Materials Technologies Explored in Smithsonian Exhibition

An exhibition entitled "A Shovel Full of the Right Dirt: New Technologies for Investigating Culture," will be presented at the Experimental Gallery in the Arts and Industries Building of the Smithsonian Institution, Washington, DC, from October 1 through December 31, 1993. The exhibition will be divided into three sections. The first section will highlight a chronological development of materials and technologies, particularly those involving pigments, bead manufacture, ceramics, metals, and glass. The second section, "Ancient Craft to Modern Technology," will explore the development of these materials technologies. The third section will have individual workstations where museum visitors will be asked to investigate property-performance-processing relationships.

MRS members are invited to participate in the third section where museum visitors will be asked to investigate property-performance processing relationships at individual workstations. One of the themes to be presented in this section is environmental degradation and what can be done to stabilize corrosion processes. Limited funds are available for setting up displays or experiments. This exhibition is viewed as a pilot trial for further development. Those interested in participating should contact Dr. Pamela Vandiver, Conservation Analytical Laboratory, Smithsonian Institution, Washington, DC 20560; telephone (301) 238- 3734; fax (301) 238-3709.

Sandia Scientists Discover New Way to Remove Metallic Impurities from Silicon Wafers

Scientists at Sandia National Laboratories have discovered that microscopic cavities created within silicon provide effective traps for copper, a detrimental impurity. This finding provides a potentially superior approach to removing harmful metallic impurities from silicon wafers during the manufacture of semiconductor devices.

The usual method for ridding silicon wafers of unwanted metallic impurities is by "gettering," introducing defects in an unimportant region of the wafer where precipitates of metal-silicides can accumulate. The usual gettering process doesn't completely eliminate all the metal impurities, however, and has other disadvantages as well. Sometimes the silicides form where they aren't wanted—for instance at the gate of a transistor—and such an effect can't be reversed.

Sandia physicist Samuel M. Myers, along with colleagues Dawn M. Bishop and David M. Follstaedt, have discovered that copper impurities can be reduced to much lower levels by creating microscopic cavities beneath the silicon surface. The copper atoms migrate to the walls of these cavities where they bind to highly reactive "dangling" silicon bonds. Thus, they are removed from the device region of the silicon.

The researchers formed the cavities within the silicon by ion implantation of helium and observed them using transmission electron microscopy. They then heated the silicon, driving off the helium and leaving in its place empty nanometer-sized bubbles or voids. The voids are stable to at least 1100°C and are therefore

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compatible with most semiconductor processoring.

Copper was introduced by ion implantation, and ion-backscattering analysis was used to observe its movements to and from the cavity-containing region of the silicon during heating. The experiments with copper reveal that the dangling-bond reaction is strong enough to provide gettering substantially superior to what is currently achieved by silicide precipitation. Findings show that the binding energy for copper in the cavities is 2.2 electron volts per atom, as compared to 1.5 electron volts per atom in the copper silicide precipitates.

Myers reports that for impurity gettering in silicon technology, the microscopic cavities appear attractive for at least four reasons: their creation is convenient and well controlled, they are structurally stable to high temperatures, the binding of copper impurities to the void walls is exceptionally strong, and relatively large numbers of impurities can be accommodated (10^{16} copper atoms per square centimeter of wafer surface has been demonstrated).

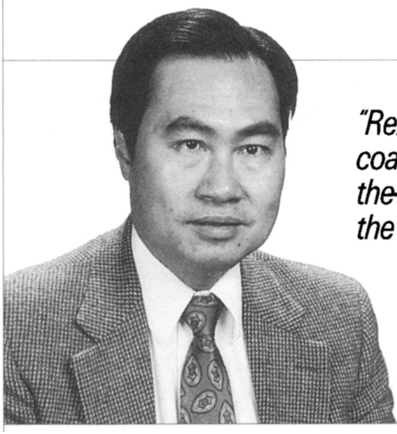
The researchers plan to work with colleagues at Sandia's Microelectronics and Photonics Center to study the same effect on prototype semiconductor wafers containing device structures. They will also extend the investigations to other metallic impurities.

LaC_2 Crystals Encapsulated in Graphite Nanoparticles

A Japanese team composed of Masato Tomita and Takayoshi Hayashi of the NTT Interdisciplinary Research Laboratory and Yahachi Saito of the Department of Electrical and Electronic Engineering, Mie University, have found a way to encapsulate LaC_2 nanocrystals within graphite nanoparticles by arc discharging composite carbon rods, as reported at the spring meeting of the Japanese Applied Physics Conference, March 1993, and in the February 15 issue of *Japanese Journal of Applied Physics.*

The positive electrode was a rod (5 mm diameter) containing 8.8 wt% La_2O_3; the negative rod (10 mm diameter) was of pure carbon. On arcing with a current of approximately 80 A in a He atmosphere of typically 50 torr, the positive electrode was preferentially consumed while producing a carbonaceous deposit on the negative electrode. The deposit contained nanoparticles comprising graphite shells around core nanocrystals averaging 10–30 nm in diameter, but also as small as 3–4 nm. Transmission electron microscopy, electron diffraction, and electron energy-loss spectroscopy have revealed that the nanocrystals are LaC_2, that the interface between the carbide and the graphite is flat, and that no intermediate layers are formed.

According to Hayashi, the present work followed earlier studies on the growth and structure of graphite tubules and polyhedral particles in an arc discharge. Hayashi and Tomita became interested in observing the C_{60} crystalline structure in the transmission electron microscope when NTT's program on intelligent materials started two years



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ago. At the time, Saito was trying to determine the growth mechanism of C_{60}, and teamed with Tomita and Hayashi by sending materials produced in his lab to NTT to be observed by high-resolution TEM. The NTT team is a competitor of Iijima of NEC, who has produced spectacular results involving carbon cylinders. It was hypothesized that it would be possible to form graphite tubules or polyhedral nanoparticles with some nanoparticle contained therewith. Also, Rodney S. Ruoff of the Molecular Physics Laboratory of SRI International, independently of Tomita, Hayashi, and Saito, published similar findings in the January 15 issue of *Science.*

Hayashi says that the main problem of encapsulating a nanoparticle in a graphite polyhedral nanoparticle (C_{60}, C_{70}, C_{82}, or higher) structure is controlling its size. Presently their work involves observing this type of structure, but not actually designing it, although they had anticipated that the structure could be formed. Empirically, lanthanum had been found to be the most easily encapsulated metal. According to Tomita, yttrium and scandium have also been encapsulated. There is no theoretical model yet for this effect, and the researchers are not speculating about applications, although they are particularly interested in the nonlinear properties of C_{60} and related materials.

F.S. Myers

Institute of Materials Honors Materials Leaders with Premier Awards

The Institute of Materials, headquarted in London, England, has announced the winners of its Premier Awards for 1993.

The Institute's premier prize, the Bessemer Gold Medal, has been awarded to **Hiroshi Saito**, president of Nippon Steel Corporation, in recognition of his services to the iron and steel industry, both in Japan and around the world.

Jack M. Buist, Abelard Management Services Ltd., has been awarded the Colwyn Medal for his pioneering work on the application of statistical methods to the analysis of test data. His achievement led to the development of new materials and new test methods, and to progress in statistical process control which paved the way toward the concept of "total quality management." Buist is also noted for his innovative work in developing polyurethane elastomers, rigid foam, and rigid foam laminates.

The Platinum Medal was presented to **David A. Melford**, a consultant to numerous United Kingdom materials organizations, for his outstanding research in metallurgy and exceptional record of service to the materials community. With Peter Duncumb, he was responsible for the design of the first scanning electron probe microanalyzer. He has also performed outstanding research on steelmaking, continuous casting, automatic inclusion counting, and magnetic stirring of molten metals.

Charles Gurney received the Griffith Silver Medal and Prize. Described as an original thinker in the world of fracture, Gurney has made significant contributions to the study of sources of weakness in glass and glass fibers and has provided a comprehensive and generalized treatment of linear and nonlinear elastic fracture mechanics.

Electrical Properties of Quarter-Micron Copper Interconnects Evaluated

The *Nikkei Weekly* newspaper in Japan reported in June that Fujitsu Ltd. has succeeded in making a prototype ultrathin copper wire needed for next-generation large-scale integrated circuits (LSIs). The wire, which is made in two layers, has a minimum width of 0.25 micron, roughly half that of existing wires used for LSIs. Fujitsu has found that its new prototype wire can endure the high current density that can cause aluminum-alloy wires to fail.

This research was presented by N. Misawa, T. Oba, Y. Furumura, and H. Tsutikawa of Fujitsu Limited, and S. Kishii and Y. Arimoto of Fujitsu Laboratories Limited at the 10th VLSI Multi-Interconnection Conference (VMIC), June 1993. According to the researchers, in conventional aluminum interconnnection systems, the reliability is reduced as the wire size is decreased due to electromigration and stress migration. Copper, which has a higher melting temperature than aluminum, and is expected to have greater resistance to electromigration and lower electrical resistivity, has been widely developed over the last several years. Assuming that an aluminum alloy interconnection could be replaced by copper under the same electrical design rules, the reduction in the wire thickness would be about 60% due to the lower resistivity of the copper, the researchers said.

However, it is difficult to form fine copper lines using reactive ion etching (RIE), because halide copper has a low vapor pressure near room temperature. Thus, other techniques are needed to optimize properties.

The Fujitsu researchers therefore evaluated the electrical performance of quarter-micron copper interconnections prepared by two kinds of self-aligned wiring methods, without using RIE. One method was a self-aligned wiring technique called the damascene method. Wiring grooves were patterned on SiO_2, and then covered with TiN deposited by chemical vapor despoition (CVD). Blanket CVD-Cu was deposited on the TiN, then the upper part of the copper and TiN was removed by chemical-mechanical polishing to form the planarized wire. The TiN was used as a diffusion barrier for the copper, and to promote adhesion. The second method was the selective CVD-Cu method. In this case, TiN films were deposited on SiO_2 and patterned into fine wires. Then the copper was selectively deposited on the TiN.

A 0.3 μm copper interconnection prepared by selective CVD showed high resistance to electromigration in high-current-density (3.0×10^6 to 2.5×10^7 A/cm^2) electromigration tests. Planarized copper interconnections and vias formed by the damascene method showed structural advantages for multilevel wiring. The film structure and resistivity of CVD-Cu were also investigated by x-ray diffraction and low-temperature electrical measurements. The copper grains grew after annealing, resulting in a lower resistivity of 1.88 μΩ cm after annealing. This suggests a reduction in the electron scattering probability at the grain boundaries. The grain size of CVD-Cu was 472 Å for an as-deposited sample and 582 Å for a 500°C annealed one.

F.S. Myers

"Mattress" of Liquid Cushions Collision of Molecular and Atomic Clusters

In a report published in the May 28 issue of *Science*, physicists at the Georgia Institute of Technology have found that a thin coating of liquid on a solid surface can act like a nanometer-scale "mattress," cushioning the forces caused by the impact of atomic and molecular clusters.

The liquid's density controls how the energy is dispersed, thus determining whether the crystalline nanostructures make a "soft landing" without changing the surface structure or if the liquid melts and "freezes" into a glassy form. This technique could produce new nanostructured surface coatings made from crystals 10 to 100 Å in size.

According to Uzi Landman, professor of physics at the Georgia Institute of Technology, "We believe this work explores some very fundamental processes of energy deposition and redistribution in collisions of complex bodies with liquids. The work has technological ramifications in the growth of nanostructured materials as coatings."

Previous work by Charles Cleveland (Georgia Tech) and Landman showed that when atomic or molecular clusters crashed into a surface, they created a "nanoshock" which fragmented and destroyed their crystalline structure. Using the soft landing technique, H-P. Cheng (Georgia Tech) and Landman wanted to see if clusters could be deposited on a surface without damage.

This could allow materials engineers to build up coatings composed of independent clusters that retain their own properties—rather than the amorphous crystalline structure created by existing coating techniques.

Initially, the researchers had used molecular dynamics simulations to explore salt clusters fired into a liquid argon film. The inert gas did not cushion the impact as they had expected. The heavy liquid acted like a hard mattress and a large amount of energy stayed in the particle. The high-temperature cluster was quickly cooled by the surrounding argon, and formed a glassy material instead of a crystal. Because the material completely bypassed the crystalline state, the researchers were led to a new idea for depositing nanoglasses.

The researchers then tried simulating salt clusters crashing into a liquid neon surface. Because Ne is less dense than argon, the liquid efficiently transferred the energy away from the cluster, allowing a "soft landing," without damage.

Recent simulations have been conducted using copper clusters. Because the clusters are heavier than salt, they make a soft landing on argon and melt when crashed into heavier xenon.

Landman believes the cluster impacts could be used to form new types of coatings—with special properties such as improved surface toughness and wear resistance—while preserving the properties of the underlying surface. This new technique could also provide a new way to prepare samples for tip-based analytical tools such as scanning tunneling and atomic force microscopies.

Six Argonne Scientists Receive Awards

Six scientists have been awarded Argonne National Laboratory's highest honor for scientific achievement. University of Chicago Distinguished Service Awards recognize the achievements or leadership of Argonne professional staff, technicians, and others engaged in scientific and technical activities. The award for each recipient includes a certificate, a medal, and a check for $3,000.

Winners include:

Joseph Berkowitz, honored for his work which has had significant impact on chemical physics and physical chemistry. His imaginative choices of experimental techniques and interpretation of data have led to new insights into chemical bonding. Berkowitz has also broadened the understanding of chemical structure and of the thermodynamic stability of important substances. His work has focused on combustion, silicon chip production, and interstellar chemistry.

David J. Hill, noted for his innovative approach to developing the probabilistic risk assessment for Argonne's Integral Fast Reactor (IFR) prototype. The IFR is designed to be passively safe against meltdown-type accidents, to burn much of its own long-lived atomic wastes, and to use a metallic alloy fuel. Hill's risk assessment incorporated the IFR's passive safety features—a first for any operating nuclear plant.

Dieter M. Gruen, Michael J. Pellin, and **Charles E. Young,** honored for their work on the fundamentals of surface science and creating a specialized instrument, SARISA (surface analysis by resonance ionization of sputtered atoms), which can detect and analyze traces of materials on a surface to 10 parts per trillion. SARISA uses an ion beam to sputter away layers of materials a few atoms thick. A laser beam then processes the sputtered atoms for analysis.

H. Péter Planchon, lauded for his significant achievements in testing and design in Argonne's Integral Fast Reactor program. Planchon directed tests which demonstrated the reactor concept's inherent safety. His work continues in the area of spent fuel handling, and refining techniques for predicting and monitoring core reactivity.

EUREKA Project Awarded to Four Companies to Develop Photon Counting Camera

A new research project entitled PHOBIA (Photon Counting Cameras for Bio-Luminescence and Auto Radiography), aims to develop two new types of biomedical instruments based on photon counting detectors.

Financially supported by both the Dutch and British Ministries of the European Council of Ministers, the research consortium consists of Rutherford Appleton Laboratory, Hi-Light Opto Electronics BV, Photek Limited, and IBH Consultants Limited.

Rutherford Appleton Laboratory will provide systems know-how and software in developing the cameras, which have an intrinsic resolution of better than 2,048 X 2,048 pixels for a wide range of scientific uses. Hi-Light Opto Electronics BV will design and develop intelligent miniaturized power supplies, microprocessor circuits, and fast gate pulse circuits to extend the dynamic range of these cameras. Photek will manufacture the primary detector and will be responsible for total system integration. IBH Consultants will be developing systems with subnanosecond time resolution, but much lower spatial resolution.

DOE Announces Three New Environmental Projects

The Department of Energy (DOE) has announced that three environmental projects will be conducted at or considered for the Idaho National Engineering Laboratory (INEL).

The first program, a feasibility study for the treatment of mixed waste, will begin immediately to explore the potential for using INEL facilities to demonstrate technology for characterizing and treating mixed waste. DOE is allocating almost $1 million from its environmental restoration and waste management funds for 1993 to support this project.

An advanced robotics program will develop technology for handling, characterizing, and treating waste. INEL is one of two finalists in DOE's site selection for this $5 million per year program.

The third program focuses on developing ways to improve the safety of storing high-level spent nuclear fuel on the INEL site. Approximately $3 million of the 1994 budget is earmarked for the development of dry storage systems and $2.7 million for handling special fuels.

Jonas Named Director of the Beckman Institute

Pioneering researcher Jiri Jonas has been appointed the new director of the Beckman Institute for Advanced Science and Technology at the University of Illinois at Urbana-Champaign.

Jonas, the director of the university's School of Chemical Sciences, was selected for the post following a national search for a director to succeed retiring

Theodore L. Brown. Known for his groundbreaking studies of liquids under extreme conditions at high pressure and high temperature, Jonas has been a member of the University of Illinois chemistry department since 1966.

The largest interdisciplinary research institute among U.S. universities, the Beckman Institute is home to more than 550 researchers in engineering and in the physical, behavioral, and life sciences; it encourages research in an environment that overcomes many limitations inherent in traditional department structures.

Born in Prague, Czechoslovakia, Jonas earned his doctorate from the Czechoslovak Academy of Science, Prague, in 1960. He was a research fellow at the Academy's Institute of Organic Chemistry before joining the University of Illinois in 1963 as a visiting scientist. He became a full professor at Illinois in 1972.

Jonas is the author of more than 250 papers covering a range of interests in the fields of biochemistry, chemical physics, and solid-state materials. He pioneered the use of nuclear magnetic resonance (NMR) and Raman laser spectroscopy in the study of the behavior of liquids under high pressure and high temperatures.

Rustum Roy Inducted into ASEE Hall of Fame

Rustum Roy, Evan Pugh Professor of the Solid State, and Professor of Science, Technology, and Society, was one of 18 individuals inducted into the Hall of Fame of the American Society for Engineering Education (ASEE) at the Society's Centennial Meeting in Champaign, Illinois.

Honored for his career-long championing of interdisciplinary education in America, Roy was cited with many accomplishments. A major figure in the creation of the national field of interdisciplinary research by organizing national meetings and analyses of the field, he was directly involved in the development of the Materials Research Society and the Materials Education Council. Besides being the founding director of Pennsylvania State University's Materials Research Laboratory, he has played a major role in establishing geochemistry and initiating solid-state science as academic disciplines. He has also played the same role in creating and directing the Science, Technology, and Society Program.

A materials scientist specializing in the design, synthesis, and preparation of new materials, Roy has authored more than 500 papers and several books. Major research contributions include invention of the sol-gel route to making ceramics, development of the hydrothermal method and its application to oxide systems, synthesis of new magnetic and ultralow expansion ceramics, and innovative radioactive waste solidification.

Roy has been involved in science and policy matters for three decades. He has served three governors as a member of the Pennsylvania Governor's Science Advisory Committee and established its Materials Advisory Panel, and helped initiate the first state-level technology funding program. At the federal level, he has served on a number of boards, committees, and panels of the NAS-NRC-NAE, the Department of Defense, and the National Science Foundation. He was particularly active in three areas: the National Materials Advisory Board, the Radioactive Waste Management Committee, and the USSR-Eastern Europe Exchange Committee.

Sandia Reports Electrically Injected Visible Surface-Emitting Lasers

Researchers at Sandia National Laboratories have produced the first electrically injected visible-light vertical-cavity surface-emitting lasers (VCSELs). The achievement is reported in the May 13 *Electronics Letters* by Sandia researchers Richard P. Schneider Jr. and James A. Lott. Adelbert Owyoung, manager of Sandia's Photonics Programs Department, said, "It's the first laser of its type to produce visible light." The first uses, he said, might be in plastic fiber communications and optical printing.

The lasers use an optical cavity active region composed of thin layers of indium aluminum gallium phosphide (InAlGaP). Vertical-cavity surface-emitting lasers emit light directly out of the surface of the semiconductor materials of which they are composed. They have many fundamental advantages over conventional semiconductor lasers, which emit sideways from a cleaved edge. Emission perpendicular to the surface makes it easier to fashion closely packed arrays of lasers. VCSELs are also well known for their tight, circular beam. These advantages make them useful for applications such as optical interconnects, fiber optical communications, and laser printing.

The new lasers emit light in the bright red (wavelengths from 639 to 661 nm) part of the visible light spectrum. Visible VCSELS that can operate at these wavelengths offer the potential for still more applications—laser pointers (which now use edge-emitting semiconductor lasers and don't have a well-defined circular beam), plastic fiber communications, arrays for displays and holographic memories, telemetry, and as a replacment for helium-neon lasers in bar-code scanning applications.

The shortest wavelength previously reported for a VCSEL diode is 699 nm, just at or beyond the edge of the visible light spectrum. Schneider said that the 639 nm wavelength reported by Sandia is getting close to that produced by the lasers used in bar-code scanners. The cumbersome helium-neon tube-type lasers used in supermarket checkout counters emit at 633 nm.

The laser, grown by metalorganic vapor phase epitaxy, consists of an InAlGaP-strained quantum well active optical cavity and AlInP phase-matching layers sandwiched between distributed Bragg reflectors (DBRs) made of AlAs/AlGaAs (55 periods on the bottom, 36 periods on the top). The cavity design was optimized on the basis of the performance of edge-emitting laser diodes with AlGaAs DBR cladding to simulate injection conditions present in a VCSEL. An invention disclosure has been filed.

At room temperature, the laser operates in a pulsed mode. Peak power occurs at 650 nm, exceeding 3.3 mW. At that wavelength, the threshold current was 30 mA, the threshold voltage 2.7 V, and the output beam 20 μm in diameter. The devices themselves are 10 to 30 μm. □