material known as a dielectric. The capacitors for the current generation of DRAMs are made with silicon dioxide as the dielectric, but it is not the most efficient of insulators. To create the next generation of DRAMs, which will be smaller but will have to store the same amount of charge, researchers must find a material that is a better insulator than silicon dioxide. One front-runner is a ceramic compound called barium strontium titanate (BST). But although this material is more effective, BST devices that have been filled with charge commonly have trouble holding onto it. So researchers have been exploring a number of approaches to reducing this current leakage, including adding impurity atoms, or dopants, to the BST. It is the effect of dopants on the performance of BST capacitors that the Berkeley Lab team studied with their library.

To create the library, the researchers first laid down a heat-stable ceramic that served as the bottom electrode for all the devices on the chip. They then deposited the different BST materials on top, varying the concentrations so that the final capacitors were divided into three main groups, each having a different ratio of barium to strontium. Within each of these groups, they spiked the BST layer with varying concentrations of one of four different dopants, in the end producing a total of 240 different dielectric compounds. Next, they heated the entire chip to allow the component materials in each of the dielectric layers to mix fully; that step also caused the BST to

align its crystalline lattice with that of the underlying electrode, a key to improving device performance. Finally, the researchers deposited a layer of platinum atop each device to serve as the top electrode.

To test the devices, the researchers applied a voltage across each one while monitoring the effects. The differences were readily detectable. “Most of the dopants didn’t help,” says Takeuchi. But particular metal dopants—which the researchers decline to specify for proprietary reasons—“seemed to reduce the leakage current a lot,” as much as three orders of magnitude over other dopants. Down the road, Takeuchi says that the team hopes to improve the various metal-doped dielectrics further, as well as try out the combinatorial approach with transistors and other types of devices. If the strategy catches on, it could end up boosting the electronics industry’s already brisk pace of advances.

HTS Wires in a Hurry

Wires made from high-temperature superconductors (HTS) are great for carrying electrical current without resistance at the relatively accessible temperatures of liquid nitrogen. But the current-carrying ability of the most common HTS material, a ceramic made of bismuth, strontium, calcium, copper, and oxygen, plummets in strong magnetic fields, severely limiting its use in applications such as electric motors and magnetic resonance imaging. New results reported at the MRS meeting should help overcome this problem.

For several years researchers have investigated using another type of HTS ceramic, made from yttrium, barium, copper, and oxygen (YBCO), that is more resistant to magnetic fields. YBCO is brittle and difficult to form into wires, however, so researchers have been coating YBCO on flexible metal wires covered with an intermediate template layer, which is needed to control the growth of the YBCO (*Science*, 5 May 1995, p. 644). But the best scheme for producing the template layer is exceedingly slow, which has so far kept YBCO wires off the market.

Now there is new hope for YBCO wires. Physicist Robert Hammond reported that he and his colleagues at Stanford University were able to kick the templating method into high gear by replacing the normal slow-growing templating material known as yttria stabilized zirconia (YSZ) with fast-growing magnesium oxide. “It’s a major step forward that gives [YBCO]-coated conductors a fighting chance,” says Paul Grant, a superconductivity expert at the Electric Power Research Institute in Palo Alto, California.

Like other high-temperature superconductors, YBCO is a crystalline ceramic that grows as a myriad of tiny crystalline grains.



Quick study. Portion of the Berkeley Lab’s combinatorial device array. Each device is about 50 micrometers across.

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The orientation of these grains relative to one another controls how well electrical current passes through. Within each grain, YBCO is composed of layers of copper and oxygen atoms separated by layers of other elements. These layers are akin to stacked sheets of grid-lined graph paper. Because superconducting electrons travel most easily through the copper-oxygen sheets and along the grid lines, YBCO wire-makers have to align all the neighboring grains in the material in two directions. All the grains must lie flat, so that the copper-oxygen sheets in neighboring grains line up, and in addition, the grids of neighboring sheets should have more or less the same orientation to allow electrons to travel unimpeded in the same direction as they propagate down the wire.

If left to their own devices, YBCO grains neither lie flat nor align their grids. But in 1993, a team of Japanese researchers got YBCO grains to do both by growing them on top of a thick YSZ template. They first had to achieve a trick with the YSZ grains themselves, however. Normally, these grains also grow randomly, but the Japanese team found that bombarding the YSZ layer with a beam of argon ions while it was growing resulted in grains that lie flat and have a common orientation. When YBCO grains are grown on top, they follow YSZ's example. Yet despite further improvements over the years, "ion beam assisted deposition [IBAD] is still too time-consuming," taking hours for just short lengths of wire, says Hammond.

Hammond's team improved matters by switching to magnesium oxide for the template layer. The key is that grains of this material prefer to lie down flat when grown. While IBAD is still needed to orient the grids of the crystals, the job can be done in less time, because as Stephen Foltyn, a YBCO wire-maker at Los Alamos National Laboratory in New Mexico puts it, "with magnesium oxide, you've already got half the battle won." What's more, a much thinner layer of magnesium oxide seems to control the YBCO grain alignment, further helping to reduce the time needed for producing the template. Indeed, the Stanford team found that it could produce a template layer of magnesium oxide with the desired texture in a single minute, compared to the hours it takes to produce a YSZ template.

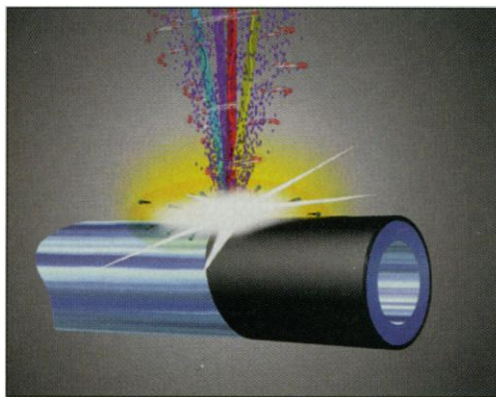
When Foltyn and his colleagues at Los Alamos produced short wires of YBCO grown on top of magnesium oxide IBAD templates, they found that the wires could conduct only about one-third the current that a YSZ/YBCO combination has achieved. But Hammond points out that YSZ/YBCO wires actually contain other thin layers above the YSZ, such as cerium oxide, which act to improve the wire's conductivity. And it's likely,

he believes, that a similar strategy could improve the magnesium oxide/YBCO wires as well. If so, IBAD may have a commercial future after all.

Laser Process Coated in Mystery

Hard coatings are vital for protecting everything from surgical blades and computer disc drives to the heads of golf clubs. But conventional coatings, painted or evaporated onto a surface, are often short-lived and eventually flake, chip, or peel away. In the last few years, however, a little-known company called QQC Inc. in Dearborn, Michigan, has come out of nowhere with a novel process that plays a set of lasers across steel and other materials to transform their outer layers into durable superhard coatings.

Nobody knows for sure how the process



Light work. Overlapping laser pulses vaporize atoms in making a superhard coating.

works, as the technology has preceded the science. But an independent researcher, materials scientist Rustum Roy of Pennsylvania State University in University Park, has now taken a close look at some of QQC's metal coatings using a variety of techniques including cross-sectional scanning electron microscopy (SEM) and x-ray diffraction. The laser process, Roy reported at the Boston meeting, rearranges the atoms of the metal itself into new, high-strength configurations, and can blend added coating material into the underlying metal, atom by atom, to form an alloy that is less likely to degrade. Roy concludes that the technique "is a major discovery."

Already, QQC has shown that the technique can harden not only metal, but also ceramics, and even plastic. "It's so generic, everyone and his brother will use it," says Roy. Prashant Kumta, a materials researcher at Carnegie Mellon University in Pittsburgh who led the MRS symposium that featured the new work, adds that unlike techniques for hardening a metal without applying a coating, which normally require

very high temperatures, the lasers can easily be targeted to transform only desired areas, with nanometer precision. "It's not possible to do this with another technique," says Kumta.

According to QQC co-founder Pravin Mistry, the new metal-transforming process works by overlapping the light pulses from at least three types of high-powered lasers—excimer, yttrium-aluminum-garnet, and carbon dioxide—each of which produces a different wavelength of light. When scanned across a steel surface, for example, these lasers vaporize a thin layer of material, creating an electrically charged, superheated plasma of iron atoms. When the beams pass on, the atoms rain down again onto the surface and then bond together. QQC can add other materials to the coating by sending a stream of particles across the surface of the steel. The lasers break apart the particles, adding their constituent atoms to the plasma, which rains down to form the new surface.

Roy analyzed the effect that this process had on a couple of different samples. The first was a standard fuel injector piece made from ferrosilicon, an alloy of silicon and iron, which QQC had coated with superhard titanium carbide. SEM and other techniques showed that the piece ended up with a top layer of graded titanium-carbide and ferrosilicon alloy, with two phases of steel underneath, both of which are harder than the conventional phase known as ferrite. First was a thin layer of martensite, and below this was a layer of amorphous steel. Creating just amorphous steel by itself, notes Roy, usually requires heating the part up to 1000 degrees Celsius and then cooling it instantly, a process known as quenching. "[Mistry] can quench it without quenching," says Roy.

Roy also looked at a hot forging punch, a cylindrical rod of coated steel used to stamp out wheel hubs for cars. QQC coats the steel rods with a layer of high-strength chromium cobalt alloy. In this sample Roy found that the laser transformation process creates a surface alloy of chromium cobalt and steel backed by a 2- to 3-millimeter-thick layer of martensite. The durability of the new coating, says Mistry, increases the industrial lifetime of the punches from a mere 1 hour of pounding out wheel hubs, to 12 days.

At present, the new coatings are not inexpensive. QQC has spent about \$15 million developing their laser setup. But Roy believes that if researchers can get a better picture of how the laser transformation method works, future versions of the setup may bring down the cost. Says Roy: "It's incredible what it's going to do."

—Robert F. Service