

Evaluation of EHS Issues Should be High Priority

To the Editor:

A ranking of desired low-*k* materials characteristics is summarized in the October 1997 *MRS Bulletin*, Table III, p. 22. The very lowest ranking of #15 was given to the "Environmental, health and safety (EHS)" category, which suggests that this area is the industry's lowest priority, potentially to be sacrificed for technological advancement and profit. I hope

this ranking results from an inadvertent oversight in deference to a technical emphasis on desired performance features of low-*k* materials.

The expert advice of industrial hygienists, toxicologists, and environmental engineers and scientists is critical to the safe utilization of novel organic polymers and other materials in the semiconductor industry. As research and development engineers and scientists, if we fail to place the highest priority on the competent eval-

uation of EHS issues, we risk the health of industry workers and the public, as well as create the potential for costly legal vulnerabilities for the industry. While defining the "safety" of an individual chemical is not our area of expertise, we should first defer to trained professionals competent to both assess risks and determine if and how such chemicals may be safely handled and used in a fab environment.

Peter B. Merkle, Ph.D.
Sandia National Laboratories

RESEARCH/RESEARCHERS

Shape Changes in Ceramic Particles: A Paradox Explained

A long-standing paradox in the theory of sintering ceramics has been resolved by Alan W. Searcy of the Ernest Orlando Lawrence Berkeley National Laboratory, Jeffrey Bullard of the University of Illinois, and W. Craig Carter of the National Institute of Standards and Technology. In sintering, fine-powder compacts are heated to temperatures only a little lower than their melting points; atoms and molecules are set in rapid motion, and the particles coalesce, reducing porosity and increasing the strength of the finished product.

The "classical" approach to sintering assumes that the initial particles in the compact are spheres and that movements of atoms or molecules are driven by differences in curvature. In order to reduce surface-free energy, atoms supposedly move from particles of smaller radius to particles of larger radius; the concave regions formed by contact between particles are filled by atoms from convex surfaces.

However, the molecules of crystalline particles sometimes move in directions the theory forbids which, according to Searcy, is not surprising, "because the classical theory fails what I think of as the 'single-particle sintering test.' It predicts that an isolated particle of arbitrary shape will evolve into a sphere. On the contrary, most single crystals, if grown slowly enough, are faceted." Unlike an idealized sphere, the surface energies of a crystal depend on the different orientation of the surfaces to the underlying crystal-lattice structure.

"So here's a theory that says you can't get sintering with particles that have planar surfaces and edges," Searcy said, "while in fact many solids, such as magnesium oxide, cobalt oxide, sodium chloride,

and lithium fluoride, keep their faceted shapes—or even grow into faceted shapes—while they are being sintered."

Josiah Willard Gibbs, the nineteenth-century founder of chemical thermodynamics, had reported that molecules at the edges between crystalline facets would leave and return to their crystal sites more often than molecules in the facet surfaces because the edge molecules are less strongly bonded. Inspired by what he calls Gibbs's "qualitative description of dynamic equilibrium," Searcy worked with Bullard and Carter to develop equations that explain shape changes during sintering in terms of energy differences among differently oriented surfaces and edges—"rate equations" founded on Searcy's "statistical thermodynamic description of the unstable internal equilibrium" in crystals of any shape.

The new equations are based on two governing principles: First, any change in shape is possible if it reduces total energy—the change need not minimize energy, merely reduce it. Second, among all possible shape changes, the one that actually occurs is the one in which the exchange of atoms or molecules and vacant crystal lattice sites is easiest—the one most favored kinetically.

The new equations show that particles do not grow faster because they are curved; instead the apparent curves are produced by the growth of new crystal layers. "Rounded" edges which appear during sintering are actually small additional facets—kinetically favored to grow because it is easier to move atoms or molecules to those sites. As these small, fast-growing facets multiply, particles begin to appear classically rounded, but become faceted again in the final stages of sintering.

For example, if two cubic crystals of

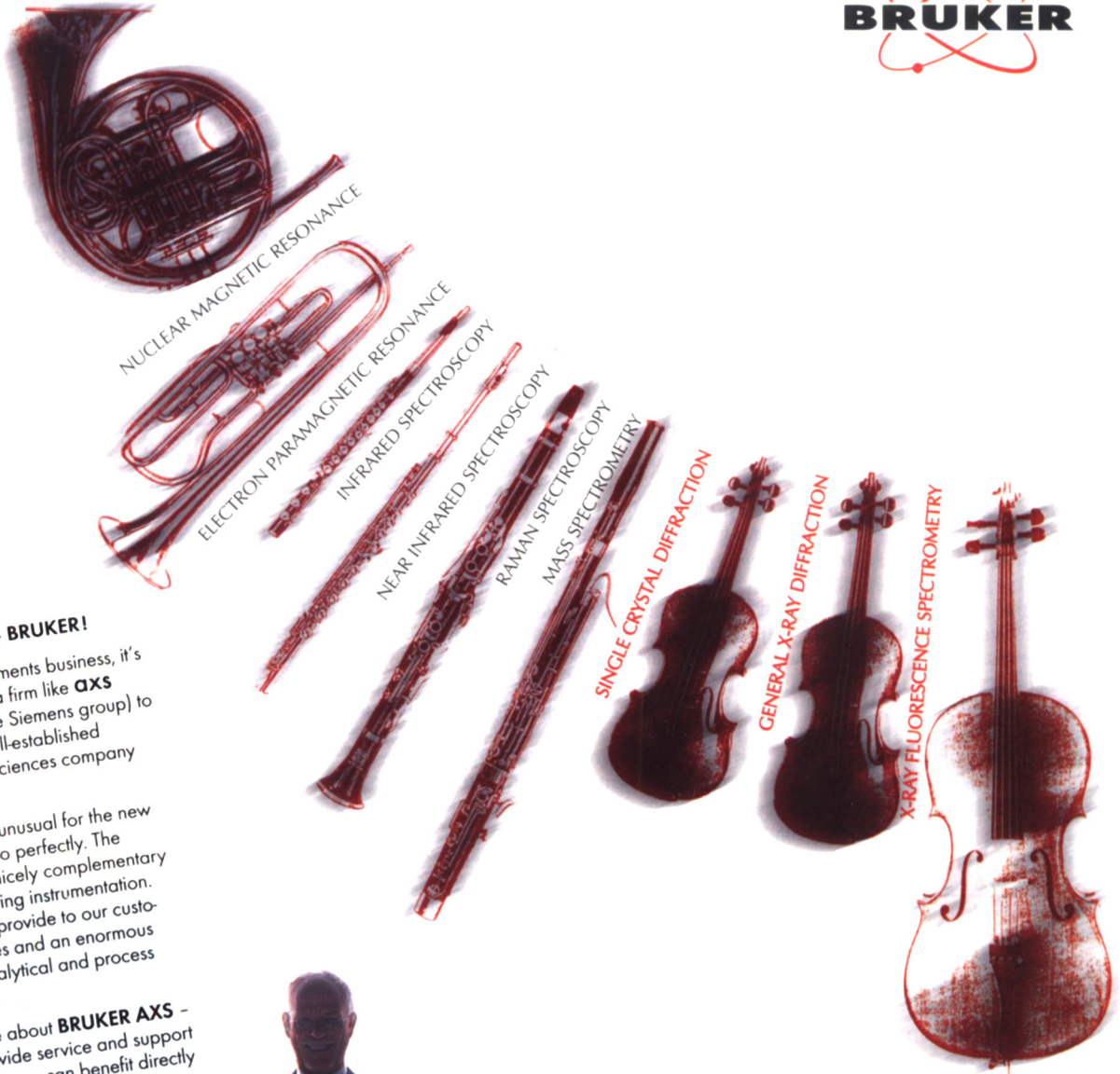
magnesium oxide are in contact with each other, the smaller rapidly transforms into a nearly spherical particle. A rounded neck forms between it and the larger particle, which is still nearly cubic—producing a sort of lopsided dumbbell. Given enough time, a single large cube with slightly rounded edges will form. At every step the changes serve to balance the demands of minimum surface energy; only changes which reduce energy are allowed.

Recently Searcy, Bullard, and Carter used their model to solve a puzzle reported by researchers at Oak Ridge National Laboratory. Observed in a transmission electron microscope (TEM), box-like particles of magnesium oxide slowly grew connecting necks during over two hours of sintering—then in 20 minutes the necks collapsed and disappeared.

Three thermodynamic pathways were suggested by the new equations, one of which matched the observations exactly by taking into account the chemical reaction of the magnesium oxide particles with the supposedly unreactive carbon substrate on which they were mounted. In the vacuum chamber of the TEM, carbon and magnesium oxide react to release carbon monoxide and magnesium vapor, reducing the overall energy of the magnesium-oxygen-carbon system. Searcy and Bullard also report confirmation of a prediction of the new model in the September 1997 issue of the *Journal of the American Ceramic Society*.

Mesostructured Ceramic-Type Materials Form Using Polymer Templates

Scientists at Max-Planck Institute for Polymer Research have reported in the December 5 issue of *Science* the use of polymers as templates to structure ceramic-type



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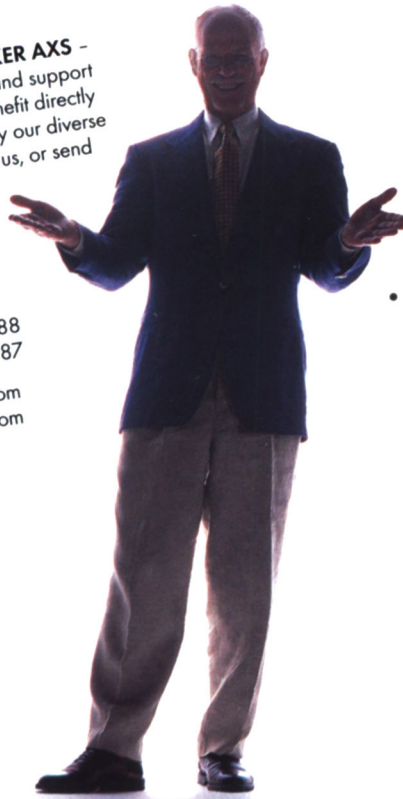
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inorganic structures formed through self-assembly. The templates are used as structure-directing agents.

Patterning synthetic inorganic materials after the morphogenesis of biological minerals, the scientists have discovered that polymeric species of molecular weights up to several tens of thousands can be employed as structure-directing agents for the synthesis of mesostructured ceramic-type materials. They used diblock copolymers, which are chain molecules consisting of two chemically distinct blocks covalently attached at their chain ends.

Ulrich Wiesner, the project leader, said, "Nature does very similar things by using e.g. proteins, which are folded structures of long chain molecules consisting of amino acids. Employing these higher molecular weight polymeric species, it now becomes possible to make the transition from the small to the large mesoscopic regime (up to several tens of nanometer) of silica-type mesostructures."

Markus Templin, PhD student and first author of the paper, said, "In addition, the combination of inorganic siliceous components with block copolymers

might lead to a desirable combination of ceramic- and polymeric-type properties not accessible so far; since the block copolymer chemistry (architecture, chain length, composition, etc.) can be varied substantially, it should be possible to fine-tune the properties of the composite."

Since the beginning of the 1990s researchers have, for example, used low molecular surfactant aggregates as templates to form structured inorganic pore networks with channel diameters of 1.5–10 nm. The Polymer Research Group has shown that by increasing the fraction of the inorganic precursors with respect to the polymer, mesostructures are obtained that exhibit the same symmetry and long-range order as phases formed by block copolymers alone. The length scale of these structures as well as the state of macroscopic alignment are varied using concepts known from the study of polymers. These results suggest a simple, easily controlled pathway for the preparation of various silica-type mesostructures. Since the inorganic material is very rigid and insoluble in organic solvents, these studies also open up the possibility to design inorganic ceramic-type struc-

tures like balls, cylinders, or lamellae on the nanoscale by dissolving the material. According to the researchers, only recently new phases in triblock copolymers including helices or other more complex forms have been discovered.

Dendrimer Supermolecule Directs Light Energy

Scientists have developed a class of large dendrimer supermolecules that funnel light energy. Raoul Kopelman, Kasimir Fajans Professor of Chemistry, Physics and Applied Physics at the University of Michigan, said, "Normally, light energy disperses randomly throughout a molecule, but these molecules have a specific tree-like structure which allows them to funnel light energy through the branches and direct it to a central point."

When photons of ultraviolet light hit a group of light-harvesting atoms on a branch of one of these supermolecules, the absorbed energy travels down the branch in the form of excitons. Losing a small amount of energy at each branching point, excitons keep falling toward the center of the molecular tree until they drop, one at a time, into a molecular



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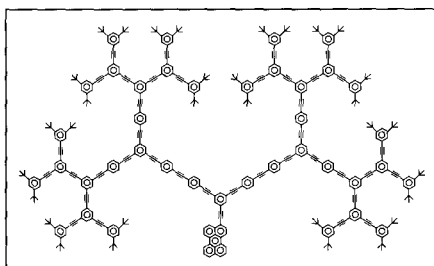
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"trap," which is attached to the dendrimer's center. In the "nanostar"—the most optimally designed version of these dendrimers to be developed so far—photosensitive molecules in the trap convert exciton energy back into visible light with up to 99% efficiency.

Stephen F. Swallen, a postdoctoral fellow in chemistry at the university, said, "It works like a miniature quantum well in a semiconducting circuit. The excitons don't have the extra energy to climb back up the molecule, so they just keep falling into the trap."



Structure of the light-harvesting nanostar supermolecule.

The synthesized form repeating molecular units called phenylacetylene monomers, which branch out from a central core, dendrimers are among the largest structurally controlled organic molecules created according to Jeffrey S. Moore, professor of chemistry at the University of Illinois at Urbana-Champaign. The largest molecule they have synthesized so far contains 127 chromophores or light-harvesting units.

Each dendrimer is custom-made by Moore and his colleagues to Kopelman's specifications to produce different chemical and physical properties for different applications. As reported in the *Journal of Physical Chemistry* **101** (1997), one of the most significant properties of the molecules is their ability to resist photobleaching. Kopelman said, "Molecules can only absorb and emit photons a limited number of times before they fall apart. Photobleaching is a particularly important factor for these dendrimers because they interact with light very strongly."

Their specific chemical composition and physical structure enable the dendrimers to resist photobleaching, according to Swallen. "While most organic molecules will decompose if multiple excitons are concentrated at the same spot, the nanostar can protect itself by diverting some excess energy away from the center back to the outer parts of the dendrimer. Because the molecule is never hit with

more energy than it can handle, it lasts much longer than ordinary molecules when exposed to light," he said.

Compressed Sulfur Found to be a Superconductor

Scientists report in the November 27 issue of *Nature* that sulfur becomes a superconductor at 93 GPa. At this pressure, pure sulfur transforms to a superconductor with a T_c of 10 K. As pressure increases, so does the superconducting temperature, at a rate of 0.06 K per GPa (up to 14 K). At a pressure of 160 GPa (the highest measured in the current experiments), T_c again increases to 17 K. In a related study, appearing in the November 1997 *Physical Review Letters*, the same scientists report the first measurements on a known superconductor, the metal niobium, above one million atmospheres (or one megabar)—to 132 GPa.

The group used a megabar high-pressure diamond-anvil cell in conjunction with a magnetic susceptibility technique: "We apply a low-frequency ($f = 22$ Hz) magnetic field with an amplitude up to several dozen oersted, which causes the destruction of the superconducting state near the superconducting transition. This in turn leads to a change in the magnetic susceptibility of the sample from -1 up to 0 (SI units) twice in a given period, and produces a modulation of the signal amplitude in the receiving coils at a frequency $2f$. The subsequently amplified signal from the lock-in amplifier is then recorded as a function of temperature on the computer. The T_c is then identified as the point where the signal goes to zero because of the disappearance of the Meissner effect."

The technique allowed Viktor Struzhkin, Russell Hemley, and Ho-kwang Mao of Carnegie Institution and Yuri Timofeev of the Russian Academy of Sciences to determine the superconducting transition temperature without the need for placing electrical leads on the sample. Thus, they could perform their measurements on very small samples (down to 0.04 of a millimeter in diameter and a few thousandths of a millimeter in thickness). Tests of the method in the megabar pressure range (above 100 GPa) were done on niobium, which has a T_c of 9.5 K at atmospheric pressure but decreases to 4.5 K at 132 GPa (rather than increases).

Sulfur's transition from insulator to superconductor at 93 GPa was unexpected. Several years ago, scientists had observed changes in optical properties of sulfur that suggested that the material transforms to a metal at about 90 GPa (at room temperature), with a corresponding

change in crystal structure, and that it transformed to another structure at about 160 GPa. Recent theoretical calculations had predicted that sulfur would become a superconductor only at much higher pressures (above 550 GPa). The new results show that the material transforms directly from an insulator to a superconductor at the first transition (at 90 GPa). The results provide an important example of the large-scale changes in physical properties that can be induced by pressure.

According to the researchers, the results are particularly notable because the metallic phases of sulfur have the highest T_c 's of any elemental solid measured to date. Sulfur now joins the heavier members of its family in the Periodic Table of the Elements (the chalcogenide family, including selenium and tellurium) as a superconductor.

Magnetic Garnet Crystal Sheet 9- μ m Thick Bonds to Semiconductor

Scientists at Columbia University and the State University of New York at Albany have sliced and bonded a sheet 9- μ m thick from a magnetic garnet crystal, a photonic material that transmits light in only one direction, to a semiconductor.

As reported in the November 3 issue of *Applied Physics Letters*, the research team fired high-energy beams of helium ions at a planar region that is just below the surface of the crystalline material, yttrium iron garnet (YIG), to loosen it from its substrate, gadolinium gallium garnet. They then applied chemicals to the region to cut the bonds entirely, slicing off an ultrathin sheet of magnetic material from a single crystal. The sample was then lifted off and bonded to a high-quality semiconductor.

Richard M. Osgood, Higgins Professor of Electrical Engineering and professor of applied physics, said that the goal of this effort is to make devices that allow light to go in only one direction on a fiber optic microchip. Light guides etched into the magnetic crystal, when exposed to a magnetic field, allow the light to travel in one direction only, making the light guide an effective routing device in an optic fiber network. Currently, optic messages travel by laser light to an isolator that prevents destabilization of the laser by outside interference, then to a modulator that imprints a signal, then to a multiplexer that combines signals of different wavelengths, each of which can carry a different message. A similar system is required at the receiving end to decode the light message into sound or picture.

Miguel Levy, senior research scientist

at Columbia University, said, "Right now, these are all very bulky devices. If you could put all these optic circuits on a chip, it would be cheaper, more efficient and sturdier, and there has been a lot of research geared toward integrating these components. Our work is an important step in this direction."

Such integration between photonics and electronics had not been possible because garnet and other magnetic crystals cannot be grown on a semiconductor substrate. Magnetic isolators cannot be made efficiently on any material other than magnetic garnets, thus the need to place garnet crystals on semiconductors, providing a bridge to an already mature technology, the researchers said.

Gschneidner and Pecharsky Receive 1997 Materials Science Award for Discovery of $Gd_5Si_2Ge_2$ Alloy for Refrigeration

Karl A. Gschneidner Jr., senior scientist and Vitalij K. Pecharsky, associate scientist, both of Ames Laboratory, have received the 1997 Materials Science Award from the U.S. Department of Energy for "Significant Implication for Department of Energy Related Technologies" in metallurgy and ceramics for their discovery of a class of materials made of a gadolinium-silicon-germanium ($Gd_5Si_2Ge_2$) alloy that is the key to magnetic refrigeration systems. The scientists discovered the class of materials when they lowered the magnetic ordering temperature of the compound gadolinium-silicon (Gd_5Si_4) by substituting germanium for silicon. The new compound, $Gd_5Si_2Ge_2$, exhibits a magnetocaloric effect about twice as large as that exhibited by gadolinium, the best-known magnetic refrigerant material for near room temperature applications. Such refrigeration systems are more energy-efficient and environmentally friendly than current gas-compression coolers. The new materials also broaden the practical applications of magnetic refrigeration and make the technology more cost effective.

Magnetic refrigeration is based on the magnetocaloric effect—the ability of some metals to heat up when they are magnetized and cool down when removed from the magnetic field. Conventional systems use liquids that are expanded and compressed to cool the air inside the refrigeration unit while drawing the warmer air outside. However, the liquids most commonly used are either chlorofluorocarbons, which have been linked to ozone depletion, or ammonia.

Because it eliminates the need to expand and compress a liquid, magnetic refrigeration consumes less electricity and

can operate at 60% efficiency, or greater. The maximum efficiency of conventional systems is about 40%, but most units run at about 25%.

The materials discovered by Gschneidner and Pecharsky are 2–10 times more effective in their cooling power than prototype alloys. With the new material, described in articles published in *Physical Review Letters* on June 9, 1997, and *Applied Physics Letters* on June 16, 1997, operating temperature can easily be changed over a wide range—from about 30 K to 290 K—without losing the magnetocaloric effect.

Newnham Receives Buessem Award

Robert E. Newnham, Alcoa Professor of Solid State Science at the Materials Research Laboratory of The Pennsylvania State University, has received the Buessem Award of the Center for Dielectric Studies on November 19, 1997 in recognition to the field of ceramic dielectrics. Founded in 1983 by the National Science Foundation, the Center for Dielectric Studies at Penn State is an Industry/University Cooperative Research Center whose members are electronic component manufacturers specializing in both fundamental and engineering aspects of capacitors, dielectrics, integrated passive components and packaging materials.

Newnham developed the concept and nomenclature for electrically active ceramic composites. He was responsible for not only the theory behind these composites, but also for their translation into "smart" materials and devices, a term he coined.

Newnham has received more than 30 honors, including the 1996 David Turnbull Lectureship from the Materials Research Society.

McDowell Receives Nadai Award

The Nadai Award of ASME International (The American Society of Mechanical Engineers) has been presented to David L. McDowell, regents' professor of mechanical engineering, Georgia Institute of Technology. He has been recognized for his outstanding contributions to the experimental study and development of constitutive equations for the rate and temperature dependent inelastic flow and damage of solids, including cyclic and

large strain phenomena, and to basic understanding and modeling of combined stress state fatigue and fracture processes. The ASME Nadai Award recognizes distinctive contributions to the field of engineering materials.

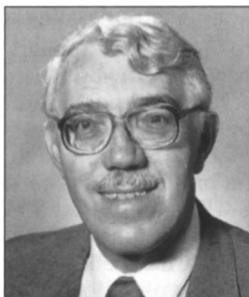
McDowell's principal contributions to mechanics of materials have revolved around the combined use of experiments and computational solid mechanics to construct appropriate evolutionary laws for inelastic deformation and damage of polycrystalline metallic materials. These contributions have been primarily influential in the subjects of multiaxial plasticity and viscoplasticity of metallic materials, multiaxial fatigue of metallic polycrystals, and high-temperature fatigue and creep-fatigue interaction.

McDowell teaches mechanical behavior of materials, continuum mechanics, nonlinear constitutive relations, fatigue and fracture. He serves as director of the Mechanical Properties Research Laboratory at Georgia Tech. As chair of the Georgia Tech Materials Council, McDowell provides leadership for the institute's strategic materials thrust, which involves over 100 faculty from a broad cross section of disciplines.

Aerogels-Based Insulator May Increase Computer Speed

At the December 10, 1997 meeting of the IEEE International Electron Devices Meeting in Washington, D.C., researchers from Texas Instruments reported that they have demonstrated the combination of copper wiring with xerogel, creating an insulator. Illustrating the importance of this type of insulator, the researchers estimated that within a decade, combining xerogel with copper wires and new designs could result in devices that are 10 times faster than current computer chips. Meanwhile, researchers in the Semiconductor Research Corporation Center for Advanced Interconnect Science and Technology (CAIST) at Rensselaer Polytechnic Institute, in November, have displayed on campus for discussion similar aerogels that they created and studied for use as insulators on computer chips. These porous materials could more than double computing speeds. CAIST, an interdisciplinary university consortium, was established by industry to improve interconnects, the minuscule system of wires and insulation that carries messages on a chip.

Good insulators—materials with a low dielectric constant—let designers place lines close together and do not slow down the signal. Silicon dioxide, the material now used on most chips, has a dielectric constant of about 4. Decreasing that number to two could at least double the speed



of computers. CAIST researchers are also studying polymers that could bring the number down to about 2.5. Air, the perfect insulator, is rated at 1.0. Since chips cannot be held together with air, Joel Plawsky, Peter Wayner Jr., and William Gill of Rensselaer's Isermann Chemical Engineering Department are studying how to solve the problem with aerogels.

Plawsky, Wayner, and Gill have created highly porous silica films that are between 65 and 90 percent air, with a dielectric constant ranging from 2.3 to 1.4. The team has shown that it can control porosity and thickness. The new films apparently do not create problems by absorbing water during processing, and they stand up well to high temperature.

Nanocomposites Take the Heat

Super plastics nanocomposites have been developed based upon compounds in which about 1-nm size particles of montmorillonite clay are dispersed throughout the polymers involved. Fire science researchers Jeffrey W. Gilman and Takashi Kashiwagi at the National Institute of Standards and Technology (NIST) have found that this class of materials is excellent in flame retardancy. The experiments conducted at NIST show that the heat-release rate—the most important parameter for predicting fire hazard—is reduced 63% in a nylon-6 clay-nanocomposite containing a clay content of only 5%. According to Gilman, the clay additive, unlike other fire-retardant additives, does not degrade the overall material.

Industry tests show the hybrid nylon-6 clay nanocomposite, compared to pure nylon-6-based plastic, has 40% higher tensile strength, 68% higher tensile modulus, 60% higher flexural strength, and 126% increased flexural modulus. Gilman and Kashiwagi also said that, unlike many chemical fire retardants, nanocomposites produce no increase in carbon monoxide or soot during combustion.

Microtransmission Increases Power of Five-Level Microengine

Steve Rodgers and Jeff Sniegowski of Sandia National Laboratories have fabricated a polysilicon microtransmission that increases the power of its microengine by a factor of 3 million. The 3 million:1 microtransmission comprises six identical transmission systems, each with two dual-level gears. The two gears, crafted one atop the other, operate at ratios of 3:1 and 4:1, which together form a 12:1 gear reduction ratio. A coupling gear allows more gear sets to be added modularly. In less than one square millimeter of

area, through use of 29 intermeshing gears, the transmission achieves a 3 million:1 gear reduction ratio. The gearing is reversible, and so can increase speed as well as decrease it. The gearing is driven by a five-level micromachine. Like its predecessors, it is powered by comb drives, but these new drives are thicker and stronger.

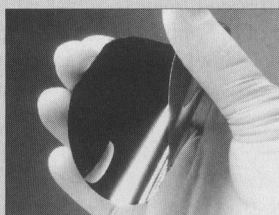
Micromachine levels refer to the tiny elevations that separate a gear or row of comb teeth in order that they may move freely. This is achieved by etching away so-called sacrificial oxide layers. The creation of additional levels permits thicker and therefore stronger comb drives. It

also allows more gears to overlap each other, compressing the amount of horizontal space needed.

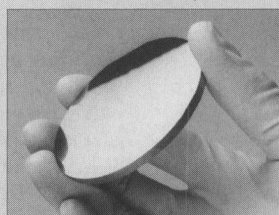
Each drive consists of two tiny comb-like structures, with teeth of one lying between the teeth of the other. By alternating tiny electric signals to the combs, they attract each other to one side and then the other. The motion is transmitted to a tiny piston-like linkage moved by one of the combs. A second comb drive provides power at right angles to the first. The piston it drives, when timed with the force of the first piston, is sufficient to turn a drive wheel on the microengine. □

"But still try—for who knows what is possible?"

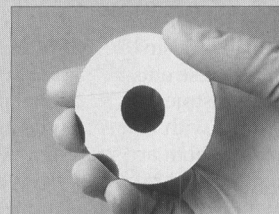
— Michael Faraday



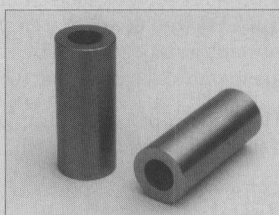
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