

RESEARCH/RESEARCHERS

From Mirror to Mist: Cracking the Secret of Fracture Instabilities

When cracks in a material propagate, bonds between atoms are broken, generating two new surfaces. Experiments have shown that cracks moving at low speeds create atomically flat, mirror-like surfaces, whereas cracks moving at higher speeds create increasingly rough fracture surfaces. Dynamical instability leads to increasing roughening transitioning from a mirror-like surface to a less reflective (mist) surface to a very rough irregularly faceted (hackle) surface. Using massively parallel large-scale atomistic simulations, Markus J. Buehler from the Massachusetts Institute of Technology (MIT) in Cambridge, Mass., and Huajian Gao from the Max Planck Institute for Metals Research in Stuttgart, Germany, (now at Brown University) have developed a new theoretical model to understand atomistic details of how cracks propagate in brittle materials, revealing the physics of dynamical fracture instabilities. As reported in the January 19 issue of *Nature* (p. 307; DOI: 10.1038/nature04408), their models indicate, in contrast to current theories, that it is critical to consider the properties of materials at large deformations, close to the crack tip, in order to understand how materials fracture. These findings have major implications for the understanding of fracture at different scales, ranging from the nanoscale to the larger scales of airplanes, buildings, or even earthquake dynamics, and predict that cracks can move at speeds faster than the velocity of sound, which has so far been considered an impenetrable barrier for crack-propagation speed.

The deformation and fracture of materials has fascinated scientists for decades, due to both their scientific relevance as well as their significance in engineering and to society. In the past, the classical physics of the continuum has been the basis for most theoretical and computational tools in engineering analyses of these phenomena, and theories relying on numerous phenomenological assumptions have been used. Scientists are now beginning to use atomistic simulation as a tool to study the behavior of materials under extreme conditions in order to gain insights about the fundamental mechanisms of deformation and failure at length and time scales unattainable by experimental measurements and which cannot be predicted using continuum theories.

This phenomenon of greater roughness for faster crack propagation is found in many different classes of brittle materials, including metals, polymers, and semiconductors, at a variety of scales. Until now, no sound understanding of the underlying physics or of the particular crack speed at which the instability occurs has been achieved. None of the existing theories explain

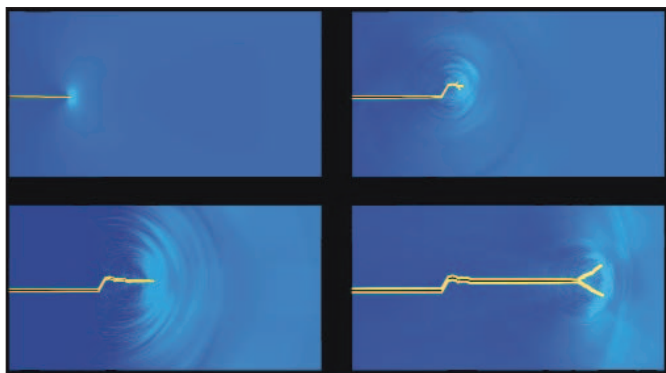


Figure 1. Atomistic simulation shows the dynamical sequence as fracture instability occurs. At a critical crack speed, straight crack motion becomes unstable and the crack starts to wiggle, creating increasingly rough surfaces. Courtesy of Markus J. Buehler, MIT.

Symposium

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Nanophotonics is rapidly developing as an integrated device technology, and is impacting applications in high speed communication systems, chemical sensing, pathogen detection, cancer research, genomics and proteomics. The significant progress in nanophotonics is related to the development of active devices whose sizes are at or below the scale of the optical wavelength. This Symposium at Caltech brings leading academic researchers in this field together with industrial scientists and investors. The day-long program will feature lectures, panel discussion and substantial opportunities for informal interactions with Symposium participants.

Speakers include:

Harry Atwater (Caltech)
Plasmonics: A Route to Nanoscale Optical Devices

Kaushik Bhattacharya (Caltech)
Ferroelectric Nanophotonic Devices

Oskar Painter (Caltech)
Photonic Crystal Devices

Axel Scherer (Caltech)
Nanophotonics and Fluidics Integration

Dwight Streit (Northrop-Grumman)

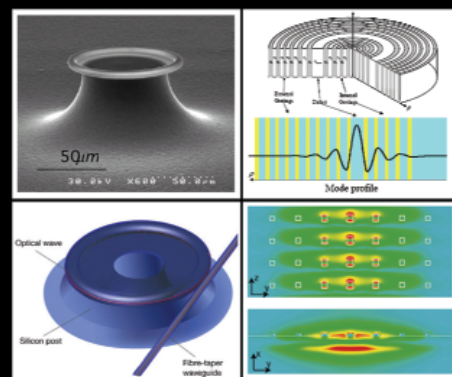
Kerry Vahala (Caltech)
Ultra High-Q Micro-Toroid Resonators

Stan Williams (Hewlett Packard)
Nanophotonics Research at HP Labs

Eli Yablonovitch (UCLA)
What is the Limit of Focusing Light?

Amnon Yariv (Caltech)
21st Century Photonics

Nai-Chang Yeh (Caltech)
Novel Organic Heterostructures for Spintronic and Optoelectronic Applications

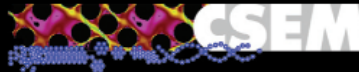


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the wide range of experimental and computational results, and many models contradict each other.

By means of large-scale atomistic simulations, Buehler and Gao show that hyperelasticity, the elasticity at large strains, can play a governing role in dynamical crack tip instabilities in the fracture of brittle materials. They report a modified instability model that treats the dynamical fracture instability as a competition between different mechanisms controlled by local energy flow and local stress field or atomic forces near the crack tip. Their results suggest that the fracture instability not only appears in materials with defects; it is an intrinsic phenomenon of dynamical fracture. See Figure 1 (p. 167).

"Our new theory reduces to existing models in limiting cases, but allows for a unified treatment of the instability problem applicable to a much wider range of materials," said Buehler. "We have discovered that the key to understanding the discrepancies in the literature is to consider the material behavior close to the breaking of bonds, rather than the material properties at small strains."

Most existing theories of fracture assume a linear elastic stress-strain law by only considering small strain deformation. However, the relation between stress and strain in real solids is strongly nonlinear due to large deformation near a moving crack tip, a phenomenon referred to as hyperelasticity or nonlinear elasticity.

The scientists have made another surprising discovery. "We find that elastic stiffening materials behavior, as found in rubber-like materials, can dramatically change the instability dynamics of cracks," Buehler said. Rubber is soft at small deformations, and becomes harder as the stretch is increased. "In such elastically stiffening materials, stable intersonic crack motion is possible," he said. These results are in contrast to existing theories, in which the speed of elastic waves is considered the limiting speed of fracture, analogous to the speed of light.

"We discovered that the classical theories of instability dynamics are only valid in a small range of material behavior," said Buehler. "In most real materials, the softening or stiffening close to bond breaking leads to a fundamental change

in the instability dynamics, because energy flow is reduced or enhanced due to change in local wave speed."

These results represent a breakthrough in understanding how cracks propagate in brittle materials, and the findings could have wide impact in many scientific and engineering disciplines, said the researchers.

Scanning Ultrasound Holography Allows Nanoscale Subsurface Imaging

The characterization of deeply buried or embedded structures and features with lateral resolution of under 100 nm is critical in a number of science and engineering arenas. Subsurface features can be imaged with techniques such as acoustic microscopy, but with limited spatial resolution. More recently, scanning probe techniques, such as ultrasonic force microscopy, have been used for nanomechanical mapping of elastic and viscoelastic properties of surfaces. However, these techniques still lack the sensitivity required for imaging buried structures. Now, Gajendra Shekhawat

Announcement and Call for Papers

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Interaction of packaging with on-chip interconnects

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Bonding and packaging issues for advanced interconnects

Abstracts are due June 2, 2006.

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and Vinayak Dravid from Northwestern University have reported the development of scanning near-field ultrasound holography (SNFUH) that allows characterization of depth information as well as spatial resolution in the 10–100-nm range. The method can be used to image buried nanostructures in diverse materials systems including microelectronic structures and biological samples. They report the technique and results in the October 7, 2005, issue of *Science* (p. 89; DOI: 10.1126/science.1117694).

SNFUH is based on scanning probe microscopy (SPM). A high-frequency acoustic wave, on the order of a megahertz or more, is applied to the bottom of a specimen, while a second wave is launched on the SPM cantilever at a slightly different frequency. The interference of these two waves forms a surface acoustic standing wave. Buried features perturb the specimen acoustic wave, and these perturbations to the phase and amplitude of the surface acoustic standing wave are monitored by the SPM cantilever. A pictorial representation of the acoustic wave perturbation is recorded

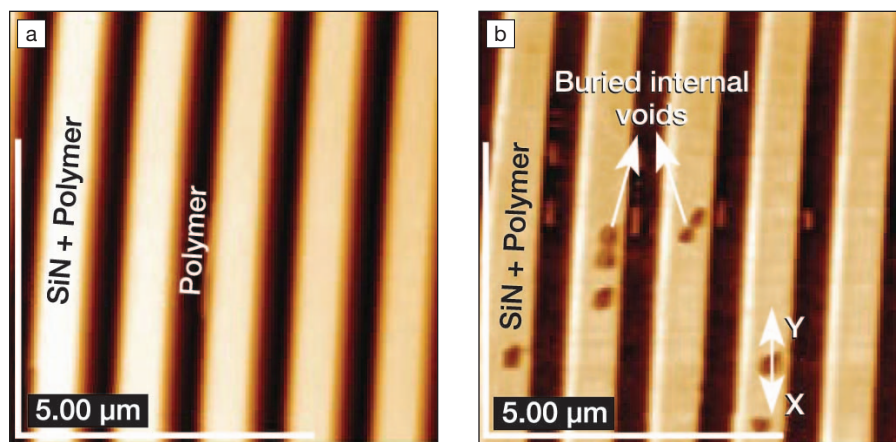


Figure 1. (a) Typical atomic force microscope topography image shows a low-dielectric polymer coating covering trenches in a model microelectronics trench structure. (b) The corresponding scanning near-field ultrasound holography phase image reveals embedded voiding in the polymer coating over the nitride. Reprinted with permission from *Science* (Oct. 7, 2005) p. 89. ©2005 AAAS.

that includes quantitative information about the internal features. The researchers used a model polymer-gold nanoparticle composite to demonstrate

the spatial resolution and depth sensitivity of the technique. The nanoparticles with an average diameter of 15 nm were buried beneath a 500-nm-thick polymer

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cover layer on a silicon substrate. While normal atomic force microscopy (AFM) showed a smooth, featureless surface of the top polymeric layer, the SNFUH phase image clearly showed the dispersed, buried gold nanoparticles.

SNFUH is particularly well suited for detecting defects and voids in microelectronics structures. For example, the scale of fabrication continues to shrink and interconnect metal lines are approaching 60-nm widths. Model shallow trench structures were fabricated in a dielectric material with a 50-nm-thick silicon nitride layer deposited as a capping layer and etched into the trenches. A 500-nm-thick low-dielectric polymer layer was deposited by spin-coating and annealing. A conventional topography scan showed a uniform and continuous polymer coating at the bottoms of the trenches as well as on the tops of the lines, as seen in Figure 1 (p. 169). However, the corresponding SNFUH phase image revealed embedded voids within the polymer and at the SiN-polymer interfaces.

The SNFUH technique can also be used to image embedded or buried sub-

structures in biological samples. A near-contact mode was used for imaging soft structures, wherein the probe tip is lifted by 2–5 nm after it touches the surface. This was used to obtain high-contrast, high-resolution images of malaria parasites inside infected red blood cells. The direct real-space *in vitro* imaging was performed without labeling or sectioning the cells under physiologically viable conditions. SNFUH was thus demonstrated to be a versatile technique for nondestructive, high-resolution, real-space imaging of diverse materials systems. In particular, it fills the spatial resolution gap at the 10–100-nm scale for nondestructive sub-surface imaging.

GOPAL RAO

Silica Nanoparticles Doped with Multiple Fluorescent Dyes Demonstrated as Potential New Barcoding Tags

Often in biological imaging experiments, it is desirable to view several components of a structure simultaneously in real time. This can be done with small-molecule fluo-

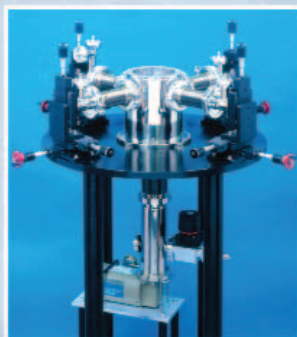
rescent tags, but this technique is limited by the number of available dyes that are excited at the same wavelength but have distinguishable emission spectra. Quantum dots, polymer microspheres, and other materials have been used to overcome this limitation and create "bio-barcoding" systems, but many of these have problems with performance. In the January 11 issue of *Nano Letters* (p. 84; DOI: 10.1021/nl052105b), researchers L. Wang and W. Tan from the University of Florida at Gainesville have presented a novel solution to this problem. The researchers incorporated combinations of three common fluorophores into silica nanoparticles (NPs) to create NPs that respond to monochromatic illumination with a range of distinguishable emission signatures. They also demonstrated the potential for using these materials as tags for biological molecules by functionalizing the NPs with biotin and binding them to avidin-functionalized microspheres.

The researchers incorporated the organic dyes FITC, R6G, and ROX into silica NPs in a variety of ratios. Alone, these dyes

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have different excitation maxima, but when incorporated together they form a tandem system for fluorescent resonant energy transfer (FRET) so that when excited at the optimal wavelength for FITC, the dye-impregnated silica NPs emit unique colors based on their dye ratios. The researchers synthesized the NPs by a modified Stöber synthesis route, in which the dyes were incorporated into the structure as the NPs grew. The NPs thus prepared were found to be uniform in size, colloidal stable, and strongly fluorescent.

To demonstrate the utility of the material, the researchers derivatized amine-modified NPs with biotin and attached them to avidin-derivatized microspheres. Under confocal fluorescent microscopy, these microsphere-NP complexes emitted light at the characteristic wavelength of the particular NP they contained. The researchers said that this approach can be generalized to label the NPs with other biopolymers for use in barcoding assays and multiplex bioanalysis. According to the researchers, the NPs can also be used as optical materials for display technologies.

KRISTA L. NIECE

Highly Ordered TiO₂ Nanotube Array Improves Dye-Sensitized Solar Cells

Dye-sensitized solar cells (DSCs) are potentially a cheap and environmentally friendly alternative to silicon-based photovoltaics. Based on an effect analogous to photosynthesis, DSCs have achieved light-to-electricity conversion efficiencies of more than 11%. In the February 8 issue of *Nano Letters* (p. 215; DOI: 10.1021/nl052099j), G.K. Mor, C.A. Grimes, and colleagues at the Pennsylvania State University have reported the fabrication of a DSC based on an ordered array of TiO₂ nanotubes. Although the photocurrent efficiency of their device is only about 3%, their results suggest that nanotube-based DSCs may achieve conversion efficiencies 10 times as high, significantly exceeding silicon-based solar cells.

Standard DSCs are typically based on a 10- μ m-thick film of randomly arranged TiO₂ nanoparticles. The nanoparticles are coated with a molecular layer of an organic dye (often extracted from blackberries, raspberries, or pomegranates) that releases electrons when illuminated with sunlight. The large surface area of the dye-coated nanoparticles enhances the efficiency of the light collection, and the released electrons become the device's photocurrent. However, the mobility of the released electrons is limited by scattering at the disordered boundaries between nanoparticles, which reduces the overall efficiency. To address this problem, the Penn State team grew

ordered arrays of TiO₂ nanotubes 360 nm in length on glass substrates by anodizing a 500-nm-thick titanium film, and then immersed the arrays in a ruthenium-based dye. When exposed to full sunlight, the resulting devices generated photocurrent with a relatively low efficiency of 2.9%. However, the lifetime of liberated electrons was significantly longer than in nanoparticle devices, implying much less electron scattering by the ordered nanotube array.

The team concluded that the conversion efficiency was limited by the relatively short length of the nanotubes, and that by improving the fabrication procedure to grow micrometer-scale nanotubes, the efficiency could be boosted to close to the ideal limit of 31%. If this proves to be the case, high-efficiency, dye-sensitized solar cells could become an important competitor to standard silicon solar cells, both in terms of lower

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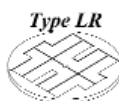
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Stage 2

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3 to 60 seconds



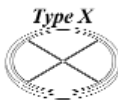
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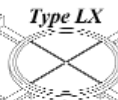
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COLIN MCCORMICK

Researchers Examine Growth and Optical Properties of Gold Nanoparticles in Stained Glass

Stained glass is best known from its extensive use in Gothic churches, although

its production dates back to ancient civilizations of Egypt and Rome. A colored "stain" is achieved when colorless metal-doped glass is heated at high temperatures. Annealing of gold-doped glass results in the formation of colloidal gold, which gives rise to a characteristic ruby-red color through surface plasmon resonance (SPR). However, tuning the SPR

Octahedral Nanocontainer Molecules Formed Spontaneously

Applications for nanocontainer molecules include stabilizing short-lived chemical species, accelerating chemical reactions, and directing regioselectivity and stereochemistry of reaction products. Supramolecular approaches to the self-assembly of nanocontainers, which involve hydrogen bonding or metal coordination chemistry, are efficient and quantitative. In contrast, nanocontainers constructed from cavitands (i.e., molecules whose constrained structure accommodates a cavity) typically require multiple steps. Recently, however, researchers from the Department of Chemistry and Chemical Biology at Rutgers University have used dynamic covalent chemistry to synthesize nanocontainers from 18 components in a single step and in high yield, greatly improving the simplicity and efficiency of nanocontainer synthesis.

As reported in *Angewandte Chemie International Edition* (DOI: 10.1002/anie.200504049), Rutgers University researcher R. Warmuth and co-researchers discovered a spontaneous formation of an octahedral nanocontainer (**B**), composed of six cavitands (**A**) linked together with 12 diamino bridges via 24 newly formed imine bonds (see Figure 1). Yields of up to 82% were achieved. The researchers used ^1H and ^{13}C nuclear magnetic resonance (NMR) spectroscopy and electrospray ionization mass spectroscopy to validate the structure of **B**. Although the researchers were unable to isolate and purify **B**, they were able to reduce all 24 imine bonds and purify the trifluoroacetate salt of the resulting polyamino nanocontainer (**C**) with reverse-phase high-pressure liquid chromatography and isolate it in an overall 63% yield. Although crystals suitable for x-ray structure determination could not be obtained, the researchers used molecular mechanics (MM) calculations to estimate the cavity volume at 1700 \AA^3 , which is large enough to encapsulate multiple guest molecules. Pulse-field gradient spin-echo NMR measurements and application of the Stokes-Einstein equation yielded a solvodynamic diameter of 3.2 nm, which is consistent with the MM model. The researchers also used MM to show that the formation of **B** requires each ethylenediamine to be in an *anti* conformation.

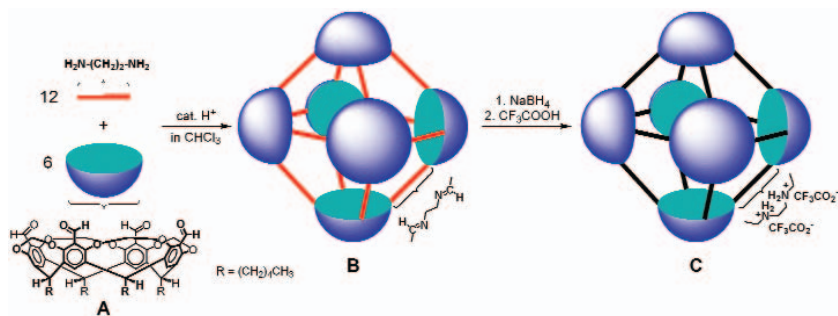


Figure 1. An octahedral nanocontainer (**B**) composed of six cavitands (**A**) linked together with 12 diamino bridges via 24 newly formed imine bonds; **B** is reduced to polyamino nanocontainer (**C**).

The researchers said, "We see potential uses for [**C**] and analogues in drug- or pesticide-delivery systems, wastewater detoxification, separation technology, and as molecular reactors for controlled oligomerizations of organic and inorganic molecules." In addition, the researchers said that "other covalent nanoassemblies with spherical or tubular shapes and different properties could be accessible through such multicomponent synthesis."

STEVEN TROHALAKI

peak in stained glass requires very long annealing times that also result in a deteriorated shape of the gold clusters. In the December 9, 2005, issue of *Angewandte Chemie International Edition* (p. 7905; DOI: 10.1002/anie.200502174), M. Eichelbaum at Humboldt-Universität zu Berlin, R. Müller of the German Federal Institute for Materials Research and Testing, and their colleagues have shown that activating the gold-doped glass with hard x-ray radiation reduces the annealing time needed for tuning the SPR peak, thereby minimizing shape degradation of the gold clusters.

Gold-doped glass was obtained by melting 0.02 mol% AuCl₃ with soda-lime silicate glass. Annealing at 590°C for 1 h produced ruby-colored glass with the SPR peak at 574 nm. Another 4 h of annealing was required to observe a shift

of 6 nm (to 580 nm) in the SPR peak, and high-resolution transmission electron microscopy images confirmed that this caused distinct deviation of particle shape from spherical symmetry. In contrast, when the gold-doped glass was activated by x-ray radiation (32 keV) for 5 min, only 30 min of annealing produced a red-colored glass with a SPR peak at 540 nm. Also, substantial changes in SPR peak positions were observed for short annealing periods: the SPR peak shifted to 547 nm after 10 min of annealing at 590°C, and to 554 nm after 15 min of annealing at 630°C. Although the shifts in SPR peaks were explained by the deviation of the gold clusters from their spherical shape, samples activated by x-rays still exhibited a sharp size distribution of gold clusters because of the shorter annealing period. Short annealing periods are due to the presence of a

very high density of gold atoms that are produced when cationic gold is reduced by x-ray radiation. X-ray near-edge spectroscopy confirmed that the x-ray-activated samples have very small quantities of Au³⁺ ions and mostly solid gold. The researchers said that x-ray-activated stained glasses are sturdy components for realizing optically tunable devices in integrated nanophotonic applications.

TUSHAR PRASAD

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News of MRS Members/Materials Researchers

Willard S. Boyle and **George E. Smith**, both formerly of Bell Laboratories, share the **Charles Stark Draper Prize**—a \$500,000 annual award that honors engineers whose accomplishments have significantly benefited society—"for the invention of the charge-coupled device (CCD), a light-sensitive component at the heart of digital cameras and other widely used imaging technologies."

Douglas B. Chrisey, formerly of the U.S. Naval Research Laboratory, has been named the Deputy Director for Research and Development at the Center for Nanoscale Science & Engineering at North Dakota State University.

Joseph A. Gardella Jr., State University of New York at Buffalo, received the 2005 *Presidential Award for Excellence in Science, Mathematics, and Engineering Mentoring*.

This presidential award recognizes individuals who have demonstrated a commitment to mentoring students and increasing the participation of minorities, women, and disabled students in science, mathematics, and engineering.

Ashok Puri, University of New Orleans, received the 2005 *Presidential Award for Excellence in Science, Mathematics, and Engineering Mentoring*. This presidential award recognizes individuals who have demonstrated a commitment to mentoring students and increasing the participation of minorities, women, and disabled students in science, mathematics and engineering.

David H. Sliney, program manager for the U.S. Army Center for Health Promotion and Preventive Medicine Laser/Optical Radiation Program, received the

24th Arthur L. Schawlow Award from the Laser Institute of America (LIA) during ICALEO 2005 (the International Congress on Applications of Lasers & Electro-Optics), which was held last October. The award is in recognition and appreciation of Sliney's extensive contributions as a pioneering leader in the field of laser safety for more than 30 years and as one of LIA's most valuable supporters.

Daniel Zajfman has been nominated as the next president of the Weizmann Institute of Science in Rehovot, Israel. In accordance with the rules of the Institute, the election of the president will be approved by the full board. Zajfman will serve as the 10th president of the Weizmann Institute, replacing the Institute's current president, Ilan Chet, whose term of office ends in December 2006.

Scientific American named its top contributors to science and technology in 2005, including:

Edward H. Sargent (University of Toronto), **Michael Grätzel** (Swiss Federal Institute of Technology), and **Tsutomu Miyasaka** and **Takurou N. Murakami** (Toin University of Yokohama) for their research in energy and to Hydrogen Solar for its business in energy;

Inez Y. Fung (University of California, Berkeley) for research in protection from the Earth's climate;

Paul W.M. Blom and **Ronald C.G. Naber** (University of Groningen and

Philips Research Eindhoven), **John Rogers** (University of Illinois at Urbana-Champaign), and **Samuel I. Stupp** (Northwestern University) for research on plastic; and

Bradley J. Nelson (Swiss Federal Institute of Technology) and the **U.K. Royal Society and the Royal Academy of Engineering** for research in nanotubes, and **James E. Jaskie** (Motorola Physical Sciences Research Laboratory, DuPont Central Research and Development, and Hewlett-Packard Laboratories) for their business contributions in the area of nanotubes.

Corrections

In the December 2005 issue of *MRS Bulletin* 30 (12) p. 965, the reference immediately following 105 was incorrectly numbered as 109; it should be 106. In the same issue, pp. 964-966, in References 17, 23-25, 30, 44, 52, 71, 73, 82, 104, 106 (corrected number), 108, 109, 111, and 112, the name T.P. Russell should have appeared instead of P.F. Green.

The Center for Nanoscale Materials at Argonne National Laboratory will begin initial operations in April 2006 and full operations in September 2007. Incorrect dates were published in the UP CLOSE article in the January 2006 issue of *MRS Bulletin* 31 (1) p. 44.