

## Transparent Solution LED Fabricated from Liquid Polymer at Room Temperature

Yang Yang and a team of researchers at the University of California—Los Angeles have developed a form of transparent polymer that they call a solution light-emitting device (SLED). The device is a sandwich of clear liquid polymer between two pieces of glass. When an electrical current is applied, the polymer molecules are excited, releasing energy in the form of light, making the transparent device glow. Yang said that although this process, called electrogenerated chemiluminescence (ECL), has been previously demonstrated in polymers in solid form, this is the first time it has been observed in liquid polymers.

As reported in the April 5 issue of *Applied Physics Letters*, the construction of a SLED can be done in a few minutes at room temperature. Two thin layers of glass are treated with a transparent coating of indium tin-oxide, a conductor of electrical current. A few drops of transparent polymer solution—consisting of blue polymer poly[9,9-bis(3,6-dioxahexyl)-fluorene-2,7-diyl] (BDOH-PF)—are placed on top of one piece of glass and the second layer of glass is added on top. The two pieces of glass are pressed together so that the fluid spreads and acts as an adhesive. Excess fluid dries along the edges to form a seal. Yang intends to seal the edges with an epoxy that would inhibit evaporation of the polymer solution. Currently, the glow lasts 30 minutes.

When unbiased, the SLED is transpar-



Transparent solution light-emitting device (SLED). (a) An unbiased transparent SLED; (b) a biased transparent SLED.

ent as glass; on biasing, the brightness can reach 100 cd/m<sup>2</sup> under 10 V (see figure). The researchers said that the SLEDs' ability to alternate between a transparent appearance and a glowing one makes them ideal for complex visuals when layered one on top of another and then superimposed on other surfaces.

## Extremely Low Metal Condensation Coefficients Determined on Low-*k* Polymers

By using a novel very sensitive radio-tracer technique, researchers at the Faculty of Engineering of the University of Kiel in Germany have determined condensation coefficients of silver, representative of low-reactivity metals, on several polymers. Axel Thran, Michael Kiene, Vladimir Zaporozhchenko, and Franz Faupel present their quantitative study on metal condensation on organic materials in the March 1 issue of *Physical Review Letters*. The scientists said that the results provide information related to the early nucleation and growth processes, which are important in determining interface structure and properties.

The condensation coefficient is defined as the ratio of the number of adsorbed atoms to the total number of metal atoms arriving at the surface. While at room temperature the silver condensation coefficient turned out to be close to unity on polyimide, a polymer used extensively in microelectronics, values as low as 0.002 were observed for Teflon AF™.

"This means that only two out of every thousand atoms impinging on the virgin surface stick to this polymer, which is a potential low-dielectric constant material for future microelectronic devices. To the best of our knowledge, condensation coefficients of metals at room temperature as low as this have not been measured previously on any material," said Faupel, who holds the Chair for Multi-component Materials in Kiel. According to the researchers, the extreme variation in the condensation behavior upon different polymers appears to be related to the polymer surface energy and suggests a connection between macroscopic wetting and atomic condensation. At elevated temperatures the condensation coefficient decreases strongly on all polymers.

Based on the temperature and deposition-rate dependence and the angular distribution of re-emitted atoms, the researchers were able to show that the re-emitted atoms have no memory of the deposition process and perform random walk prior to re-emission. Nucleation was found to take place at preferred sites.

"The nature of these sites is not known yet; one can think of terminal groups on the polymer chains, impurities, or attractive local arrangements of the chains," Faupel said.

The density of these sites, and thus the condensation coefficient, turned out to increase substantially upon ion beam treatment. At sufficiently high metal coverages the condensation coefficient approaches unity, as expected for metal-on-metal condensation.

Faupel said, "Apart from its potential technological applicability, our study provides the basis for getting quantitative results on metal-polymer interfaces with surface analytical tools where metal coverages are generally determined indirectly, for instance by the use of quartz-crystal monitors."

## Dopant Interdiffusion Limits Device Stability

As development of semiconductor electronics moves toward smaller and smaller device dimensions, researchers consider the impact of various criteria such as minimum voltage and minimum time for switching when estimating the lifetime of the product. However, researchers from Weizmann Institute of Science in Israel have noted that the limiting factor of the maximum distance over which dopants may move should also be considered. They said that dopant interdiffusion in (Al,Ga)As light-emitting diode devices, for example, has been shown to cause long-term device instability.

In their article published in the March 1999 issue of *Electrochemical and Solid-State Letters*, Igor Lubomirsky and David Cahen of the Department of Materials and Interfaces in Weizmann Institute determined that consideration of the minimum energy density with the minimum time for

## SBIR Update

**Symyx Technologies, Inc.** (Santa Clara, California) has been awarded a Department of Energy Phase II Small Business Innovative Research grant toward accelerating the search for novel anode materials for use in a direct methanol fuel cell.

**Materials Resources International (MRI)** (North Wales, Pennsylvania) has been selected by the Ballistic Missile Defense Organization to receive a Phase I SBIR grant to demonstrate its ceramic/metal joining technology for joining metal matrix and ceramic composites.

switching can be used to estimate the "chemical kinetic stability of semiconductor devices." They reported that junction degradation due to dopant interdiffusion can be estimated by using the formula  $D = d^2/t$  in which  $D$  is the ion's chemical diffusion coefficient,  $d$  is the space-charge layer width, and  $t$  is time. They further determined that the highest possible diffusion coefficient that assures stability over a three year time period is when  $D \leq 10^{-22}$  cm<sup>2</sup>/s. For example, they said, common dopants in Si-based devices such as boron or phosphorus are estimated to be below  $10^{-25}$  cm<sup>2</sup>/s at and near room temperature, which means that such devices are not limited by dopant interdiffusion. However, room-temperature diffusion coefficients of most common dopants in binary compound semiconductors (III-V and II-VI) are estimated to be  $10^{-23}$  cm<sup>2</sup>/s or higher, requiring that dopant interdiffusion be slowed down.

In studying the effects of forward and reverse bias, interstitial-substitutional diffusion, and partial diffusion, Lubomirsky and Cahen plotted their results in tables published in the article and derived their formula. They verified their procedure against the results of previous experiments on III-V semiconductors doped with Be, showing a degradation time of only a few months. The researchers suggest that in this case diffusion retardation by use of multiple quantum well, layered structures, and/or replacement of Be as dopant by C<sup>35</sup> are worthwhile avenues to pursue.



### Cherry A. Murray Named to National Academy of Sciences

Cherry A. Murray, director of the Physical Research Laboratory at Bell Labs and a member of the Materials Research Society, is one of 60 new members elected to the National Academy of Sciences (NAS) in recognition of her distinguished and continuing achievements in original research. NAS made the announcement on April 27.

Murray began her career at Bell Labs as a member of the technical staff in 1978 after receiving her PhD degree in physics from the Massachusetts Institute of Technology. In 1987, she became head of the Solid State and Low Temperature Physics Research Department. Several years later she assumed leadership of the Condensed Matter Physics Department and later led the Semiconductor Physics Research Department. Most recently she

has been studying colloids, particles that neither dissolve nor settle while suspended in a liquid, that may have applications in advanced optics and optoelectronics.

Election to membership in the Academy is considered one of the highest honors that can be accorded a U.S. scientist or engineer. The total number of active members is now 1,825.



### Sōmiya Receives Japan Academy Prize

Shigeyuki Sōmiya of Teikyo University of Science and Technology in Japan has received the Japan Academy Prize in June for "Studies on

Hydrothermal reactions and Their Applications for the Synthesis of Inorganic Materials." The Japan Academy Prize is the highest prize in the academic field. Ten prizes are given annually, five in liberal arts and five in science. Sōmiya is the only recipient this year in the field of engineering.

Sōmiya is associate editor in Japan of the *Journal of Materials Research*, a publication of the Materials Research Society, and a member of MRS. He is also chair of the Awards Committee of the International Union of Materials Research Societies.



### Philibert Awarded 1999 Acta Metallurgica Gold Medal

The 1999 Acta Metallurgica Gold Medal, established to recognize outstanding ability and leadership in materials research, has been awarded to Jean Philibert of France. During his early research studies, Philibert was entrusted with the management of the first electron microprobe, a prototype electron probe microanalyzer. He has developed many original applications in several fields of metallurgy—including solidification, dendritic segregation, nonmetallic inclusions, precipitation, interdiffusion, oxidation, and grain boundary segregation—and he presented the first metallurgical applications of EPMA in 1957 at the AIME meeting in New Orleans. He was simultaneously involved in the basic problems of quantitative analysis and derived a useful and well-known formula for the absorption correction. In research collaborations, he later developed extensions to thin foils and extraction replicas. With other collaborators, Philibert developed applications of

cathodoluminescence to the study of non-metallic inclusions in steels, and of backscattered electrons to the imaging of magnetic domains.

In 1969, Philibert was appointed associate professor at the University of Paris-Sud in order to organize new degrees in engineering education with a specific curriculum in materials science and engineering. Simultaneously, within the French Center for Scientific Research (CNRS), he launched a laboratory in Bellevue under the heading Physics of Materials in order to apply the methods and approaches of physical metallurgy to nonmetallic materials. In 1985 he became director of the Structural Metallurgy Laboratory.

He is the author of over 200 scientific papers and has been an invited speaker at numerous international conferences, mainly in the area of solid state diffusion. Philibert is a member of several societies, including the Société Française de Métallurgie et des Matériaux, Société Française de Physique, and the Materials Research Society.

Severed Rat Nerves Fused with Polyethylene Glycol Solution Regain Functionality

### Severed Rat Nerves Fused with Polyethylene Glycol Solution Regain Functionality

Researchers from the University of Texas—Austin, the Swiss Federal Institute of Technology and University of Zurich, and the University of Texas Medical Branch have developed a procedure to repair central and peripheral nervous systems (CNS and PNS) in invertebrates and mammals by fusing severed nerves with a calcium-free solution of polyethylene glycol (PEG), then sealing the attachment with a PEG hydrogel. While the researchers have already tested the hydrogel *in vivo* on earthworms, they know only that the material is nontoxic to nerve cells in intact rats, but have yet to test the full technique *in vivo* in rats.

For mammalian nerves, the scientists apply the Ca<sup>2+</sup>-free PEG for 1–2 min to the cut ends of severed axons. Once the ends are fused, the PEG solution is washed off and replaced with calcium-containing solutions that mimic the salt composition of mammalian body fluids. The scientists reported in the April 1 issue of *The Journal of Neuroscience* that many of the once-severed axons can again transmit electrical impulses through the lesion site within 2–30 min. Complementing repair of the axons with the application of hydrogel, the researchers demonstrated that the rejoined axons in earthworms can be maintained for 20 days, permanently reestablishing myelinated medial giant axon (MGA)-mediated behaviors.



To synthesize the PEG hydrogel, neurobiologist George D. Bittner of the University of Texas and his research team photochemically induced the polymerization of a precursor solution to form a crosslinked hydrogel directly on the tissue being treated *in situ*. The precursor solution consisted of 23% 8 kDa PEG diacrylate, 1 mM eosin Y, 100 mM triethanolamine, and 1500 ppm *N*-vinylpyrrolidone. The researchers reported several advantages of their PEG-based hydrogel: It transforms within 30 s from a liquid precursor into a solid gel that adheres to the sciatic nerve tissues; it is biocompatible and noninflammatory *in vivo* because PEG interacts favorably with proteins and blocks interactions with cell-surface receptors, "producing low levels of protein adsorption and inflammatory cell adhesion"; it can be designed to degrade over a few days to several months by incorporating ester linkages "to provide sites for nonenzymatic hydrolysis"; and the hydrogel does not affect axonal structure or function.

The researchers said that the ability to keep severed axons alive would give surgeons a longer time to rejoin those axons with PEG solutions.

**Gamma Irradiation Reduces Wear of Polyethylene Used in Hip Replacement**

Thierry Blanchet, associate professor of mechanical engineering at Rensselaer Polytechnic Institute, reported in April to the annual meeting of the Society for Biomaterials his preliminary research results showing that tying up free radicals in the polyethylene used in hip replacement can reduce wear by a factor of three. In total hip replacements, surgeons cement a polyethylene cup in the hipbone socket. They also insert a metal shaft in the femur. A ball on the end of the shaft rides in the cup. This replacement is expected to last for 15 years. Blanchet said that one major cause of failure is a "cascading chain of events" that begins when the polyethylene cup is irradiated during manufacture to sterilize it.

He said that the radiation strips hydrogen from the hydrocarbon chains, creating free radicals that react with oxygen from the surrounding environment. These aging reactions cut the polymer chains, and the polymer abrades, releasing debris. The bone reacts, and the implant loosens.

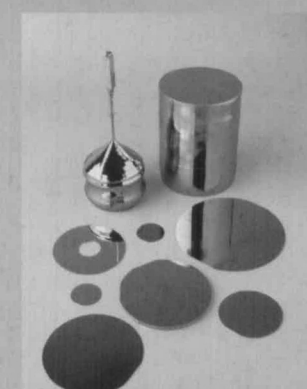
Blanchet and doctoral student Brian Burroughs showed that during irradiation of ultrahigh molecular weight polyethylene (UHMWPE), or directly after, they could force the free radicals to react with hydrogen, ethylene gas, or with each

other, tying them up so they could not react later with oxygen. As reported at the meeting, the researchers gamma irradiated 4 mm x 4 mm cross-sections of UHMWPE pin specimens in either an air or vacuum environment at room temperature to a dose of 4 Mrad. For two months, they aged the irradiated specimens in various environments, including vacuum storage at 20°C and 70°C, and C<sub>2</sub>H<sub>4</sub> storage and H<sub>2</sub> storage each at 70°C.


They used Fourier transform infrared spectroscopy to quantify the amount of oxidation. The researchers reported a reduction in oxidation when the specimens were stored at 70°C, especially in C<sub>2</sub>H<sub>4</sub> and H<sub>2</sub> environments.

To test wear, the researchers slid the UHMWPE pins against a stainless steel disk in room air (20°C) at a sliding speed of 0.1 m/s and a nominal contact pressure of 6.5 MPa. They reported the most

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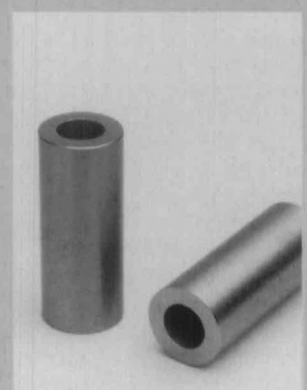
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wear resistance of specimens stored in the C<sub>2</sub>H<sub>4</sub> and H<sub>2</sub> environments. The researchers said, "In the case of vacuum storage with temperature increased to 200°C, a wear resistance of at least twice that of unirradiated UHMWPE was maintained, despite aging, upon either gamma or electron (10 MeV) irradiation."

### Degradable Polymer Bone Replacement Developed

Researchers at the University of Colorado—Boulder and the Massachusetts Institute of Technology (MIT) have developed a family of photopolymerizable, methacrylated anhydride monomers and oligomers used as bone replacement that gradually breaks down in the body, enabling natural bone to regrow in its place. Kristi S. Anseth of Colorado and Robert Langer and Venkatram R. Shastri of MIT reported in the February issue of *Nature Biotechnology* that they conducted photopolymerization using 1.0 wt% camphorquinone (CQ) and 1.0 wt% ethyl-4-*N,N*-dimethylaminobenzoate (4EDMAB) with 150 mW/cm<sup>2</sup> of blue light. They attributed the rapid formation of the highly crosslinked, degradable polymer networks to the photobleaching of CQ that allowed penetration of the blue light, enabling the conversion.

The researchers said that they synthesized dimethacrylated anhydride monomers from precursor diacid molecules of sebacic acid (SA), 1,3-bis(*p*-carboxy phenoxy) propane (CPP), and 1,6-bis(*p*-carboxy phenoxy) hexane (CPH), which they subsequently photopolymerized. They reported that the modulus tensile of the polymer networks MSA and MCPH were 2 GPa and 1.5 GPa, respectively, with tensile strength of 27 ± 9 MPa (MSA) and 15 ± 8 MPa (MCPH) and compressive strength of 34 ± 4 MPa (MSA) and 39 ± 11 MPa (MCPH). The strength, then, of each polymer network is comparable to that of trabecular bone but significantly lower than cortical bone.

When they tested degradation, the researchers found that the crosslinked SA completely degraded within one week and that only 25% of the crosslinked CPH degraded after three months. Noting that the degradation predominantly occurred by surface erosion, the researchers said, "Assuming surface erosion dominates, this time scale would suggest that the total time for degradation of the more hydrophobic CPH networks will be close to one year." The researchers expect to be able to copolymerize the dimethacrylated SA and CPH networks, developing high-strength polymers that degrade on time scales ranging from one week to one year.

The researchers tested formation of the polymer on a bone screw such as that used to hold together fractures and *in vivo* in a rat. In the latter experiment, they said, "The absence of inflammatory cells in the amorphous region between the polymer network and the cortical bone suggests that the polymer network is well tolerated by the surrounding tissue components."

### Support Structures Play Catalytic Role in Radiation-Induced Degradation of Chemical Wastes

To reduce the danger of nuclear wastes stored in underground tanks, researchers from Northwestern University and the University of Notre Dame have demonstrated how radiation reacts with minerals in the tank to form gases. In their study of radiation-induced surface chemical processes on Al<sub>2</sub>O<sub>3</sub>, reported in the April 8 issue of *Journal of Physical Chemistry B*, George A. Zacheis and Kimberly A. Gray of Northwestern and Prashant V. Kamat of Notre Dame said that they chose alumina not only because of its presence in waste tanks but because of its general environmental significance since it is found in a wide range of concentrations in soil. They used KBr as a control in their experiments since it is noncatalytic. The researchers chose to experiment with hexachlorobenzene (HCB) because it is a common soil contaminant associated with chemical manufacturing and use and, they said, "it is also a useful surrogate for a large class of chlorinated, hydrophobic, aromatic pollutants commonly encountered at environmental sites." The researchers monitored the reaction with diffuse reflectance ultraviolet and Fourier transform infrared spectroscopy, and they identified and quantified the results by gas chromatography with electron capture detection (GC-ECD) and gas chromatography-mass spectroscopy (GC-MS).

According to the researchers' article,  $\gamma$ -irradiation of HCB-coated alumina at a dose of 82.8 kGy resulted in a catalytic conversion of HCB, producing pentachlorobenzene (PeCB) and tetrachlorobenzene (TeCB) on the surface of alumina. "The rate and extent of PeCB formation was directly dependent and inversely proportional to HCB surface coverage," they said; "Higher HCB surface coverage on alumina caused the blocking of active surface sites and the reactive intermediates necessary for HCB transformation." According to the scientists, past research shows that gamma rays create electron-hole pairs in oxide particles, producing triplet excitons and that research with silica has demon-

strated that triplet excitons and trapped electrons can react with surface-bound OH groups to generate H-atoms. The researchers suggest that, on the basis of past research, "the role of trapped electrons and holes, as well as conduction band electrons, is central to the degradation of surface adsorbed HCB on alumina."

The researchers conclude that their observation of the degradation of HCB leading to hydrogen gas may lead to strategies to "enhance or control radiolytic reactions in environmental systems by the addition of metal oxides." The build-up of hydrogen and other volatile gases in storage tanks is one of the most serious safety issues associated with long-term storage of radioactive wastes.

### Fraunhofer Diffraction Studies of Polymer-Blend Droplets Indicate Homogeneous Growth Below 10 $\mu$ m Diameter

Researchers at Oak Ridge National Laboratory (ORNL) and Iowa State University have discovered a technique to blend two polymers within a single microparticle in an evaporating solution provided that the initial droplet is less than 10  $\mu$ m in diameter. Mike Barnes of ORNL's Chemical and Analytical Sciences Division said, "For small droplets of a solution, solvent evaporation takes place quickly enough to inhibit phase separation, producing dry polymer blend particles that have uniform structure to within molecular dimensions." According to the researchers' article published in the February 1 issue of *Optics Letters*, the problem of phase separation from bulk-immiscible components in solution has prevented adequate production of homogeneous polymer blends.

In their studies, the researchers codissolved poly(vinyl chloride) (PVC) and polystyrene (PS) in tetrahydro-furan (THF) with variable droplet sizes ~10–40  $\mu$ m. They then analyzed the Fraunhofer diffraction pattern of the blend to reveal the real and the imaginary parts of the refractive index which can be tuned by adjusting the relative weight fractions of the polymers in the solution. Their analysis of 20:80 w/w PVC-PS blend and 50:50 w/w PVC-PS blend with initial droplet sizes of 9–10  $\mu$ m reveals a lack of distortion in the diffraction data, indicating, they reported, that "the mixed-polymer particles are homogeneous to a length scale of  $\leq$ 25 nm." They determined that the refractive index of the particle in the 20:80 PVC-PS-PVC is (1.5636, 0.0001) and in the 50:50 PVC-PS blend is (1.5285, 0.0008), concluding in their report that "the polymers are homogeneously



blended within the particle."

The researchers further determined that droplets initially larger than 15–20  $\mu\text{m}$  do not form homogeneous blend particles as revealed, in one example, by a distorted diffraction pattern obtained from the 50:50 PVC-PS blend in THF with an initial droplet size of  $\sim 35 \mu\text{m}$ . The researchers said, "Our results show that for large droplets the time scale for solvent evaporation is too long to inhibit phase separation."

According to the researchers, the finding clears the way for development of materials in the form of bulk composites and blends that can be used for coatings, optoelectronic components, magnetic media, ceramics and special materials, micro- or nanomanufacturing, and bioengineering.

### Activated Carbon Created from Coal Waste

High-value carbon products like activated carbons may become a commercially viable by-product of the new, environmentally friendly methods used to burn coal,

according to M. Mercedes Maroto-Valer, research associate in the Energy Institute of The Pennsylvania State University.

Maroto-Valer's research team used both unburned carbon and anthracite coal to create activated carbon and compared the results from both. After separation from the fly ash, they activated the unburned carbon with steam at 850°C. The unburned carbon contained few volatile components because it had already been heated while in the combustor.

The researchers crushed the anthracite before treating it with steam.

"It appears that the unburned carbon is suitable for manufacturing activated carbon products," said Maroto-Valer. "We get high surface area after short activation times and with product yields over 70 percent."

As Maroto-Valer reported at the annual meeting of the American Chemical Society in Anaheim, Calif. on March 23, activated carbon from wood products has about 10% yield. The anthracite coal activated for the same amount of time as the

unburned carbon had about 59% yield, and higher surface area, and the anthracite activated for slightly longer had 33% yield and even better surface area.

While both anthracite and unburned carbon can produce acceptable activated carbon, unburned carbon is probably less expensive and better for the environment, according to the researchers. Unburned carbon, separated from fly ash, does not need cleaning or crushing, nor does it need heating to remove volatile components. Also, while anthracite sells for about \$50 a ton, the waste from power plants can be separated for \$10–15 per ton, and the fly ash could be sold to cement manufacturers.

"The combustion of 920 million tons of coal generates about 80 million tons of fly ash and unburned carbon as combustion by-products," said Maroto-Valer. "Separating this waste and using both components is certainly more economical and environmentally friendly than simply disposing of the waste." □

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