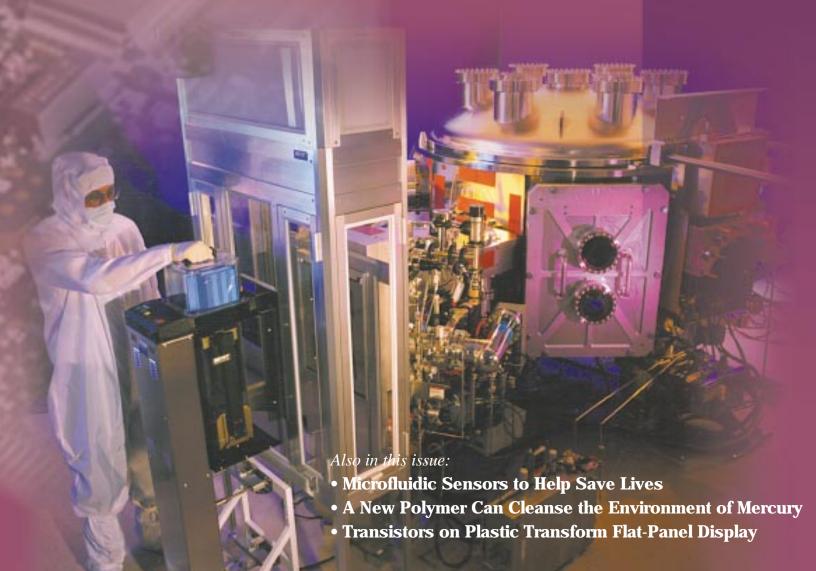
November 1999



U.S. Department of Energy's Lawrence Livermore National Laboratory



# **Imaging the Future** of Microcomputer Chips



#### About the Cover

Lawrence Livermore has joined Lawrence Berkeley and Sandia national laboratories and a consortium of leading U.S. semiconductor manufacturers to develop extreme ultraviolet lithography (EUVL) for making the next generation of high-capacity computer microchips. The article beginning on p. 4 is a progress report on the partnership's efforts. Pictured on the cover is a key component of EUVL's success—the Ultra Clean Ion Beam Sputter Deposition System. Developed at Livermore, it creates the precise, uniform, highly reflective, low-defect masks (or master patterns) used to "print" semiconductor circuits on next-generation silicon microchips.



#### **About the Review**

Lawrence Livermore National Laboratory is operated by the University of California for the Department of Energy. At Livermore, we focus science and technology on assuring our nation's security. We also apply that expertise to solve other important national problems in energy, bioscience, and the environment. *Science & Technology Review* is published 10 times a year to communicate, to a broad audience, the Laboratory's scientific and technological accomplishments in fulfilling its primary missions. The publication's goal is to help readers understand these accomplishments and appreciate their value to the individual citizen, the nation, and the world.

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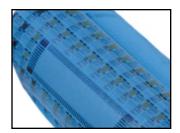
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#### Adaptive optics center funded

In late July, the National Science Foundation's governing body, the National Science Board, approved a proposal to establish a Center for Adaptive Optics at the University of California at Santa Cruz, with Lawrence Livermore as an important partner. The center will coordinate the work of researchers across the country involved in the rapidly developing field of adaptive optics, which has major applications in astronomy, vision science, and high-power laser beams. (See *S&TR*, July/August 1999, pp. 12–19.)

The Center for Adaptive Optics, which begins operation in November 1999, is one of five science and technology centers approved by the National Science Foundation this year. NSF program guidelines allow for financial commitments of up to \$20 million over five years for each center, with an option to renew for an additional five years.

UC Santa Cruz's 27 partner institutions in the center include Lawrence Livermore; the University of California at Berkeley, San Diego, Los Angeles, and Irvine; the University of Chicago; the California Institute of Technology; the University of Rochester; the University of Houston; Indiana University; and 17 other national laboratory, industry, and international partners.

Claire Max, director of the Laboratory's University Relations Program, said that Livermore is well positioned to play a big role in the collaboration. According to Max, "The Center for Adaptive Optics will provide the sustained effort needed to bring adaptive optics from promise to widespread use by astronomers and vision researchers."

Contact: Claire Max (925) 422-5442 (max1@llnl.gov).

#### Two DOE centers study CO<sub>2</sub> storage

As part of its global climate change research program, the Department of Energy has formed two centers to study the capture and long-term storage (sequestration) of atmospheric carbon dioxide (CO<sub>2</sub>) in terrestrial and oceanic ecosystems. The ultimate goal of these centers is to make carbon sequestration a potential component of future international efforts to reduce CO<sub>2</sub> buildup in the atmosphere, which is believed to contribute to global warming.

The DOE Center for Research on Ocean Carbon Sequestration (DOCS) will focus on oceanic ecosystems. It is a collaboration of numerous academic and oceanographic institutions led by Lawrence Livermore and Lawrence Berkeley national laboratories and will receive \$3 million over three years.

Livermore's Ken Caldeira and Berkeley's Jim Bishop are the center's codirectors. According to Caldeira, "The Lab and Berkeley bring complementary activities to the center. We have experience in modeling the oceanic carbon cycle and in simulation, and Berkeley has experience in observation and monitoring."

DOCS will research the feasibility, effectiveness, and environmental acceptability of ocean carbon sequestration.

Research will assess the environmental consequence of possibly increasing the amount of CO<sub>2</sub> absorbed by the ocean through CO<sub>2</sub> injection into the deep ocean and CO<sub>2</sub> fertilization of ocean organisms.

The Center for Research on Enhancing Carbon Sequestration in Terrestrial Ecosystems (CSITE) is also a diverse collaboration, led by Oak Ridge, Pacific Northwest, and Argonne national laboratories. It will receive \$6 million over three years.

CSITE will research ways to use plants, microbes, and soil management practices to cause more carbon to be stored below ground without major sacrifices in aboveground yields. It will also investigate lengthening the time carbon is sequestered in the soil as a means of limiting atmospheric concentrations. And it will study ways to measure, monitor, and verify sequestration so that national inventories of greenhouse gas emissions can be appropriately accounted for.

Contact: Ken Caldeira (925) 423-4191 (caldeira2@llnl.gov).

#### **Lab part of Next-Generation Internet**

In early August, the Department of Energy announced appropriation of \$15 million to finance 19 projects related to the emerging government-supported network called the Next-Generation Internet. The network will have the capability of carrying massive amounts of electronic, video, and voice signals at the speed of light—that is, about a thousand times faster than a standard Internet connection.

A localized version of the network already connects agencies in the Los Angeles and San Francisco area. The expanded network is expected to link a select group of agencies across the nation and around the globe.

Lawrence Livermore, Sandia/California and Lawrence Berkeley national laboratories and the Stanford Linear Accelerator Center are among the contributors to this Next Generation Internet project.

Bill Lennon, a program leader in Next Generation Internet research at Livermore, says that the increase in financial support will allow researchers to create software that can manage and secure data on the new network. "We have to customize the way that the data is sent, to do applications that have never been done before, . . . to identify the holes—the things that people haven't thought of," said Lennon.

Livermore is the lead in one Next Generation Internet project that will study weather change and predictability by conducting high-definition simulations of global weather patterns. The project will receive \$3.6 million in support per year for three years.

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## **Infinite Riches in a Little Space**

MAGINE owning a desktop computer as powerful as today's supercomputers. A computer in which each memory chip can hold tens of gigabytes of information. A computer that can recognize speech in context, hold an intelligent conversation, and translate languages. Now, imagine driving a car that can follow verbal commands, visualize the surrounding traffic environment, sound an alert upon encountering hazardous conditions, and provide high-resolution "heads-up" displays of local roadways and intersections.

Such possibilities seem as unreal today as the idea of a "personal" computer seemed 30 years ago. To a large degree, today's technological miracles—and tomorrow's technical dreams—are the result of the semiconductor industry's efforts to continually shrink the size of the basic elements (transistors, capacitors, resistors, etc.) that make up a computer chip. When chip features get smaller, speed, reliability, functionality, and efficiency all improve, while costs decrease.

For the past 30 years, optical lithography has been the process by which these features are printed on semiconductor substrates to produce integrated circuits. This optical process, however, is rapidly approaching its limits. The international semiconductor industry must decide—and soon—which next-generation lithographic technology it will embrace for manufacturing chips in the first two decades of the 21st century.

In 1997, three Department of Energy laboratories-Lawrence Livermore, Lawrence Berkeley, and Sandiajoined together in the Virtual National Laboratory (VNL) to develop one such promising technology: extreme ultraviolet lithography (EUVL). Each national laboratory contributes unique expertise to the program: Sandia brings system design and process development, Lawrence Berkeley brings precision metrology and patterning, and Lawrence Livermore brings expertise in complex multilayer reflective coatings, optics design, and precision engineering. To fully develop and help commercialize EUVL, the VNL is partnering with an industry consortium that includes some of the country's leading semiconductor manufacturers. The article beginning on p. 4 gives an overview of the VNL-EUVL program and Lawrence Livermore's contributions to it.

The VNL has been extremely successful to date. For instance, the three laboratories have garnered numerous R&D 100 awards as a direct result of technologies developed for EUVL. Even more to the point, a year ago, the international semiconductor community voted EUVL "the most promising technology" for printing future generations of computer chips with features as small as and smaller than 70 nanometers. (Current features are 180 nanometers.) The fact that EUVL technology is extendable is a major attribute.

Why all the interest in making something that's already so small even smaller?

For the VNL, helping to power the information age through the development of ever-smaller chips that make computers more compact yet more powerful furthers our nation's economic objectives. The VNL's efforts also support the national laboratories' missions in national security, proliferation prevention, energy, and environmental monitoring through advances in micromachining, sensor technology, precision measurement, and supercomputing.

To answer the question in a larger context, we need only look around us. Much of the modern world and its economic well-being are made possible by the computer chip. According to Department of Commerce data from 1998, the semiconductor industry is the United States' largest manufacturing industry in terms of value added, contributing 20 percent more to the economy than its nearest rival. As impressive as this figure is, it doesn't begin to explain the role semiconductors have played in enabling the revolution in today's electronics and information technologies.

The EUVL technology being developed by the VNL and its industry partners could take semiconductor manufacturing to the end of the "silicon cycle," sometime in the second decade of the 21st century. Then, it is predicted, the computer industry will have fully exploited silicon as a substrate material. It will have crammed as many features on a silicon chip as the material can support, and it will be time to dream again. Lawrence Livermore and the VNL hope to again play a significant part in making those new dreams come true.

■ James Glaze is Executive Director of the Virtual National Laboratory.



Livermore researchers
are part of a collaboration
between private industry
and DOE laboratories
dedicated to making extreme
ultraviolet lithography
the technology of choice
for manufacturing the
next generation of
microcomputer chips.

WENTY-FIVE years ago, the computing equivalent of today's laptop was a room full of computer hardware and a cartload of punch cards.

Since then, computers have become much more compact and increasingly powerful largely because of lithography, a basically photographic process that allows more and more features to be crammed onto a computer chip. Light is directed onto a mask—a sort of stencil of an integrated circuit pattern—and the image of that pattern is then projected onto a semiconductor wafer covered with light-sensitive photoresist. Creating circuits with smaller and smaller features has required using shorter and shorter wavelengths of light.

However, current lithography techniques have been pushed just about as far as they can go. They use light in the deep ultraviolet range—at about 248-nanometer wavelengths—to print 150- to 120-nanometer-size features on a chip. (A nanometer is a billionth of a meter.) In the next half dozen years, manufacturers plan to make chips with features measuring from 100 to 70 nanometers, using deep ultraviolet light of 193- and 157-nanometer wavelengths. Beyond that point, smaller features require wavelengths in the extreme ultraviolet (EUV) range. Light at these wavelengths is absorbed instead of transmitted by conventional lenses. The result: no light, no image, no circuit.

Semiconductor manufacturers are, therefore, at a critical juncture. Soon, they must decide which lithographic horse to back in the race to the next

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generation of microchip manufacturing. There are currently four possible alternatives: EUV, x-ray, electronbeam, and ion-beam lithography.

#### **Creating a Virtual Laboratory**

Two years ago, three Department of Energy national laboratories-Lawrence Livermore, Lawrence Berkeley, and Sandia/California formed the Virtual National Laboratory (VNL) to research and develop extreme ultraviolet lithography (EUVL) technology. The VNL is funded by the Extreme Ultraviolet Limited Liability Company—a consortium of Intel Corporation, Motorola Corporation, Advanced Micro Devices Corporation, and Micron Technology, Incorporatedin one of the largest cooperative research and development agreements within the Department of Energy. The threeyear, \$250-million venture is dedicated to developing the EUVL technology for commercial manufacturing of computer chips and to move this technology into production facilities in the first decade of the 21st century.

Each national laboratory brings unique contributions to this effort. Lawrence Livermore supplies its expertise in optics, precision engineering, and multilayer coatings. Sandia provides systems engineering, the photoactive polymer thin film exposed by the light, and the light source. Berkeley contributes its Advanced Light Source capability to generate EUV light to characterize optics and resists at the nanometer scale.

The VNL's lithography system uses mirrors to project the image of a reflective mask onto the photoresist-coated semiconductor wafer. Ultimately, this system will enable a microchip to be manufactured with etched circuit lines smaller than 100 nanometers in width, extendable to below 30 nanometers.

The resulting microprocessors would be a hundred times more powerful than those made today. Memory chips would be able to store a thousand times more information than at present.

"Lithography is generally viewed as the enabling technology for each new generation of semiconductor devices,"

Lawrence Livermore National Laboratory

says Don Sweeney, Lawrence Livermore's program manager for EUVL. "To put this technology into production facilities in 10 years, we need to show that the technology can work under real manufacturing conditions."

The VNL's current focus is on building and integrating the necessary

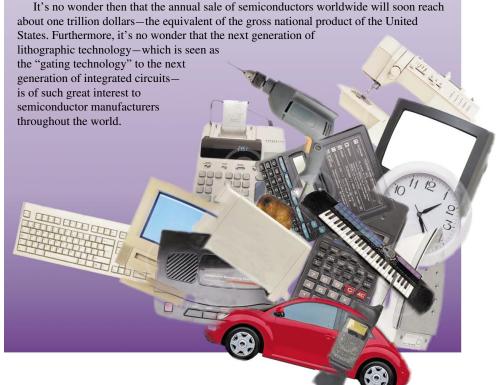
#### What's at Stake

... [T]he first 30 years of the integrated circuit had from two to five times the impact on the U.S. economy as the first 30 years of the railroad. Or, to put it another way, the transformation of the nineteenth century U.S. economy by the railroad took 60 years to achieve half the effect that microelectronics had over 30 years.

-Kenneth Flamm
"More for Less: The Economic Impact of Semiconductors"

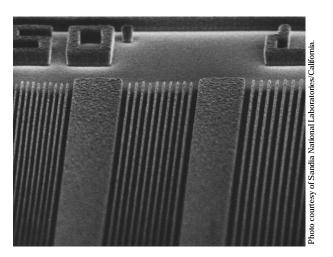
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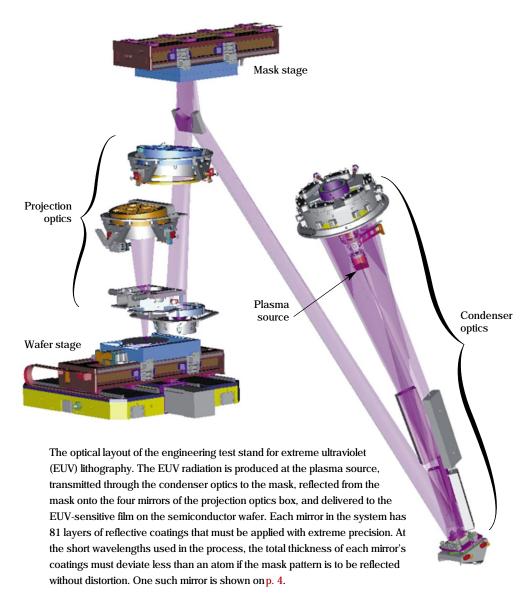
Semiconductors are ubiquitous in our lives. They are found in our cars, televisions, radios, telephones, stereos, personal computers, children's toys—even dishwashers and ovens. According to research conducted by the Semiconductor Industry Association and the World Bank, each person worldwide uses on the average about ten million transistors in a lifetime, with this consumption increasing 55 percent each year. By the year 2008, projections are that each person will consume a billion transistors.



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Using a prototype system, the Virtual National Laboratory has successfully printed lines as small as 50 nanometers (billionths of a meter) wide in photoresist. Current lithographic tools used in the semiconductor industry print patterns with 180-nanometer-size features.





technologies into an engineering test stand (ETS). Each national laboratory spearheads specific development areas for the ETS and for the systems beyond. Lawrence Livermore is leading the efforts to develop the optical systems and components, thin films, masks, and submicrometer metrology required for EUVL.

#### **Brighter Light Is Key**

The ETS (see the box on p. 7) includes a condenser optics box and a projection optics box. Both boxes house complex optical trains of precision concave and convex aspherical mirrors.

The main role of the condenser optics box is to bring light to the reflective pattern on the mask. "We want to bring as much light to the mask and, ultimately, the wafer, as possible," explains Sweeney. "The more light we deliver, the shorter the exposure time. It's like taking a picture with a camera. A picture taken in bright noonday sun requires a shorter exposure time than does a picture of the same scene taken at twilight."

For the semiconductor industry, brighter EUV images mean shorter exposure times, which translate to manufacturing more chips at a faster rate. The optics design team from Lawrence Livermore and Sandia designed a condenser optics system that collects and transports a significant fraction of the EUV light from the source to the reflective mask.

Once the image is reflected from the mask, it travels through the projection optics system. According to Sweeney, the projection optics box is the optical heart of the lithographic exposure system. "It is to the system what an engine is to a car," he explains. The four mirrors of the ETS projection optics system reduce the image and form it onto the wafer. "Again, imagine using a pocket camera. The camera lens transmits an image to the film, which—like the wafer—has a light-sensitive surface," says Sweeney.

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The optics teams are now working on advanced designs for the projection optics. They have a six-mirror design that promises to extend EUVL systems so that they can print features as small as 30 nanometers— a significant jump from the 70-nanometer limit of the ETS.

According to Sweeney, extendability to smaller features is an important requirement for whatever lithographic technology the semiconductor industry finally decides to back.

#### **Applying Uniform Thin Films**

Part of the success of the EUVL technology is due to the immense strides Lawrence Livermore has made in producing the highly reflective multilayers that are used on the ETS's optical mirrors as well as on the mask.

The projection and condenser optical systems require mirrors that reflect as much EUV light as possible. Manufacturing these mirrors has been a challenge because, in addition to being highly reflective, they must have surface coatings that are essentially perfectly uniform.

Lawrence Livermore and Lawrence Berkeley developed advanced multilayer coatings of molybdenum and silicon that can reflect nearly 70 percent of the EUV light at a wavelength of 13.4 nanometers. Applying these coatings evenly is a difficult task even when a mirror is flat, but EUVL mirrors are either convex or concave. Any small nonuniformity in the coatings destroys the shape of the optics and results in distorted patterns printed on the chips.

In the past year, the development of a new precision deposition system provided a major advance in applying these thin films to optics. (See *S&TR*, October 1999, p. 12.) This system, which won a 1999 R&D 100 Award, is so precise that 81 layers of molybdenum and silicon, each about 3.5 nanometers thick, can be deposited over a 150-millimeter area so that the total thickness over the surface deviates by less than an atom. The technique

can be used to coat mirrors as large as 40 centimeters in diameter.

#### The Mask-Making Challenge

Industry experts generally agree that the biggest challenges and risks for the next generation of lithography systems involve the mask—that is, the master pattern used to "print" the semiconductor circuits onto the silicon wafers or chips. The technology that successfully overcomes the hurdles of mask production has a good chance of becoming the preferred choice.

In EUVL, a mask is produced by applying multilayers of molybdenum and silicon to a flat substrate. The circuit pattern is produced by applying a final



The extreme ultraviolet lithography projection optics system in final assembly.

#### The Engineering Test Stand Provides a Prototype

The Virtual National Laboratory is developing, designing, and building a prototype extreme ultraviolet lithography (EUVL) system called the engineering test stand (ETS) at Sandia National Laboratories/California. The ETS uses laser-produced plasmas to supply the extreme ultraviolet radiation needed. The radiation travels through a complex condenser optics system before reflecting from a lithographic mask. That image is then projected by the projection optics onto a semiconductor wafer.

"The basic building blocks are the same as those found in systems operating at visible wavelengths, except their forms are different, because of the short wavelength of EUV," says Don Sweeney, Lawrence Livermore's program leader for EUVL.

Because all materials, including nitrogen and oxygen, absorb EUV, the machine must operate in a vacuum and use reflective mirrors and masks. The ETS has six essential subsystems: a laser-produced plasma EUV source, condenser optics, projection optics, a mask, precision scanning stages, and a vacuum enclosure.



A conceptual drawing of the extreme ultraviolet engineering test stand. The goal of the ETS is to demonstrate how ultraviolet wavelengths can be used to print patterns on integrated circuits at production levels and sizes.

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EUV-absorbing metal layer and then etching away the metal to form the image of the circuit.

One key requirement is to produce a mask with essentially no defects. Any small defect ends up being replicated, or printed, in the lithography process onto the computer chips being manufactured, thus damaging the chips' complex circuitry. A key breakthrough in this area was the development of an Ultra Clean Ion Beam Sputter Deposition System about two years ago. This system-also an R&D 100 Award winner-produces precise, uniform, highly reflective masks with fewer defects than those produced by conventional physical deposition processes. (See S&TR, October 1997, p. 8.) In April 1999, the team made significant improvements to the system's sputtering shield design and other operational parameters. The system now consistently produces fewer than 0.1 defects per square centimeter a factor-of-8 improvement over defect densities produced in 1998.

In fact, under the best operating conditions, the system adds as few as 0.04 median defects per square centimeter during a coating run of 25 wafers. The

ultimate goal for the system is to add no more than 0.001 defects per square centimeter to the finished wafer blank.

The system has also been upgraded to process 200-millimeter wafers—the size used in industry—up from 150-millimeter wafers. The ability to process larger wafers for mask substrates means both that the technology is working with industry standards and that patterns for larger chips can be placed on the wafer.

The Lawrence Livermore team has also conducted groundbreaking experiments looking at the propagation of defects during multilayer film growth. All masks have defects of some kindsome more, some less. First of all, there are defects that arrive on the wafer from the manufacturer. These are analogous to the pinholes and dust one finds on photographic negatives. Defects smaller than a certain critical size are covered up by the film layers and present no problem. However, defects larger than this critical size persist through the coating process and must be repaired or reckoned with in some way.

There are also defects created by the coating process itself: a few atoms too many in any one area can create a bump that will affect the final circuit pattern.

The question becomes, what is this critical size? "We've been modeling for a long time to see how different sizes and kinds of defects affect the final product," says Scott Burkhart, group leader for mask blank development. "We finally conducted experiments that are setting the lower bound of critical defect size."

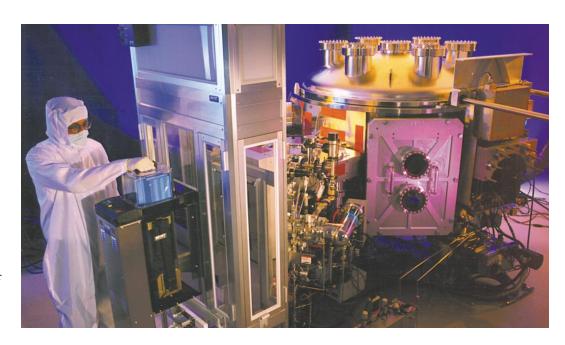
The group has also made strides in repair strategies for mask defects. "One mask can cost tens of thousands of dollars," notes Burkhart. "When possible, repairing the defects saves the industry a lot of money."

#### **Measuring at the Atomic Level**

Until recently, it was impossible to accurately measure a mirror surface for high and low spots of a few atoms. An R&D 100 Award–winning interferometer developed at the Laboratory two years ago—called the phase-shifting diffraction interferometer (PSDI)—changed all that. (See *S&TR*, October 1997, p. 6.)

Like all interferometers, the PSDI uses the interference pattern of two waves of light to measure objects or phenomena. These light waves are usually imperfect because of the

The Ultra Clean Ion Beam Sputter Deposition System, developed at Lawrence Livermore, is used to produce precise, uniform, highly reflective masks. A key requirement of the nextgeneration lithography system is that it produce virtually defect-free masks. The system contributes fewer than 0.1 defects per square centimeter to each mask. The ultimate goal for extreme ultraviolet lithography is to add no more than 0.001 defects per square centimeter to a finished wafer blank.



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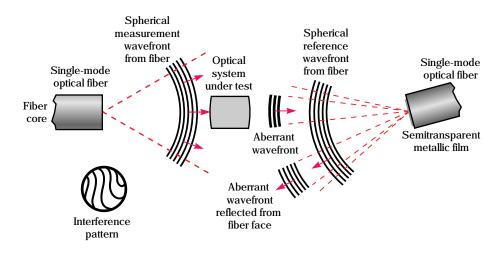
imperfect condition of the surface or lens from which they emanate. Any imperfection introduces error into the measurements. The PSDI produces a nearly perfect spherical wavefront using diffraction. In diffraction, light passes around an object or through a hole, breaking up in the process. In the PSDI, two light beams pass through two separate optical fibers. When light exits the surface of each fiber, it diffracts, forming nearly perfect spherical wavefronts. Because the two wavefronts are generated independently, their relative amplitude and phase can be controlled, providing contrast adjustment and phase-shifting capability for the highest possible accuracy.

The measurement wavefront passes through the optical system being tested, which induces aberrations in the wavefront and causes it to focus on the endface of the other fiber. Here, the wavefront reflects off a semitransparent metallic film of the fiber end's surface and interferes with the reference wavefront to generate an interference pattern. The pattern is then recorded by a charge-coupled-device camera.

Over the past three years, many EUV optics have been measured using this interferometer, including both concave and convex spherical and aspherical mirrors and completed projection systems. The PSDI is now a reliable production tool for measuring the overall surface shape of those aspherical optics that have a specification of 0.50 nanometers or less and has successfully measured errors in the surface shape down to 0.35 nanometers. The Livermore metrology team is upgrading the system so that it can be used to measure errors in the overall surface shape as small as 0.15 nanometers.

#### **EUV Pulling Ahead in the Race**

Last December, the VNL's work paid off with a vote of confidence from International Sematech, a privately



Schematic of the phase-shifting diffraction interferometer which uses two single-mode optical fibers. In this example, the interferometer is testing a lens, but the setup is similar for testing mirrors.

funded organization of semiconductor manufacturers.

At its annual meeting, International Sematech evaluated the four next-generation lithographic technologies— EUV, x-ray, electron-beam, and ion-beam—and strongly recommended EUV lithography. "Their recommendation gave our efforts important momentum," notes Sweeney. "It validated what we already knew: that we have a winning combination in the three national laboratories and our industrial partners and that our strength comes from working together."

-Ann Parker

Key Words: Extreme Ultraviolet Limited Liability Company, extreme ultraviolet lithography (EUVL), masks, phase-shifting diffraction interferometer (PSDI), precision deposition system, reflective multilayers, submicrometer metrology, thin films, Ultra Clean Ion Beam Sputter Deposition System, Virtual National Laboratory (VNL).

For further information contact Donald Sweeney (925) 422-5877 (sweeney4@llnl.gov). Or visit the Lawrence Livermore EUV lithography Web site at http://lasers.llnl.gov/ lasers/IST/euvl.html.

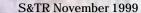
#### **About the Scientist**



DONALD SWEENEY received his B.S., M.S., and Ph.D in mechanical engineering from the University of Michigan at Ann Arbor in 1968, 1969, and 1972, respectively. He was a professor at Purdue University from 1972 to 1983, after which he became a department manager at Sandia National Laboratories/California and assumed responsibility for a research program in optical diagnostics.

Sweeney joined Lawrence Livermore's Advanced

Microtechnology Program (AMP) in early 1993 and soon became deputy program leader for Optics Technology. In February 1999, AMP reorganized and became the Information Science and Technology Program, where he is currently deputy program leader for Extreme Ultraviolet Lithography and Advanced Optics.



## Handling Fluids in Microsensors

Wanted: Small, portable devices for automatically detecting and identifying viruses, bacteria, and toxic chemicals.

**Reward:** Satisfaction in knowing that microsensors may help save lives.

ORK has been under way for several years at sites throughout the departments of Energy and Defense on autonomous devices for detecting biological and chemical agents. The goal is to install them in subways, major office complexes, convention centers, or other sites where the public is at high risk of exposure to a covert release of biological or chemical agents. They will also be part of a network of sensors that will monitor urban areas or large events such as inaugurations or the Olympics. They will find their way onto the battlefield to protect soldiers in action. Used to analyze blood or other samples, such systems may detect and diagnose diseases in the field, far from laboratories and hospitals.

These monitoring systems must be robust and easy to operate and maintain. They must also have low power requirements to be truly portable and not rely on the large batteries that frequently accompany so-called fieldportable devices today. And of course, the systems, like the tiny components that make them up, must be small and lightweight, which would not be possible without the ongoing revolution in microtechnology, particularly microelectromechanical systems. (See "The Microtechnology Center: When Smaller Is Better," S&TR, July/August 1997, pp. 11–17, and "Countering the Bioweapons Threat," *S&TR*, June 1998, pp. 4–9.)

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Handheld instruments will incorporate microchip devices to take in an air or fluid sample; filter out smoke, dust, and other contaminants; mix the desired particles with fluid reagents as needed; and pump the mixture to sensors at the other end to determine what pathogens or toxins, if any, are present. Micromachined from silicon, glass, plastics, and ceramics, the components will have channels 20 to 200 micrometers deep and up to a millimeter wide through which fluids will travel. (Keep in mind that a human hair is just about 50 micrometers in diameter.)

Complete microfluidic systems have been a dream for more than a decade. At Livermore's Center for Microtechnology, experts in biology, electronics, optics, and engineering are working together on several unique components that will make them a reality.

#### Two Projects, One Goal

Two microfluidic projects, one sponsored by the Department of Energy and the other by the Department of Defense, are currently under way at the Center for Microtechnology. The sponsorship is different but the goals for both projects are to develop systems for handling fluids in autonomous detectors for biological pathogens.

Robin Miles leads the engineering team that is developing fluidic systems for DOE. This project, which includes participants from several DOE sites, is developing an autonomous device dubbed Sentry, whose functions will include continuous or on-demand air sampling, sample preparation, automated fluidic sample handling and transport, detection and identification of pathogens by immunoassay and DNA recognition, and automated data analysis and reporting.

Engineer Peter Krulevitch and his team are working under a subcontract

to researchers at the University of Texas M. D. Anderson Cancer Center on the DoD project, which is sponsored by the Defense Advanced Research Projects Agency (DARPA) under the Microfluidic Molecular System (MicroFLUMES) Program. The purpose of this project is to develop an instrument that incorporates new technologies for separating particles (known as fractionation), sensing them, and identifying them based on their dielectric properties. Its first use will be to perform differential cell analysis on blood samples.

Building sophisticated, multifunctional, automated sample preparation systems for field use is still primarily a research and development activity. Most systems available commercially either assume a laboratory setting for testing or are designed for one use only. Also, many fractionation methods require filters that become clogged over time and contribute to the carryover of particles between tests. Furthermore, most operate using pneumatic power, which is excellent for microfluidic actuation because it can provide large forces over long distances and conform to the tiny tubes. But such systems require carting around a bulky canister of compressed air, and pneumatic valves cannot be miniaturized sufficiently to make these systems easily portable.

Both Livermore projects circumvent these challenges by using new methods of pumping, fractionation, mixing, and sample concentration and purification. Krulevitch's team has also developed a new sensing device that uses changes in impedance to identify particles.

The teams are working toward integrating their assorted components into a single instrument. Fabrication techniques, fluid conductivities, and fluid velocities, among other concerns,

must be compatible for each overall system to perform optimally. System integration is key for both programs.

#### **Inside Sentry**

Because Sentry is intended for detecting and identifying biological or chemical warfare agents in the field, it is being designed to collect samples from the air. But samples could just as easily be blood or tissue for diagnosing disease. Processing the samples involves mixing reagents or microscopic polystyrene beads coated with antibodies with the potentially pathogenic particles. Pathogens in the sample will cling to beads coated with the appropriate antibody. Then the sample will be concentrated to facilitate the use of DNA-based assays. Combining a DNAbased assay with an antibody-based assay greatly increases Sentry's overall reliability in identifying pathogens.

Livermore engineer Amy Wang is exploring the use of acoustic energy to manipulate particles in the sample and to enhance the mixing of sample and reagent. Microscale mixing is a challenge because small channel dimensions make it difficult to create turbulence. Acoustic mixing brings with it the advantage of rapid mixing, no moving parts, and no need for nozzles or external injection of fluids to create turbulence. The ability to mix samples on the chip will speed up the rate of binding for immunoassays, increasing the throughput and speed of the system.

#### **Electronic Filtering**

Miles is using dielectrophoresis as a method of filtering the sample to collect the particles of interest in Sentry. Dielectrophoresis is an electrical phenomenon that allows particles to be trapped or manipulated by applying nonuniform electrical fields, which Microfluidics S&TR November 1999

induce electrical polarization in the particles. Depending on the polarizability of the particle with respect to the medium in which it is suspended, it will move toward or away from regions of high field intensity. Motion toward the regions of high field intensity is termed positive dielectrophoresis, while motion away from them is negative.

Dielectrophoretic forces provide an electrically switchable means to discriminate between particles and to manipulate them according to their dielectric properties. This phenomenon is ideally suited to microfluidic situations because large field strengths and correspondingly high dielectrophoretic forces are readily achievable with electrodes spaced less than a millimeter apart. Spores, bacteria, and cells, whose sizes range from 1 to 10 micrometers, may be captured with dielectrophoretic electrodes using less than 2 volts. Furthermore, dielectrophoretic forces are effective even for extremely small particles such as DNA.

Miles notes that this use of positive dielectrophoretic forces to electronically filter a sample—which has never been done commercially before—solves a couple of problems. First, it can be used to remove soil, smoke, pollen, mold spores, oil, and other contaminants from the raw sample. These contaminants may inhibit the operation of assays for DNA recognition based on the polymerase chain reaction (PCR), the most reliable method of identifying biowarfare organisms. Contaminants make antibody-based assays operate less effectively, too. Antibody assays are less specific than PCR, but they can

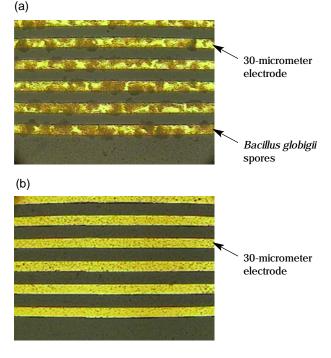
be faster when the target organism is present in high concentrations. These assays also allow for simultaneous detection and identification of multiple biowarfare agents including toxins. Finally, using an electronic method to filter the sample eliminates the manual handling of samples and the large volumes of reagents and filter media typically used in laboratory analyses.

The dielectric properties of cells have been characterized by numerous experiments. But the dielectrophoretic properties of antibody-coated beads, spores, and DNA are less well known. Miles and her team have explored them in numerous experiments.

They studied the nontoxic *Bacillus* globigii spores and the vegetative bacteria Erwinia herbicola-simulants for anthrax and plague, respectivelyto demonstrate their capture using dielectrophoretic electrodes. As shown at left, the force of attraction to the electrodes is sufficient to overcome the force on the spores due to fluid flow, allowing debris in the carrier fluid to be washed away while the spores are held in place. Another series of experiments determined the optimal capture frequency for several bioparticles of interest, including various types of DNA, Bacillus globigii, Erwinia herbicola, and beads.

On the basis of these and other experiments, the team believes it knows the best way to capture particles. Three parameters—the magnitude and direction of the dielectrophoretic force, the frequency of the electrodes, and the electrode geometry—are adjusted to selectively capture cells, spores, polystyrene beads, or DNA. Once the particles are captured, they are held in place against the flow of fresh carrier solution for a short time. Then the electric field is removed so the particles can be suspended in clean solution.

(a) Bacillus globigii, a simulant for anthrax, collects on activated electrodes as it flows through water. (b) The spores do not collect on inactivated electrodes.



The figure at top right shows the existing prototype of the component for testing the dielectrophoretic concentration of particles suspended in water. Other microfluidic pieces of Sentry are being designed to fit together with this component in an integrated system.

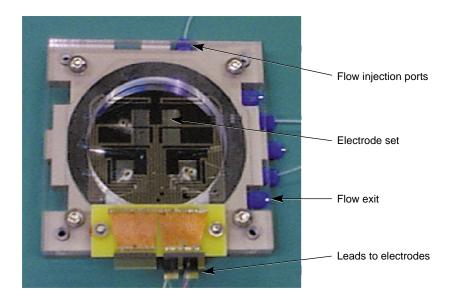
#### **Keeping It Moving**

Magnetohydrodynamic (MHD) pumps are being developed by physicist Sony Lemoff and others at Livermore to move fluid through all phases of Sentry's microfluidic system. They have been the first to demonstrate MHD pumps for aqueous solutions that function on a microchip and are pioneering the use of MHD pumps for micromachined applications.

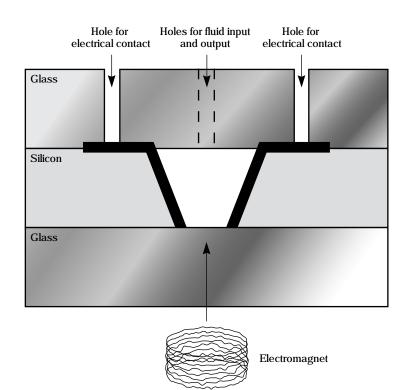
There are several types of nonmechanical pumps, but thus far, the MHD pump is the most effective for producing a continuous, nonpulsating flow in a complex microchannel design. An MHD pump consists primarily of an electromagnet and a series of metal electrodes. Multiple pumps on the same chip can be driven independently by varying their electrode current amplitude and phase relative to the electromagnet, thus enabling routing in complex integrated microfluidic systems.

As shown in the figure at the lower right, the channels are etched through a silicon wafer with electrodes deposited on the walls of the channels. The silicon is then sandwiched between glass plates with holes for electrical contacts and fluid input and output. An electromagnet is positioned beneath the device.

The first-generation MHD pump had channels 380 micrometers deep and 800 micrometers wide, huge in the microtechnology world. Lemoff notes, "We are aiming for channels that are 20 by 50 or 50 by 100 micrometers. But



Component for testing the dielectrophoretic concentration of particles suspended in water. Acoustic subsystems and magnetohydrodynamic pumps for Sentry are being designed to integrate with it.



A cross section of the magnetohydrodynamic micropump. It is currently fabricated of glass and silicon, but the use of other materials is being explored. Microfluidics S&TR November 1999

with such small cross-sectional areas, we need higher magnetic field strength to push the fluid through." The early system used 0.025-tesla magnets, but they are being replaced with much more powerful 0.2-tesla ones.

#### **Another Approach**

The project with the University of Texas M. D. Anderson Cancer Center takes a different tack. Krulevitch and his team are responsible for microfabrication on the project and have designed the microfluidic chip.

For fractionation, the DARPA project team is using negative dielectrophoresis to keep particles "levitated" in the microfluidic channel away from electrodes. Krulevitch's team together with the Anderson researchers have designed the microchannels and dielectrophoretic electrode arrays that make this separation system work.

The DARPA project team has also developed an impedance sensor that

can detect particles as they leave the system and characterize them both by their time of passage through the separation column and by their size and dielectric frequency spectrum. This sensor consists of two electrodes on opposite sides of the channel. The resistance and capacitance—and hence the impedance—between the two electrodes is already known. When a particle passes between the electrodes, the impedance value changes. Directcurrent impedance sensors are used in commercial blood cell and microparticle counters, but they indicate only particle size. This new version, using alternating current, represents a considerable improvement.

The integrated injection—separation—detection component shown below consists of microscopic sorting channels 150 micrometers deep, 1 millimeter wide, and 10 centimeters long arranged in serpentine fashion on a 2.5- by 4-centimeter substrate. Channels are equipped with integrated arrays of

dielectrophoretic electrodes 50 micrometers wide with 50micrometer-wide gaps. At the exit of the channel are impedance sensors with their independent electrodes.

In operation, a micropump sends 1-microliter samples of 10-micrometer-diameter surface-coated beads (for detecting warfare agents) or human cells (for detecting disease) into the microchannel. The samples are then slowly flushed through with fluid from another small reservoir. Beads or cells are detected as they pass the impedance sensors after fractionation along the channel.

At the M. D. Anderson Cancer Center, both the fractionation process and the impedance sensor using both beads and cells have performed well in initial tests. Plans call for the fully integrated blood-analysis fractionation system, which is being packaged by LYNNTECH, Inc., of College Station, Texas, to be delivered to DARPA in the fall of 2000.

#### **Simulations of Microactivity**

The transport and manipulation of beads and pathogenic particles must be predictable for all of the components to operate together in the DOE and DARPA systems.

To guide work on these subsystems, engineer David Clague has developed an enhanced three-dimensional, discrete simulation model that permits the study of stationary and mobile particles in microfluidic devices. The model is being extended to incorporate intermolecular force interactions. Because the channels in microfluidic systems are so small, intermolecular forces, which are typically masked in laboratory-scale instruments, affect the behavior of particles as they move through the system and are manipulated.

Known as lattice Boltzmann, the model is based on the Boltzmann



The integrated injection–separation–detection system being developed by the DARPA project team resides on a 2.5- by 4-centimeter substrate.

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equation, which easily incorporates external forces, thermal diffusion, and device wall interactions in studies of the dynamic behavior of a collection of particles (see the figure at right). This contrasts with traditional microfluidic modeling based on finite-element analysis and boundary-element methods, which in this area of research typically deal only with pure fluid effects.

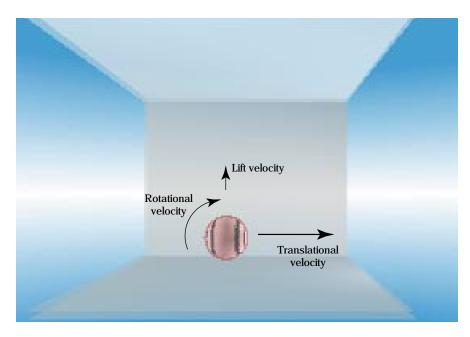
In the near future, Clague will incorporate electromagnetic-induced pressure fields to study particle behavior during each process in the two instruments. As work on the components progresses and the configuration of the overall systems is determined, simulations will help explain the interplay of fundamental-force interactions in each subsystem.

#### **Fabricating the Systems**

The goal of all this work is to have fully integrated systems. Nowhere does this present a greater challenge than in the area of fabrication. Currently, no single-material system has proven superior to all others. Each has its merits and drawbacks, depending on the specific function of the component. When the various functions are being integrated, multiple-material systems must be considered, including associated packaging and interconnection technologies.

The most commonly used materials for microfluidic systems are polymers, silicon, and Pyrex glass. Livermore has also begun fabricating microfluidic devices using ceramics and other glasses. Engineer Harold Ackler is developing fabrication processes for both projects and exploring a number of fabrication methods for the various subsystems.

He and other Livermore researchers are developing proprietary technology that will integrate new ceramic and glass devices with commercially available microelectronic packaging and other



A lattice Boltzmann model of fluid flow and particle movement in a microchannel. In this cutaway side view of a microchannel, the sphere representing a particle moves from left to right. Inertial forces and wall interactions tend to lift the rotating sphere, translating it toward the center of the microchannel. The DARPA project team is attempting to exploit this lift phenomenon in the design of a fractionation device based on negative dielectrophoresis.

proprietary Livermore fluidic interconnect technology. This integrative capability will make the now troublesome task of making fluidic and electronic connections as simple as plugging in a packaged integrated circuit. Ackler notes, "Being able to integrate these devices so easily is extremely attractive for systems deployed in the field in which replacement of a component or a consumable material like a reagent must be quick and simple."

The team has several material-related issues to handle. Eliminating the most rapid corrosion mechanism has solved the problem of electrode corrosion. Problems with spores, dirt, DNA, and other materials adhering to the various surfaces in the system are being dealt with. The first-generation MHD pump was made of silicon and Pyrex glass, but newer ceramic and glass alternatives are also being studied.

While it may be possible to fabricate all components with the same materials, some functions may not be optimal. So Livermore is taking a dual approach, examining the use of discrete components as well as pursuing the development of a fully integrated fabrication method wherein all components are integrated into the same piece of material. The latter approach should reduce system size, power requirements, use of consumables, packaging problems, and manufacturing costs. But if the use of discrete components results in superior system performance, Ackler will take that route, making use of Livermore's integration and packaging technology.

#### **Complete Systems Soon**

The Center for Microtechnology is involved in numerous microfluidic projects. In addition to the microfluidics Microfluidics S&TR November 1999

work for DOE and DARPA, the center is participating with the University of Minnesota and three other universities in a proposal submitted in recently to the National Science Foundation for a Center for Biomedical Microsystems. If the proposal is funded, 22 industrial partners have committed to assist the collaboration in developing diagnostic and therapeutic microsystems, including micropumps and other microfluidic subsystems.

For work under way now, a fully integrated microfluidic system for the DARPA project will be delivered next year, while completion of the DOE detector is about three years away. Ray Mariella, director of Livermore's

Center for Microtechnology, is pleased with successes to date. "Livermore has been a leader in the microfabrication world for quite a while. Producing a usable microfluidic system as part of a detector for biological and chemical agents will be a real feather in our cap."

—Katie Walter

**Key Words:** Center for Microtechnology, dielectrophoresis, impedance sensor, magnetohydrodynamic (MHD) pump, microdevices, microfluidics.

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#### **About the Engineers**



ROBIN MILES joined Lawrence Livermore in 1997 as a mechanical engineer specializing in the development of microdevices for biological applications. Currently, she is an engineer and group leader in the Laboratory's Center for Microtechnology. She is coprincipal investigator for sample preparation on an autonomous, continuous monitoring system for counterbiological warfare pathogens and principal investigator on a project to build a biological

processor using electric fields.

Miles earned an B.S. in mechanical engineering from the Massachusetts Institute of Technology, an M.S. in mechanical engineering from Stanford University, and an M.B.A. from the University of California at Berkeley.



PETER KRULEVITCH holds a B.S., M.S., and Ph.D. from the University of California—all in mechanical engineering. He joined Lawrence Livermore in 1994 as a postdoctoral fellow and is currently a microelectromechanical systems researcher in the Engineering Directorate's Center for Microtechnology. He is principal investigator for a project to develop microfabricated cell separation and detection systems in collaboration with the University of Texas M. D. Anderson

Cancer Center. He is also working on projects to develop shape-memory film microactuators for medical and microfluidic applications, to create finite-element models of micromechanical devices, to microfabricate a temperature–pH biosensor, and to investigate the mechanical properties of thin films for multilayer mirrors.

Krulevitch is the coholder of eight U.S. patents for a variety of microdevices and their fabrication methods, primarily for medical and biotechnology applications.

## A Crowning Achievement for Removing Toxic Mercury

IKE its namesake, the messenger of the gods, mercury is notoriously mobile in the environment. Watersoluble and toxic mercury readily leach out of landfills and even wastes solidified with cement. In recent years, environmental scientists and regulators have focused on the development of new processes to remove mercury ions from solutions more efficiently and cheaply than present methods.

The technical challenge is formidable because any method must be impervious to the corrosive nature of the waste streams that contain these ions. In addition, these waste streams can contain a variety of other metal ions (sometimes in much higher concentrations)—some of which also possess the same +2 charge as mercury ions. So an effective removal process must be selective of only mercury ions.

In response to the need for a better method for mercury removal, a team of Lawrence Livermore chemists (Glenn Fox, John Reynolds, and Ted Baumann) has designed an organic polymer called Mercaptoplex that demonstrates an unusually strong affinity for mercury ions in solution. Tests at Livermore show that Mercaptoplex extracts more than 95 percent of mercury ions and does so faster and more selectively than other techniques such as precipitation and activated carbon absorption. Originally developed for use in processing nuclear fuel rods at the Department of Energy's Idaho National Engineering and Environmental Laboratory, the molecule can also remove mercury from both industrial waste streams and public water supplies.

Mercaptoplex has demonstrated a remarkable capacity for removing mercury ions under a broad range of conditions, including those currently found in government and industrial waste streams. In addition, the molecule can



Glenn Fox (left), John Reynolds, and Ted Baumann have developed Mercaptoplex, a polymer for removing toxic mercury from waste streams.

be reused indefinitely after the bound mercury is removed, making the process cost-effective. Because of its ability to be recycled, the molecule minimizes the amount of secondary waste generated during extraction, a major challenge in waste treatment.

#### Three Molecules in One

Mercaptoplex is really three molecules combined into one. The business end belongs to a class of organic compounds called crowns, which are molecular rings that contain metal-binding atoms incorporated into their carbon frameworks. The original crowns featured oxygen atoms linked together in a ring by carbon atoms. They earned their name because the molecule looks like a crown when viewed from the side.

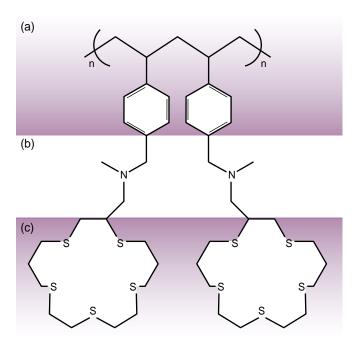
Crown Polymers S&TR November 1999

The oxygen atoms can be replaced with sulfur atoms to form a crown that exhibits a high affinity for mercury ions, through the donation of electrons to the positively charged mercury ion. (The molecule is still called a crown in chemistry parlance, although the sulfur atoms do not confer a crown appearance.) Together, the five linked sulfur atoms of Mercaptoplex form a strong complex with a single mercury ion.

The sulfur crown is attached to the second Mercaptoplex constituent, a nitrogen-linking unit. The researchers surmise that this unit facilitates interaction between the crown and the acidic aqueous solution. It also links the sulfur-containing crown to the third component, a backbone of cross-linked polystyrene molecules (polystyrene is the chief ingredient of the ubiquitous Styrofoam coffee cup).

#### **Strong Polystyrene Backbone**

The Livermore chemists chose a backbone of polystyrene because its chemistry is well understood and its simple cross-



Two repeating units of the mercury-extraction polymer, Mercaptoplex. The molecule consists of three parts: (a) a backbone of cross-linked polystyrene molecules (n) makes the molecule insoluble in water; (b) a nitrogen-linking unit (N) facilitates interaction between the crown and acidic solutions; and (c) a "crown" of five linked sulfur atoms (S) binds to a single mercury ion in solution.

links of divinylbenzene transform the molecule into a highly entangled and thereby insoluble repeating unit (or polymer) that does not dissolve in water. The Livermore team postulates that other materials, such as polymers of polyethylene, may also prove effective as backbones.

In solution, because of the entangled nature of the Mercaptoplex polymer, it is probable that neighboring crowns combine to trap mercury ions. For example, two sulfur atoms from one crown may combine with three sulfur atoms from a nearby crown to bind to a mercury ion. Studies using spectroscopic techniques are under way at Lawrence Livermore to gain a better understanding of the bonding mechanism.

The Livermore chemists have shown that Mercaptoplex is effective at pH ranging from 1.5 (extremely acidic) to 7.0 (neutral). In contrast, precipitation, a common technique of mercury removal, requires constant pH adjustment. If the pH gets too low (too acidic), the precipitation process produces hydrogen sulfide, a highly toxic gas, and does not remove the mercury. The other popular mercury removal process, activated carbon, also requires continuous adjustment of pH.

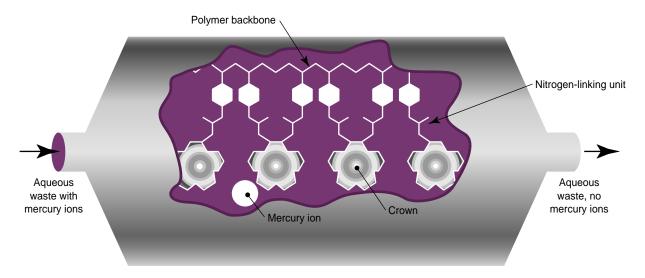
Mercaptoplex is also faster and more selective in removing mercury than other techniques. When mixed with solutions containing mercury ions, it captures virtually all of the mercury within 30 minutes. This extraction rate is much faster than that seen in other systems, which can take up to 20 hours to do their job. Baumann says the ultimate goal is to use Mercaptoplex as packing for large columns to speed up the waste treatment process. In this design (shown on p. 19), the waste stream would simply flow through the Mercaptoplex without the need for mixing.

Fox notes that because typical mixed waste streams (those combining both toxic and radioactive materials) contain a variety of other metal ions, such as aluminum, iron, cadmium, and lead, removal of mercury requires a highly selective process. The chemists have tested Mercaptoplex in solutions of mercury ions ranging from 4 to 200 parts per million and when concentrations of other ions outnumber mercury by 100 to 1. In every case, Mercaptoplex has selectively removed mercury with an efficiency of 95 percent or greater. (Baumann says mercury removal is probably greater than 99 percent, but the amount of mercury left in solution after treatment is too small for the chemists to measure accurately.)

#### **Recycling Is a Big Advantage**

Because Mercaptoplex is insoluble in water, it can be easily separated from solution by filtration once the extraction

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Schematic of column treatment of mercury waste using Mercaptoplex.

is complete. The mercury can then be recovered, and the Mercaptoplex regenerated by a variety of treatments. One method developed by the Livermore team is to use chloroform solutions of diphenylthiocarbazone to strip the bound mercury from the polymer. Under these regeneration conditions, the diphenylthiocarbazone has an even greater affinity for mercury ions than does the sulfur crown. Once rinsed and dried, Mercaptoplex has been used to effectively treat additional volumes of mercury. The team is investigating other methods of stripping the mercury ions from the polymer such as electrochemically reducing the ions to the safer metallic mercury.

In comparison to Mercaptoplex, other techniques typically require additional treatment steps and generate large amounts of secondary waste. Precipitation generates mercury sludges that require further treatment. Activated carbon columns loaded with mercury are rarely regenerated, and the spent columns require additional processing.

At the Idaho National Engineering and Environmental Laboratory, where mercaptoplex was first used, mercury is used as a catalyst to treat spent fuel rods from U.S. Navy submarines. The Livermore process is also applicable at other DOE sites that need selective and cost-effective treatments for mixed waste.

The process should prove useful in treating industrial waste streams and water supplies that contain mercury. For example, the Livermore team has discussed the process with representatives from the bleach manufacturing and oil

industries, who must meet strict federal regulations concerning mercury levels in their waste streams.

By simple substitution of the sulfur atoms, the molecule can be tailored to target other metal ions, such as cadmium, silver, and lead, commonly found in mixed waste streams and water supplies. "There is a lot of synthetic chemistry you can do with crowns," says Fox, "such as modifying the number of noncarbon atoms in the ring to better bond to the ion in the solution of interest. In this way, chemists can target a particular metal pollutant through careful molecular design."

-Arnie Heller

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**Key Words:** activated carbon, crown polymers, Idaho National Engineering and Environmental Laboratory, Mercaptoplex, mercury, precipitation.

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OR years, manufacturers of electronics with flat-panel displays have dreamed of using plastic as a cheaper, more compact, more rugged, and far more lightweight alternative to glass. The Department of Defense is particularly interested in ultrathin yet flexible screens as standard equipment for the Pentagon's "information warrior" of the next century. With plastic displays, soldiers could hang satellite navigation system displays on their belts or keep electronic maps rolled up in a back pocket.

The most advanced type of flat-panel displays, used in most portable computers, is active-matrix liquid-crystal displays. In this display, each of the million or so tiny screen pixels is controlled by thin-film transistors (TFTs) that act as tiny on/off electrical switches. By turning on and off dozens of times a second, the TFTs permit continuously changing images of words, pictures, and video.

Currently, TFTs for active-matrix displays are manufactured onto a rigid glass substrate in a process that involves baking glass sheets at temperatures of up to 600°C. This conventional process is far hotter than any plastic can withstand without deforming and melting. But now a team of

Lawrence Livermore researchers is showing how TFTs can be manufactured on top of thin, flexible plastic sheets instead of glass by keeping manufacturing temperatures at or below 100°C.

The work was carried out by a group of electrical engineers, physicists, and materials scientists in the Device and Process Group in the Information Science and Technology Program of the Laser Programs Directorate. The research is part of a larger effort by Livermore scientists and their Department of Energy colleagues to apply laser-based processing techniques to current U.S. semiconductor production problems. The plastic substrate project, now in its third year, is funded by the Defense Advanced Research Projects Agency's High-Definition Systems Program, which sponsors development of new display concepts that address the issues of lighter weight, improved ruggedness, lower power, higher resolution, and easier use.

#### Laser Pulses Fast, Precise

The novel Livermore transistor fabrication process combines well-established, low-temperature deposition techniques with excimer lasers that produce pulsed beams of ultraviolet light. These lasers are a much more powerful version S&TR November 1999 Plastic Display Substrates

of the instruments that are used in eyesight correction surgery to literally vaporize corneal tissue without damaging surrounding tissue. In fact, the lasers are so precise they can make precise notches in human hair that can be exactly and repeatedly duplicated. The Livermore team takes advantage of the laser's extreme precision and ultrafast operation to melt, crystallize, and dope (add impurities to) the silicon layers forming the TFTs at substrate temperatures lower than the melting temperature of plastic.

The Livermore team chose one of the most common plastics for the substrate: polyethyleneterephthalate (PET), more commonly known as polyester. Thin (175 micrometers), cheap, flexible, transparent, and rugged, PET is used for many other purposes, including the Mylar for viewgraphs. Standard 10-centimeter-diameter wafers are cut from 61-centimeter-wide rolls of PET. Onto these plastic circles are applied the materials fundamental to integrated circuits: an insulator (silicon dioxide), semiconductor (crystallized silicon or polysilicon), dopants of selected elements, and metal connectors.

The process begins with a thin layer of silicon dioxide deposited on the plastic wafer through a conventional process called plasma-enhanced chemical-vapor deposition that produces uniform films of molecules. Next, the team uses sputter deposition to apply an amorphous layer of silicon atoms to the substrate. Both of these layers are applied at a relatively cool temperature of about 100°C to keep the plastic intact.

The excimer laser irradiates the amorphous silicon layer from 3 to 10 times at an ultraviolet (UV) wavelength of 308 nanometers. Each pulse lasts only 35 nanoseconds (billionths of a second) while melting the amorphous silicon. The result (shown on p. 22) is a highly ordered, polycrystalline layer of silicon atoms some 40 nanometers thick. (This transformed silicon, typically called polysilicon, permits electrons to move more easily through its highly ordered lattices.)

#### **Plastic Doesn't Melt**

During the melting process, the fleeting UV laser energy is absorbed mainly in the top 10 nanometers of the amorphous silicon layer before it diffuses downward into the plastic. That localization of the laser energy, together with the silicon dioxide layer that acts as a thermal barrier, keeps the plastic substrate from heating and melting.

Although the silicon layer melts at 1,400°C, the plastic barely notices the heat from the deposited laser energy. The team's understanding of the physics and chemistry of the laser processing steps is aided by advanced simulation work done at Livermore.

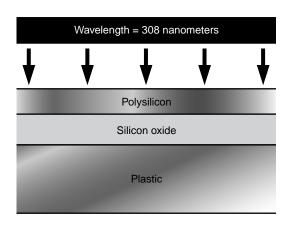
The laser beam is adjusted to cover from 1 to 11 square millimeters at the wafer surface. Covering the entire wafer takes about one minute. In contrast, traditional processes require baking glass sheets in high-temperature furnaces for many hours.

The next steps are modified, lower-temperature versions of traditional semiconductor processing involving photolithography, which uses a sequence of photomasks. These masks act as photographic negatives do, allowing light to imprint a pattern on the wafer. The pattern defines the areas to be removed through etching, doped with impurities, and deposited with aluminum connectors.

The doping with boron and other elements is accomplished using another pulsed excimer laser in a technique also developed at Livermore. First, a thin layer of doping atoms is deposited using plasma-enhanced chemical-vapor deposition. Then repeated laser pulses drive the atoms deep into the polysilicon. (Doping allows the polysilicon, which is essentially an insulator, to conduct electricity by giving up or attracting electrons.)

#### **Switches Ready for Connection**

The result is a 10-centimeter-diameter array of several hundred simple switches ready to be joined to its neighbors and to a liquid-crystal-display system. The Livermore team is continuing to refine the low-temperature manufacturing process. In particular, it is working to achieve TFTs that permit electrical current with higher "mobility," or speed. The bigger the display, the higher the desired mobility.



An excimer laser irradiates a layer of amorphous silicon atoms at a wavelength of 308 nanometers for about 35 nanoseconds. The process is repeated up to 10 times.

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#### (a) Before laser

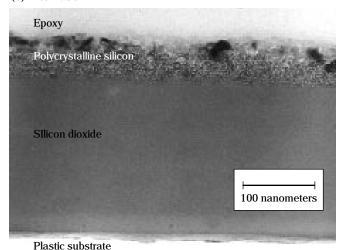
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Epoxy
Amorphous silicon

Silicon dioxide

Plastic substrate

#### (b) After laser



Electron micrographs show how the excimer laser pulses transform the amorphous silicon layer into a 40-nanometer-thick layer of polycrystalline silicon.

The research has progressed sufficiently that discussions are taking place with U.S. flat-panel-display manufacturers to license the technology. It is anticipated that an industry—Livermore project to develop a complete prototype would combine Livermore's plastic "backplane" of TFT-driven picture elements, or pixels, with liquid crystals or organic light-emitting materials furnished by a display manufacturer.

Display manufacturers are particularly interested in the potential to manufacture large displays inexpensively, particularly with a roll-to-roll continuous manufacturing technique much like the roll-to-roll printing process. In this scenario, the plastic would roll through processing stations similar to those of a printing press, and finished displays would be cut to size.

The Livermore breakthrough may well make possible within a few years a new generation of ultralight, flexible, and inexpensive displays. Applications could include notebook and desktop computer displays, instrument panels, video game machines, videophones, mobile phones, handheld PCs, camcorders, satellite navigation systems, smart cards, toys, and a new generation of electronic devices for which flat-panel displays have been too heavy or too costly. Indeed, it looks as if plastic flat-panel displays will be used by everyone, from couch potatoes to information warriors.

-Arnie Heller

**Key Words:** active-matrix liquid-crystal display, amorphous silicon, Defense Advanced Research Projects Agency, enhanced chemical-vapor deposition, excimer laser, flat-panel display, liquid-crystal display, polysilicon, sputtering, thin-film transistor (TFT).

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Each month in this space we report on the patents issued to and/or the awards received by Laboratory employees. Our goal is to showcase the distinguished scientific and technical achievements of our employees as well as to indicate the scale and scope of the work done at the Laboratory.

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#### **Patents**

Patent issued to	Patent title, number, and date of issue	Summary of disclosure
Paul Wickboldt Paul G. Carey Patrick M. Smith Albert R. Ellingboe	Deposition of Dopant Impurities and Pulsed Energy Drive-In U.S. Patent 5,918,140 June 29, 1999	A semiconductor doping process that enhances the dopant incorporation by using the gas-immersion laser-doping technique. The enhanced doping is achieved by first depositing a thin layer of dopant atoms on a semiconductor surface, followed by exposing the semiconductor to one or more pulses from either a laser or an ion beam to melt a portion of the semiconductor to a desired depth. This process causes the dopant atoms to be incorporated into the molten region. After the molten region recrystallizes, the dopant atoms are electrically active. The dopant atoms are deposited by plasma-enhanced chemical-vapor deposition or another known deposition technique.
Joseph P. Fitch	Sparse Aperture Endoscope U.S. Patent 5,919,128 July 6, 1999	An endoscope that has a smaller imaging component, maintains resolution of a wide-diameter optical system while increasing tool access, and allows stereographic or interferometric processing for depth and perspective information and visualization. Because its imaging optics are smaller, more of its volume can be used for nonimaging tools, thus permitting smaller incisions when it is used in surgical and diagnostic medical applications. In turn, it produces less trauma to the patient or allows access to smaller volumes than is possible with larger instruments. The endoscope has fiber-optic light pipes in an outer layer for illumination, a multipupil imaging system in an inner annulus, and an access channel for other tools in the center. The endoscope can be used as a flexible scope, thus increasing its utility. Because the endoscope uses a multiaperture pupil, it can also be used as an optical array, allowing stereographic or interferometric processing.
G. Bryan Balazs Patricia R. Lewis	Mediated Electrochemical Oxidation of Organic Wastes Using a Co (III) Mediator in a Neutral Electrolyte U.S. Patent 5,919,350 July 6, 1999	An electrochemical cell with a cobalt (III) mediator and neutral pH anolyte, which provides efficient destruction of organic and mixed wastes. The organic waste is concentrated in the anolyte reservoir, where the cobalt mediator oxidizes the organics and insoluble radioactive species and is regenerated at the anode until all organics are converted to carbon dioxide and destroyed. The neutral electrolyte is noncorrosive and thus extends the lifetime of the cell and its components.
Jay A. Skidmore Barry L. Freitas	Microlens Frames for Laser Diode Arrays  U.S. Patent 5,923,481  July 13, 1999	Monolithic microlens frames to enable the fabrication of monolithic laser diode arrays. They can be manufactured inexpensively and have high registration and inherent focal length compensation for any lens diameter variation. A monolithic substrate is used to fabricate a low-cost microlens array. The substrate is wet-etched or sawed with a series of V grooves. The V grooves can be created using wet-etching by exploiting the large etch-rate selectivity of different crystal planes. The V grooves provide a support frame for either cylindrical or custom-shaped microlenses. Because the microlens frames are formed by photolithographic semiconductor batch-processing techniques, they can be formed inexpensively over large areas with precise lateral and vertical registration. The V groove has an important advantage for preserving the correct focus for lenses of varying diameters.

Patents and Awards S&TR November 1999

Patent issued to	Patent title, number, and date of issue	Summary of disclosure
Thomas C. Kuklo	Concentric Ring Flywheel with Hooked Ring Carbon Fiber Separator/Torque Coupler	A concentric-ring flywheel with expandable separators (which function as torque couplers) between the rings to take up the gap formed between adjacent rings due to differential expansion between different-radius rings during rotation of the flywheel. The expandable separators or
	U.S. Patent 5,924,335 July 20, 1999	torque couplers include a hooklike section at an upper end that is positioned over an inner ring and a shelflike or flange section at a lower end onto which the next adjacent outer ring is positioned. As the concentric rings are rotated, the gap formed by the differential expansion between them is partially taken up by the expandable separators or torque couplers to maintain torque and centering attachment of the concentric rings.
Peter A. Krulevitch Abraham P. Lee M. Allen Northrup William J. Benett	Microbiopsy/Precision Cutting Devices	Devices for performing tissue biopsy on a small scale (microbiopsy). By reducing the size of the biopsy tool and removing only a small amount of tissue or other material in a minimally invasive manner,
	U.S. Patent 5,928,161 July 27, 1999	these devices reduce the risk, cost, injury, and patient discomfort associated with traditional biopsy procedures. By using micromachining and precision machining capabilities, it is possible to fabricate small biopsy/cutting devices from silicon. These devices can be used in one of four ways: (1) intravascularly, (2) extravascularly, (3) by vessel puncture, and (4) externally. Additionally, the devices may be used in precision surgical cutting.

#### **Awards**

Robin Newmark and Roger Aines, together with collaborators at the University of California at Berkeley and Southern California Edison, were recently awarded the Environmental Protection Agency's Outstanding Remediation Technology Award for their work on dynamic underground stripping and hydrous pyrolysis/oxidation, technologies that heat soil and groundwater to remove contaminants and destroy them in place. (See *S&TR*, May 1998, pp. 4–11.) The award officially recognizes "technical excellence in the development of innovative in situ thermal treatment technologies."

Charles A. McDonald, Jr., associate director emeritus at-large of the Laboratory and a member the U.S. Strategic Command's Strategic Advisory Group, was recently presented the Department of Defense Distinguished Public Service Award. Given on behalf of Defense Secretary William S. Cohen, the award recognizes "exceptionally superior civilian public service." McDonald was specifically cited for his tireless efforts in developing ways to monitor the safety and reliability of the nuclear stockpile.

McDonald, who retired from the Laboratory in 1993 but still works as a participating guest and consultant, was also recognized for his leadership of the 1997–1998 Stockpile Assessment Team, a group of civilian and retired military analysts who reviewed testimony from several government agencies and provided the U.S. Strategic Command with an in-depth assessment of the nation's nuclear stockpile.

Paula Trinoskey, a health physicist in the Education and Training Division of the Hazards Control Department, was awarded emeritus status by the Board of Directors of the National Registry of Radiation Protection Technologists.

Trinoskey has served on the Panel Examiners and the Board of Directors of the National Registry of Radiation Protection Technologists. She is currently the liaison between the American Academy of Health Physics and the Board of the National Registry of Radiation Protection Technologists.

Emeritus status is awarded in recognition of outstanding contributions to the registry and has only been awarded to 22 individuals since 1976.

## Extreme Ultraviolet Lithography: Imaging the Future

Lawrence Livermore has joined forces with Sandia/California and Lawrence Berkeley national laboratories to research and develop extreme ultraviolet lithography (EUVL) for use in the semiconductor industry. Lithography is the critical technology that enables computer microchip manufacturers to place more and more features on each chip, thus increasing computer power while shrinking computer size. With current lithography techniques pushed about as far as they can go, semiconductor industries must soon decide on a standard lithography technology for producing the next generation of computer microchips. EUVL uses extreme ultraviolet light to produce microchip circuit lines smaller than 100 nanometers in width. The three laboratories are integrating the needed technologies into an engineering test stand to demonstrate how EUVL can meet industry requirements. Lawrence Livermore is leading efforts to develop the optical systems and components, thin films, masks, and submicrometer metrology needed to bring this technology into everyday use in the semiconductor industry of the future.

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#### **Handling Fluids in Microsensors**

Lawrence Livermore's Center for Microtechnology has been a leader in the design and fabrication of microelectromechanical systems (MEMS) for several years. Current work in integrated microfluidic systems is a relatively new avenue for MEMS research. These systems will handle the air and fluid samples that are taken to identify biological or chemical warfare agents or to identify the cells that cause disease. (In other work at Livermore, detectors are being developed that identify the specific pathogen or disease present in the sample.) Devices small enough to fit on a microchip of silicon, glass, or plastic will take in samples; mix them with reagents; separate out DNA, cells, or other agents in the sample; and sense the presence of those agents.

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## Coming Next Month

## Modeling the Internal Combustion Engine

Using their chemical kinetics code to model combustion processes, Livermore researchers are developing ways to increase fuel efficiency in engines while reducing exhaust emissions.

#### Also in December

- Similarities between the human and mouse genomes help us understand gene functions and inherited diseases.
- Multilayer optics reveal solar activity in greater detail.
- The spheromak offers an alternative concept for achieving magnetic fusion energy.

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