Sensor Probes Atomic-Level Interfacial Forces

A sensor that can measure the atomic forces that develop between two surfaces as they approach and contact each other has been developed at Sandia National Laboratories. The researchers say it is the first device that can measure interfacial forces over the entire range of separation at the atomic scale.

The device, called an interfacial force sensor, is a key part of the laboratory's program to achieve a fundamental understanding how solid surfaces adhere. The sensor is already providing scientific insights into how surface modification can be controlled to vary the binding energy between solid surfaces. Fracture, friction, wear, lubrication, and corrosion can also be studied at a fundamental level with this technique.

Adhesion is usually studied by forming an adhesive bond and then trying to pull it apart. The sensor provides a way to study the binding between materials by watching the adhesive bond form as the materials come into contact.

The interfacial force sensor combines aspects of atomic force microscopy (AFM) and scanning tunneling microscopy (STM). Both techniques involve scanning a sharp probe tip at atomic distances above a sample. When combined with existing imaging technology and mounted on a three-axis piezoelectric drive, the sensor lends itself to raster scanning to make an interfacial force microscope (IFM). The scientists say such a microscope will be able to form three-dimensional images for the full range of interfacial forces, both attractive and repulsive.

The sensor has a sharp tip similar to that used in an STM. The IFM's sensor, a differential-capacitance detector, consists of a capacitor plate supported by torsion bars like a seesaw. The tip is at one end of this teeter-totter. The sensor incorporates an electronic force-feedback control system that maintains the capacitor plate at its rest position.

Conventional sensors use cantileverbased sensors that gradually respond to the attractive force, but then suddenly rush together as the rate of change of the attractive force exceeds the mechanical stiffness of the cantilever. This instability makes it impossible to measure the interfacial forces at separations any closer than where this jump occurs.

The IFM sensor avoids the problem by using a feedback loop that automatically applies an electrical potential that creates a counter force. Researchers can then determine the strength of interfacial forces by

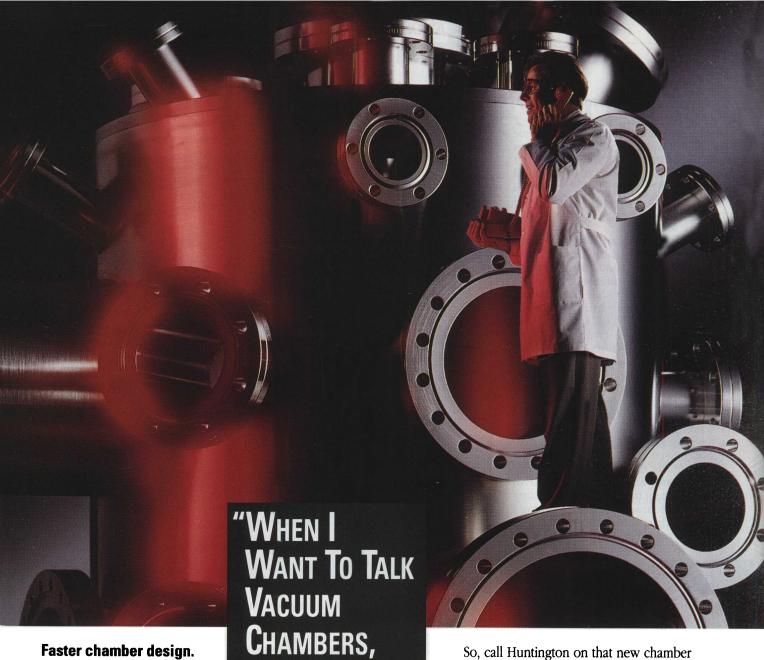
measuring the counter force needed to overcome them. The capacitor acts both as the displacement detector, through changes in capacitance, and as the counterbalance, by the application of electrostatic restoring forces. Thus, the capacitor provides a continuous measurement of the interfacial force as the sensor tip and a flat surface come together and the entire range of interfacial forces can be probed in one experiment.

The IFM sensor can detect interfacial



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Circle No. 10 on Reader Service Card. Please visit Booth No. 419 at the MRS Exhibit in San Francisco, April 28-30, 1992. forces as small as 1 fN (about the force needed to separate a sodium atom from a chlorine atom in table salt) and changes in interfacial separation of about 0.5 nm.

The researchers have collaborated with the University of New Mexico's Department of Chemistry to study the adhesive behavior between a tungsten tip, interacting with a gold surface when the surface is covered with a single layer of a lubricant, a normal alkane thiol. This molecule is a 16-carbon-atom hydrocarbon chain with a sulfur atom at one end and a methyl group at the other. The sulfur atom interacts strongly with the gold surface, and the hydrocarbon chains stand virtually erect in an ordered monolayer.

While the research group doesn't see any attractive interaction to the methyl terminal groups, they have found that when the molecules are pressed down, they recover in a second or so. This is the first such time measurement of this monolayer "self assembly" process.

The researchers also found that the normally strong plastic interaction between tungsten and gold (an unwanted "galling" interaction when the two metals are in con-

tact) is completely passivated by the organic layer, even when the layer is pressed down. This is an important insight into understanding the unique lubricating effect of the organic layer.

GM, Oak Ridge to Cooperate on Automotive R&D

Operating under a Cooperative Research and Development Agreement (CRADA), General Motors-Saginaw Division and Oak Ridge National Laboratory will cooperatively develop improved, longer-life, heat-resistant support assemblies for heat-treating furnaces that use a carburizing atmosphere. Focusing on nickel aluminide materials developed at Oak Ridge, the work will concentrate on improvements in the casting process and possible modification of the alloy to optimize its performance and improve manufacturing flexibility.

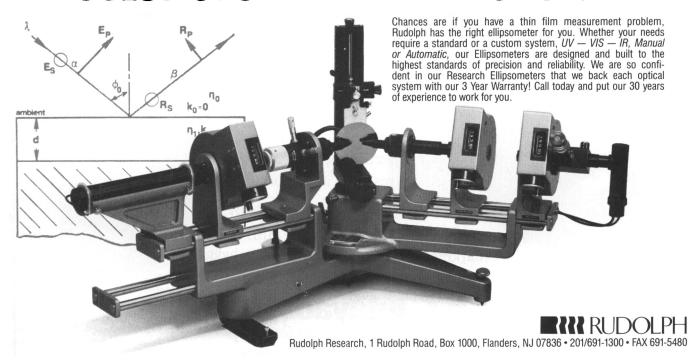
The aim is to achieve a more energyefficient manufacturing process for producing automotive parts, with an increase in throughput and a reduction in component cost due to improved tool life. General Motors will contribute \$630,000 to the two and one-half year project; the Department of Energy's Office of Industrial Technologies will contribute \$650,000.

Catalyst Reduces Toxic Byproducts in Acetaldehyde Manufacturing

A proprietary catalyst system developed by Catalytica controls the toxic chlorinated organic byproducts created in conventional acetaldehyde manufacturing technology, according to John H. Grate, who directed its invention and development. The process can be used in existing two-stage Wacker-type acetaldehyde plants with little or no modification to existing hardware and has performed successfully in a commercial-scale test, he said.

Acetaldehyde is used to make chemicals for adhesives, rubber additives, and plastics. The current Wacker process for making acetaldehyde produces substantial amounts of chlorinated organic byproducts, which are either destroyed at high cost or disposed into the environment. The new catalyst system is designed to essen-

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tially eliminate these byproducts and disposal concerns while providing acetaldehyde yields equivalent to the Wacker process.

As in the Wacker system, a palladium catalyst is used to oxidize ethylene to acetaldehyde. Instead of the copper chloride co-catalyst that produces the chlorinated byproducts, the new system uses phosphomolybdovanadate polyoxoanions.

According to its developers, the technology can also be used for oxidation of higher olefins to ketones now done by more complex, indirect routes, as for example, the oxidation of linear butenes to methyl ethyl ketone.

Magnetic Spin Observed with STM

Direct observation of the magnetic moments of individual atoms on a surface has been announced by groups working in collaboration in Switzerland and Ireland (*Science*, January 31, 1992 p. 583). Besides providing magnetism specialists with a direct view of their subject at the atomic

level, this discovery could potentially lead to the ultimate in magnetic recording—a "bit" of information encoded in the spin of electrons located on a single atom.

Carried out at the University of Basel by a group including R. Wiesendanger, I.V. Shvets, D. Bürgler, G. Tarrach, H-J. Güntherodt, and J.M.D. Coey, the scanning tunneling microscopy experiments are the first to achieve atomic resolution on a magnetic surface using a magnetic tip. In scanning tunneling microscopy, an atomically sharp tip is scanned a nanometer or two above an atomically flat surface while the tunnel current is monitored. This produces an image of the joint electronic density of states with a spatial resolution better than 0.1 nm. Images of atomic surface reconstruction and of individual molecules on the surfaces have been achieved and individual atoms have been moved with the tip to specific sites on the surface.

The new Wiesendanger-Shvets experiment uses the (100) surface of magnetite (Fe₃O₄) as the sample, and a ferromagnetic tip of iron in place of the usual tungsten or Pt-Ir tip. The (100) surface contains rows of

octahedral cation sites that accommodate equal numbers of Fe²⁺(3d⁶) and Fe³⁺(3d⁵) ions. These electronic configurations differ by the presence of a single spin-down electron. The spin-down electrons normally hop rapidly among all the available octahedral sites at a frequency in excess of 10°Hz at room temperature, giving magnetite its metallic conduction and luster. In the bulk, the electrons freeze on a superlattice below the order-disorder (Verwey) transition at 120 K.

The first surprise of the Wiesendanger-Shvets experiment was that these charge fluctuations freeze on the surface. Along the rows of octahedral sites, the researchers observed bright and dark contrast at room temperature, which is associated with *pairs* of Fe²⁺ and Fe³⁺ ions. (Figure a.)

Sometimes, rescanning the same area reveals that the picture has changed. This means that a pair of electrons has jumped, either spontaneously or under the influence of the scanning tip, to a new spot. (Figure b.)

None of this contrast along the rows is apparent if a nonmagnetic tungsten tip is

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used, which indicates a new magnetic contrast mechanism involving iron. Further experiments to rotate the relative directions of magnetization of surface and tip are now needed to establish the physical basis of the contrast.

metal. The amount of light indicates weld penetration—how much metal is being melted. A computer reads the measurement and feeds the information back to the welding machine, automatically adjusting the heat. The process was altered to work with John Deere's carbon steel parts, rather than the stainless steel used by Sandia. Researchers are adapting the system, originally designed for gas tungsten arc processes, to gas metal arc welding.





Scanning tunneling microscopy performed with a ferromagnetic tip of iron on a (100) surface of magnetite shows (a) bright and dark contrast at room temperature which is associated with pairs of Fe²⁺ and Fe³⁺ ions and (b) a change in contrast (arrow) on rescanning the same area, indicating a pair of electrons has jumped to a new spot.

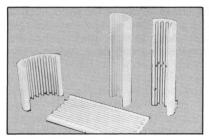
John Deere Adapts Sandia Welding Technology to Tractor Manufacturing

The John Deere Company is adapting an arc-welding feedback control system developed by Sandia for in-process inspection of welds in weapons systems to tractor parts manufacturing. Precise control during welding reduces the need for both destructive and nondestructive post-weld testing of costly components.

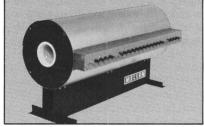
The system uses a fiber-optic cable to measure light emitted by the glowing



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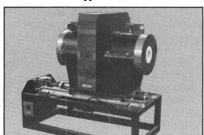
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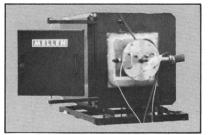
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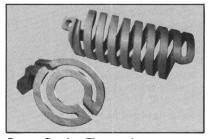
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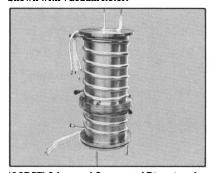
1500°C Silicon Carbide Spike Furnace on TDS-H-02 Translation Mechanism



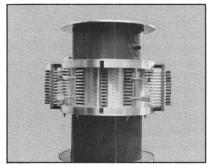
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National Academy of Engineering Elects Members, Foreign Associates

Robert M. White, president of the National Academy of Engineering, announced the election of 79 engineers to membership in the Academy and seven as foreign associates. This brings the total U.S. membership to 1,628 and the number of foreign associates to 136.

Election to the Academy is among the highest professional distinctions accorded an engineer. Academy membership honors those who have made "important contributions to engineering theory and practice, including significant contributions to the literature of engineering theory and practice," and those who have demonstrated "unusual accomplishment in new and developing fields of technology."

MRS members newly elected to the Academy include **Peter R. Bridenbaugh**, Aluminum Company of America, for contributions to the development, manufacturing, and commercialization of advanced materials; **Lance A. Davis**, Allied-Signal Inc., for personal contributions to and leadership of the development of novel amorphous and microcrystalline materials via rapid solidification technology; and **Frederick F. Lange**, University of California, Santa Barbara, for innovative contributions to the understanding of ceramic processing.

Institute of Materials in London Merges Programs of Three Founding Bodies

The Institute of Materials, formally launched at a January reception in London, will work toward merging the assets and programs of its three founding bodies, the Institute of Ceramics, the Institute of Metals, and the Plastics and Rubber Institute. A fully operational Institute of Materials is expected by January 1, 1993.

The Institute of Materials, whose Council members represent the areas of ceramics, metals, and polymers, will be governed by a Royal Charter approved by the Privy Council. With a combined membership of nearly 20,000, the Institute expects to make major contributions to the development and application of new and improved economically viable materials.

The Institute is also concerned with continuing education and the maintenance of high professional standards, particularly in industry. A wide range of technical activities and services is planned, as is accreditation recognized throughout Europe. The first president of the Institute is Sir John Collyear.

Magnetic Bearings Aid GaAs Growth

Researchers at the Institute for Boundary Layer Research and Vacuum Physics at the Research Center Jülich have developed a procedure for gallium arsenide crystal growth using an airtight crucible and drawing zone and noncontact magnetic bearings and drives that ensure jolt and vibration-free mechanical operation.

GaAs crystals are usually grown the same way as silicon crystals, via the Czochralski process in which the crucible and the crystal rotate slowly in opposite directions. However, vibrations caused by the drive motor or drawing can affect the lattice structure or growth. GaAs is even more prone to this problem than silicon.

The Jülich crucible is positioned on a vertical axle, while the crystal hangs vertically from a drawing axle. The ends of both axles consist of specially structured materials that can be rotated by magnetic forces, as with the rotor of an electromotor. The drive elements of the axles, the stators, are pow-

ered by both permanent magnets and adjustable 'electromagnets. The forces generated keep the axles precisely at the desired height and ensure they remain perfectly centered during rotation.

The housing walls must be heated to 650°C to prevent arsenic vapor condensation, so elements for internal heating and external cooling have been integrated into the interior and exterior of the walls. However, the materials used in this process still pose problems. Although the system has dispensed with seals, conduits, windows, or measuring devices susceptible to corrosion, the walls of the housing and the axles still require protection against the arsenic, extremely aggressive at this temperature.

Even so, the researchers can measure the rate of crystal growth and plan the diameter of a crystal by weighing the developing crystal and the crucible, then regulating the drawing speed. Based on their experiments, in cooperation with Leybold AG in Alzenau, the scientists believe they can manufacture GaAs crystals weighing up to 10 kg

Tests are already being conducted on the first industrial prototype of the crystal breeding apparatus. Crystals from other materials have also been grown, and progress in improving the quality of manufactured silicon crystals seems possible.

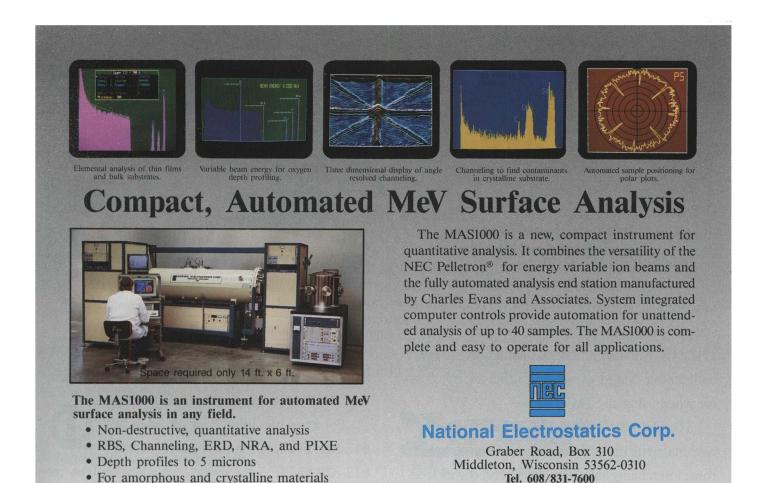
From: German Research Service, Special VII

Barium Titanate Thin Film Contract Expected to Contribute to DRAM Commercialization

Advanced Technology Materials (ATM), Danbury, Connecticut, announced Phase I and II contracts with the Strategic Defense Initiative Office (SDIO) for development of barium titanate (BaTiO₃) thin films using chemical vapor deposition (CVD) technology. The contracts total more than \$550,000.

Growing barium titanate and advanced dielectric thin films on a semiconductor substrate is considered critical for the commercialization and high-volume produc-

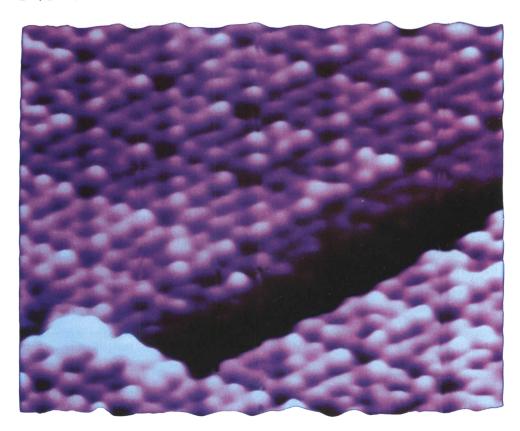
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tion of 256 Mb dynamic random access memory (DRAM) semiconductors. These chips have already served as the manufacturing technology driver for the semiconductor industry for more than two decades. In 1991 the DRAM market exceeded \$3.5 billion.

Under the contracts, ATM is developing CVD techniques to fabricate barium titanate-based thin films for high-precision/low-intensity laser applications, including frequency-doubling devices that can increase the storage capacity of CD-ROM disks and also provide the capability for highly accurate laser eye surgery. Barium titanate's nonlinear optical properties may also accelerate the development of high-speed optical computers by boosting the fabrication of integrated optical phase modulators, switches, and couplers.

Spriggs Made ACerS Distinguished Life Member

Richard M. Spriggs, the John F. McMahon Professor of Ceramic Engineering at the New York State College of Ceramics at Alfred University and director of the New York State Center for Advanced Ceramic Technology, is being inducted as a Distinguished Life Member of the American Ceramic Society (ACerS) at its 94th annual meeting April 12-16 in Minneapolis.

Spriggs, a member of ACerS for 43 years, was its president during 1984-85. He came to Alfred University in 1987 after seven years at the National Research Council of the National Academy of Sciences in Washington, DC. He earned his bachelor's degree from Pennsylvania State University and master's and doctoral degrees, all in ceramic engineering, from the University of Illinois.

S. Shah Named Murphy Professor of Engineering

Surendra P. Shah has been appointed Walter P. Murphy Professor of Civil Engineering at Northwestern University. The chair, endowed in 1942 by industrialist and philanthropist Walter P. Murphy, recognizes distinguished professors at the Robert R. McCormick School of Engineering and Applied Science.

Shah joined Northwestern's Department of Civil Engineering in 1981. He is director of the Center for Advanced Cement-Based Materials at Northwestern, a science and technology center funded by the National Science Foundation, and serves on the Nationally Coordinated Program on High-Performance Concrete and Steel and the American Concrete Institute (ACI) Council on Concrete Research.

A fellow of ACI, Shah is past chairman of ACI's Committee on Fiber Reinforced Concrete, chairman of the RILEM Committee on Fracture of Concrete, and vice chairman of the Society of Experimental Mechanics Committee on Fatigue of Concrete Structures. The receipient of many awards for his work and teaching, Shah is on the board of editors of three international journals and has published more than 300 papers and edited more than 10 books.

Shah, who has served as an MRS symposium organizer, has been a visiting professor at the Massachusetts Institute of Technology, the University of Sydney, Delft Technological Institute, and the Technical University of Denmark.

Kalonji is New Kyocera Chair Holder

Gretchen Kalonji is the new holder of the Kyocera Chair in Ceramic Engineering at the University of Washington in Seattle. A professor in the Department of Materials Science and Engineering, Kalonji is deeply involved in educational projects to enhance student learning in the University's introductory materials science course, and has developed a laboratory-based version of this course. She serves as co-principal investigator for the University's portion of the nationwide project, Engineering Schools for Excellence in Education and Leadership (ECSEL), and also as the national coordinator of outreach activities for ECSEL.

Kalonji earned her BS and PhD degrees from the Massachusetts Institute of Technology, and served on the MIT faculty prior to coming to the University of Washington. At MIT she was Norton assistant professor and held a Presidential Young Investigator award. Her current research includes rapid solidification of ceramics, atomistic computer simulation techniques in materials science, and the theory of defects in crystalline solids.



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