

Synthesis of Layered LiMnO₂ Holds Potential as an Electrode for Rechargeable Lithium Batteries

Researchers at the University of St. Andrews, United Kingdom, reported the synthesis and performance of layered, anhydrous and stoichiometric LiMnO₂ as an electrode for rechargeable lithium batteries. They created the material by ion exchange from NaMnO₂. The researchers, A. Robert Armstrong and Peter G. Bruce, reported in the June 9 issue of *Nature* that they have obtained LiMnO₂ by refluxing NaMnO₂ with an excess of LiCl or LiBr in *n*-hexanol at 145–150°C for 6–8 hours. After cooling to room temperature they filtered the product under suction and washed it, first with *n*-hexanol and then with ethanol, then dried it. They established phase purity by powder x-ray diffraction. The oxide ions are stacked in cubic close-packed layers. Manganese ions reside in each octahedral site between the first and second oxide layers, and Li⁺ ions between the second and third layers. According to the researchers, "up to 0.95 lithiums per formula unit may be extracted from LiMnO₂ on initial charging, corresponding to a capacity of 270 mA h g⁻¹." At a current density of 0.5 mA cm⁻², a capacity of 200 mA h g⁻¹ may be reached up to a cut-off potential of 4.3 V.

Thin-Film Zirconia Electrolyte Doubles Fuel Cell Performance at 800°C

Focusing on solid oxide fuel cells (SOFCs), researchers at Ernest Orlando Lawrence Berkeley National Laboratory (Berkeley Lab) have used a ceramic process to develop a thin-film electrolyte that both doubles the current power output and significantly reduces the cost of SOFCs.

The researchers report that cells with the ultra-thin ceramic electrolyte generate 2 W/cm² of cell surface area. During testing, the electrical output remains constant over 700 hours.

The electrolyte, a yttria-stabilized zirconia film, starts out as a ceramic powder suspended in solution. The solution is painted onto the anode (electrode) and then fired or sintered. The process readily lends itself to assembly line usage. The anode that is painted must be porous in order to allow for the flow of hydrogen. Paints want to fill and plug up these pores rather than to sit on top of the porous anode. When the anode and ceramic coat are sintered, both shrink. Unless they shrink in unison, the ceramic cracks or pinholes form.

SOFCs have been most fuel-efficient operating at 1000°C. This high temperature increases the cost of materials and decreases the lifetime of the cell. According to one of the researchers, Selmar de Souza, "SOFCs are solid-state devices. We know how to drop their operating temperature but the problem has been the electrolyte. It conducts ions between electrodes and when you drop the temperature, you lose conductivity. One way to deal with this is by making the electrolyte thinner."

Institute of Materials Announces 1996 Awards

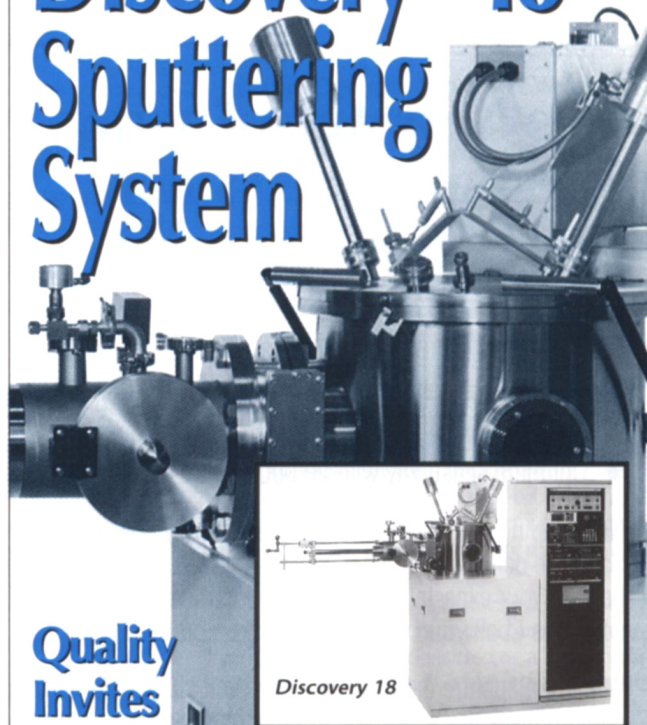
The Institute of Materials awarded nearly 30 medals and prizes for outstanding achievement in the field of engineered materials, given at the Institute's Annual General Meeting held in London on June 5, 1996. Among the recipients are, for the Griffith Silver Medal and Prize, **A.J. Kinloch** of Imperial College, a pioneer in using fracture mechanics in the analysis of adhesion; for the Swinburne Award, **E.J. Kramer** of Cornell University, a 25-year contributor to the understanding of polymer physics; for the Platinum Medal, **D.R.F. West** of Imperial College whose main interests have been alloy constitution, phase equilibria, phase transformation, and structure/property relationships; and for Honorary Fellowship of The Institute of Materials, **D. McLean**, in recognition of his lifetime contribution to physical metallurgy.

The following awards were given for personal achievements

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and outstanding papers: The Guy Bengough Medal & Prize to **A.M. Beccaria** and **G. Pigi** of Instituto per la Corrosione Marina dei Metalli; the Colclough Medal & Prize to **G. Walker** of British Steel, Scunthorpe Works; the Cook Prize to **S.C. Flood** of Powdrex Limited and **P.A. Davidson** of Cambridge University; the Dowding Medal & Prize to **J.L. Evans**, consultant to British Steel; the Elegant Work Prize to **P.D. Brown** and **C.J. Humphreys** of the University of Cambridge; the Grunfeld Medal & Prize to **L. Christodoulou** of Imperial College; the Hadfield Medal & Prize to **R.N. Younger**, formerly Technical Director, Davy International; the Hancock Medal to **D.A. Hills**, consultant; the Hume Rothery Prize to **L. Kaufman** of Cambridge Technology Centre; the Jenkins Award to **A. Lawley** of Drexel University; the Kroll Medal & Prize to **K.C. Mills** of the National Physical Laboratory; the Nelton Award to **R.J. Crawford** of Queens' University; the Prain

Medal & Prize (sponsored by the Copper Development Association) to **W.S. Lyman**, consultant; the Rosenhain Medal & Prize to **P. Gregson** of Southampton University; the Stokowiec Medal & Prize to **P. Beckley** of European Electrical Steels Ltd.; the Thomas Medal & Prize to **C.M. Sellars** of the University of Sheffield; the Verulam Medal & Prize to **R.C. Hudd** of British Steel Strip Products; the Williams Prize to **S.G. Thornton** of British Steel Teeside and **F. Haers** of Sidmar NV; the Vanadium Award (sponsored by VANITEC) to **W.B. Morrison** of British Steel; the Charles Hatchett Award 1996 (sponsored by Niobium Products Company GmbH) to **G.I. Rees** of TWI, **J. Perdix** of Sollac, **T. Maurickx** of Sollac, **H.K.D. Bhadeshia** of Cambridge University, and **C. Fossaert** of Sollac; the A.T. Green Award to **R. Damani** of the University of Surrey; and the T.B. Marsden Award to **B.D. Gibson** of the Institute of Materials.

From the National Lecture Competition,

first prize went to **Jacqueline Butterfield** of British Steel, second prize went to **Ruth Withey** of the University of Birmingham, and third prize went to **John Pugh** of the University of Strathclyde.

Further information on the prizes and the 1996 recipients is available from Carolyn Shaw, Awards Administrator, The Institute of Materials, 1 Carlton House Terrace, London SW1Y 5DB; 44-171-839-4071; fax 44-171-839-2071.

SBIR Update

Advanced Refractory Technology, Inc. (Buffalo, New York) was awarded \$69,000 by the Naval Surface Warfare Center for evaluation of AlN separators in high-temperature batteries for underwater vehicle propulsion, as used for underwater unmanned vehicles (UUV).

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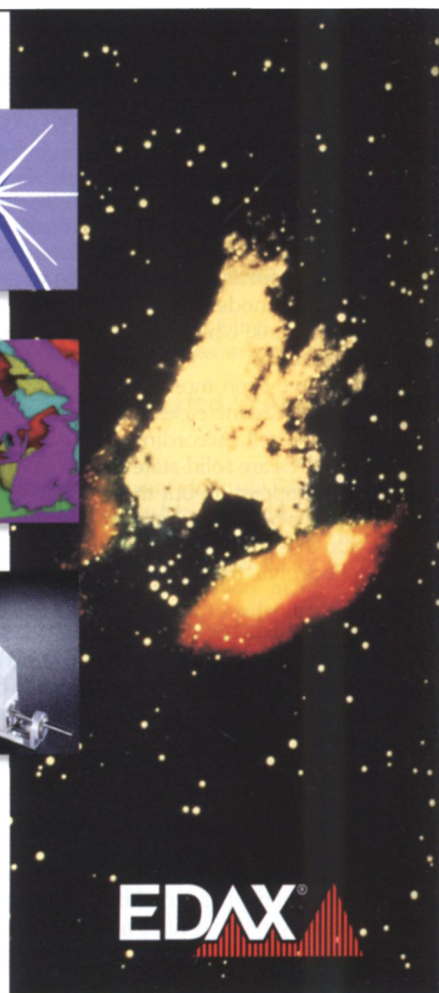
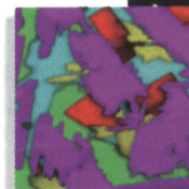
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Oxyanions Promote Crystallization of Zeolites

Researchers from the National Chemical Laboratory in India reported in the May 23 issue of *Nature* various "promoter" ions that speed up the crystallization rate of zeolites. With the addition of oxyanions (ClO_4^- , PO_4^{3-} , NO_3^- , SO_4^{2-} , CO_3^{2-} , AsO_4^{3-} , ClO_3^- , BrO_3^- , and IO_3^-) to the molar gel composition, the crystallization process of a range of zeolite structures (NU-1, FER, MFI, ZSM-48, MTW, and BEA) accelerated. The researchers used fumed silica as a silica source, stirred with template species, such as quaternary ammonium ions, and alkali in deionized water for one hour. Then the solution of an aluminum source (NaAlO_2 or aluminum sulfate) was stirred into the silica. The promoter was then stirred into the gel for one hour at room temperature, enhancing the crystallization process. At a crystallization temperature of 140°C for BEA, the crystallization process took nearly 200 h crystallization time with no promoter as compared to under 50 h with the promoters perchlorate and phosphate and a little over 50 h with arsenate. According to the researchers, "promoter oxyanions greatly polarizes the hydrophobic hydration spheres formed around silicate units and the alkyl groups of the organic structure-directing agent, thereby facilitating the overlapping of TPA-enclathrated silicate polyanions. Such overlapping, in turn, forms the composite species at the onset of crystallization." The researchers reported a correlation between the Z/r (charge/radius ratio) of the central cation of promoter oxyanions and the corresponding crystallization time of ZSM-5 synthesized with the promoters. As the polarizing ability of the oxyanions increases, the crystallization rate increases. The scientists concluded that ionic species with greater polarizing ability enhances the process of crystallization by accelerating the condensation process.

Plasma Technology Destroys Organic Contaminants in Soil and Ground Water

Los Alamos National Laboratory scientists have demonstrated a system that destroys volatile organic compounds (VOCs) found in ground water and soil. Using a nonthermal plasma that creates highly reactive molecules (free radicals) to break down the contaminants, the technique has been used to extract VOCs from soil at McClellan Air Force Base, California, and from ground water at Tinker Air Force Base, Oklahoma. The plasma technology is likened to cold combustion; instead of using heat to break up contaminants, plasma destroys molecules

using atoms or molecules that have unpaired electrons.

At McClellan, the contaminants include benzene, toluene, acetone, freon, xylene, trichloroethylene, trichloroethane, and perchloroethylene, some of which react in the atmosphere and contribute to the depletion of high-altitude ozone, the formation of ground-level ozone and, to a lesser extent, acid rain. The plasma technology treated a maximum of $10\text{ ft}^3/\text{min}$ of air and operated for 300 hours over a two-month period. Researchers monitored gas flow, temperature, pressure, and electrical power.

At Tinker Air Force Base, ground water was pumped through an air stripper that cascades the water within the stripping tower causing the volatile contaminants to become airborne. Researchers pumped the air that came off the water stripper through the plasma, where it was treated. Clean air exited the system, while the leftover, less-hazardous compounds were recovered in small, stainless steel tanks that contained a dry scrubber material.

The plasma system consists of a 20-foot trailer filled with specialized equipment. The chemical reactions occur in an aluminum tank that houses 20 nonthermal plasma cells. The nonthermal plasma cells use high-voltage electrical energy to create large quantities of highly reactive free radicals. The free radicals subsequently react with and break up hazardous organic chemicals, converting them to nonhazardous substances such as carbon dioxide, water, and acids that can be neutralized. Most of the free radicals are oxygen atoms and hydroxyl molecules that react with and oxidize the larger molecules, thereby functioning much the same as incineration, but without the heat and added fuel exhaust.

Multiple Laser Reflections of Stress Monitoring Improve Electronic, Magnetic, Optical Devices in Noisy Environments

Scientists at Sandia National Laboratories have developed a method to determine stresses in very thin films of materials used in advanced electronic, optical, and magnetic devices. By beaming a one-half milliwatt laser into an angled, partially reflective mirror coated on both sides, an etalon (here, not used as an interferometer), researchers Eric Chason and Jerry Floro create a series of internal reflections which emerge as parallel beams of light. The beams are directed into the process chamber and onto the material surface over a region of about one centimeter. The spacing between reflections of the beams are

monitored electronically using a video camera. Distortions in the material as small as one-hundredth of a micron can be measured by the change in distance between the reflected beams. The changing distance becomes a continuous measure of warpage and thus, stress. To calibrate the amount of stress in thin films, Chason and Floro monitor the curvature of the supporting material to determine its warp while varying the temperature or thickness of the film.

Instrument vibration never appears in the readings because the rigid connection between laser and etalon causes all the beams to move together. This method enables researchers to measure stress as it builds in a noisy environment rather than interrupt the process to remove the product for off-line analysis.

In a further improvement, the researchers have placed a second etalon rotated at right angles to the first. This piggyback arrangement creates a square array of beams from the single source, making possible simultaneous measurement over a square area rather than just along a line, and allowing analysis of complex shapes.

Liquid Carbon Dioxide Cleans Up Dry Cleaning

A system, developed under a collaboration between Los Alamos and Hughes Aircraft, replaces hazardous dry cleaning chemicals with a liquid carbon dioxide cleaning process. Under 800 to 1,000 psi, carbon dioxide acts like a liquid and serves as an organic, entirely recyclable solvent that extracts dirt from clothing. When the liquid carbon dioxide is allowed to return to its gas state, dirt just falls out. Repressurized, the carbon dioxide can be used again. This process generates no waste, other than the dirt.

According to Craig Taylor, principal investigator on the project in Los Alamos National Laboratory, carbon dioxide dissolves sweat, oils, and dirt.

The primary solvent used for many years by dry cleaners is perchloroethylene, or PERC, which is classified as a hazardous toxic substance and is strictly regulated under the Clean Air Act and the Comprehensive Environmental Response, Compensation and Liability Act. The liquid carbon dioxide system will clean any material that is currently dry cleaned, and, in addition, clean furs, leathers, and sequins.

ONR Honors Padture with Young Investigator Award

Nitin P. Padture, an assistant professor in the Department of Metallurgy and Materials Engineering at the University of Connecticut, Storrs, has received a Young

Investigator Award from the Office of Naval Research (ONR) for "exceptional promise for doing creative research and teaching." This award carries with it a three-year, \$300,000 research grant, which Padture will use for studying mechanical behavior of *in situ*-reinforced ceramics.

Padture received his PhD degree from Lehigh University and has spent three years at the National Institute of Science and Technology as a postdoctoral fellow before joining the University of Connecticut faculty in January 1995. His research interests include processing and mechanical behavior of ceramics. He has published 27 papers and is an advisor to the Materials Research Society student chapter at the University of Connecticut.

Tuller Named Editor-in-Chief of *Journal of Electroceramics*

Harry L. Tuller has been named the Editor-in-Chief of the newly formed Kluwer *Journal of Electroceramics*. As Sumitomo Electric Industries Professor of Ceramics and Electronic Materials and Director of the Crystal Physics and Electroceramics Laboratory at the Massachusetts Institute of Technology, Tuller is known for his research in solid-state ionics, grain-boundary-controlled semiconducting devices, defect theory, optical properties, sensor materials development, and semiconductor micromachining. The journal's editorial board comprises 29 internationally known scientists with specializations in the fields of electro-optics; ferro-, piezo-, and pyro-electrics; magnetic ceramics; processing/microstructure development; semiconductors/dielectrics; sensors; and solid-state ionics.

1997 National Medal of Technology Call for Applications

The National Medal of Technology is the highest honor awarded by the President of the United States for technological innovation. Since 1985, the Medal has annually been bestowed upon individuals, teams, or companies for accomplishments in the innovation, development, and commercialization of technology as evidenced by the establishment of new or significantly improved products, processes, or services.

The purpose of the program is to create an environment that fosters technological innovation and recognizes the significant contributions the U.S. leading innovators have made to the country's economic strength and standard of living.

Nomination application packets for 1997 can be obtained from the U.S. Department of Commerce by contacting the National Medal of Technology Program

Director, Technology Administration, Room 4814C, U.S. Department of Commerce, 14th Street and Constitution Avenue, NW, Washington, DC 20230; 202-482-5572; fax 202-482-4817; e-mail nmt_usotp@banyan.doc.gov; or the world wide web <http://www.ta.doc.gov/nmthome/nmt.htm>.

The deadline for submission is **September 27, 1996**.

SARIS Analyzes Surface Structure and Chemical Reactions

Wayne Rabalais, chemistry professor at the University of Houston, and two of his graduate students have developed a crystallography technique called scattering and recoiling imaging spectrometry (SARIS) for monitoring surface structure and chemical reactions on surfaces. By capturing images of ions in flight, Rabalais and his team are able to follow chemical reactions at a surface in real time. Using a large area, position sensitive microchannel plate detector which is gated so it can accept data only in small time windows—as short as 10 ns—Rabalais and his team are gathering information about changes on the surface. Images showing spacial distribution of scattered particles are collected digitally, then compared. This enables the researchers to determine where a molecule reacts on a surface and how the atomic structure of the surface changes as a result of the chemical reaction.

Rabalais and his team use a custom-made chamber that includes a 75 mm × 95 mm detector plate surrounded by a vacuum chamber. The chamber needs to be unusually large and oddly-shaped so the detector could be moved through wide arcs inside it.

Now beginning the third year on this project funded by the National Science Foundation, Rabalais is producing colorful and artistic looking images of surfaces.

Cottrell Scholars to Receive \$50,000 Research and Teaching Awards

Eighteen young faculty scientists embarking on research and teaching careers are the 1996 recipients of \$50,000 Cottrell Scholars Awards, which were announced by Research Corporation, a foundation for the advancement of science. The awards require no fixed budgets, permitting recipients to apply funds as they think best to meet research and teaching objectives. Among the recipients are **George Nicholas Gibson**, University of Connecticut: "Ultrafast Time-Resolved Measurements of the Ionization and Dissociation of Molecules by Strong Laser

Fields"; **David Howard Reitze**, University of Florida: "Coherent Control of Charge Carrier Motion in Semiconductor Heterostructures"; **Xiaoyang Zhu**, Southern Illinois University: "Surface Reaction Dynamics in Chemical Vapor Deposition"; **David Z. Besson**, University of Kansas: "Measurement of Coherent Radio Wave Emission Induced by Ultrahigh Energy Neutrino Interactions"; **Ziqiang Wang**, Boston University, "Coulomb Interaction, and Electronic Transport in High Magnetic Fields"; **Mark Thomas Tuominen**, University of Massachusetts: "Measurements of Single-Electron Nanostructures in a Quantum Cavity: Manipulating the Quantum Interaction of Matter and Light"; **Grover A. Swartzlander, Jr.**, Worcester Polytechnic Institute: "Experimental Investigation of Optical Solitons Used as 'Optical Tweezers'"; **Karl Todd Mueller**, The Pennsylvania State University: "Efficient NMR Methods for Determining Multiple Internuclear Distances in Complex Solids"; **Mark Lee**, University of Virginia: "New Physics From an Old Material: Exploring the Correlated Motion of Interacting Electrons in Silicon"; **Brian Max Tissue**, Virginia Polytechnic Institute and State University: "Characterization of Interfaces in Nanocrystalline Materials Using Laser Spectroscopy of Lanthanide Probe Ions."

The Cottrell Scholars Awards are named after Frederick Gardner Cottrell, the scientist, inventor, and philanthropist who established Research Corporation, a science advancement foundation, in 1912. The goals of the foundation are to make inventions "more available and effective in the useful arts" and "to provide means for scientific research and experimentation."

Charge Density Wave Observed on Lead-Coated Surface of Ge Crystal

Researchers at the University of Tennessee and Sandia National Laboratories have observed a charge density wave (CDW) localized at the lead-coated (111) surface of a germanium crystal. While at room temperature a thin film of lead-coated germanium appears to be metallic with a surface unit cell containing one Pb atom and three outer layer Ge atoms, each with an unpaired electron, at -20°C, a gradual and reversible electronic transition occurs. A small bandgap develops as the temperature decreases, resulting in a honeycomb pattern charge arrangement. The researchers report in the May 30 issue of *Nature*, "On decreasing temperature, the Pb/Ge(111)- α system forms a commensurate surface charge density wave (SCDW) independent of any bulk phenomena."

According to their report, the researchers used low-energy electron diffraction to ascertain the symmetry of the surface atomic lattice, electron energy-loss spectroscopy (EELS) to probe the excitation spectra of the interface, and scanning tunneling microscopy to view the spatial variations in the surface charge density with atomic resolution. One main inconsistency they observed was, at low temperature, the unit cell contains three Pb and nine Ge atoms, and 21 valence electrons. This finding contradicted the EELS observation.

Microchip Thermocapillary Pump Drives Analysis of DNA

In an article published in the May 28 issue of the *Proceedings of the National Academy of Sciences*, David T. Burke, an assistant professor of human genetics at the University of Michigan, and his colleagues reported initial test results on five microfabricated components and their preliminary integration into a DNA-analyzing chip that is 3 cm long and 0.5 cm wide.

"The heart of the device is a thermocapillary pump which uses surface tension rather than valves or moving parts, to mix drops of pure DNA with an enzyme solution and drive the DNA through five different components on the microchip," said Mark A. Burns, associate professor of chemical engineering at the University of Michigan.

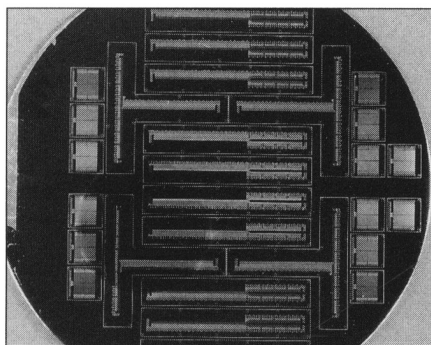
The DNA solution forms discrete drops with curved surfaces inside a series of narrow channels on the device. Using microscopic heaters built into the chip, the researchers heat one side of a drop which reduces the surface tension and increases the internal pressure on that side. The pressure difference pushes the drop through the channel toward the direction of lower pressure.

Researchers have moved samples through all five processing steps on one substrate to produce DNA separations, which were visible through a microscope, according to Burns.

According to Burke, the current analysis technique for DNA involves a time-consuming process in which sequencing genes with polymerase chain reaction requires a molecular biology laboratory and at least 10 individual procedures. He said, "Our goal is to automate the process by shrinking the lab to fit on one silicon microchip."

Technical problems related to handling such small amounts of liquid, and interactions between liquids and materials in the

chip remain to be solved. Work is currently underway to reduce the size of the microchip to about one square centimeter.



This silicon wafer contains three of five components fabricated for use in one integrated microchip for DNA analysis. Individual components in the microchip include entry ports, transport channels, a thermally controlled reactor where mixing and enzyme digestion or polymerase chain reaction amplification occurs, an electrophoresis channel, and a detector.

The square sections shown on the silicon wafer are microscopic heaters which drive the thermocapillary pumping function of the device. Long, rectangular structures are micron-sized channels for measuring, mixing, and moving tiny drops of DNA.

Be Atom Illustrates "Schrödinger's Cat" by Superposition of Two States

When a team of physicists at the National Institute of Standards and Technology supercooled a beryllium atom with a laser, then hit it with five sequential pulses of Raman beams, they caused the atom to oscillate, appearing in two positions at once, that is, two coherent-state wave packets. According to the physicists' report in the May 24 issue of *Science*, the series of laser pulses entangled the internal (electronic) and external (motional) states of the ion, creating a mesoscopic "Schrödinger's cat" state. The wave packets were separated by a mesoscopic distance of more than 80 nm.

The researchers said that each wave packet is correlated with a particular internal state of the atom. They applied another laser pulse to couple the internal states then measured the interference of the distinct wave packets. They reported, that the difference frequency of the carrier Raman beams was set near $\omega_{HF}/2\pi \approx 1.250$ GHz

and the difference frequency of the displacement Raman beams was set to $\omega_x/2\pi \approx 11.2$ MHz, which excited the motion of the ion to a coherent state from an initial zero-point state of motion in the harmonic potential. The displacement force supplies quantum entanglement of the internal state with the external motional state. The physicists described the occurrence of the Schrödinger's cat state in five steps: the original wave packet correlates to the quantum ground state of motion after laser-cooling; the wave packet is split after application of the carrier beam; the initial internal state wave packet is excited to a coherent state by the displacement beam force, entangling the internal and motional systems; two distinct internal electronic quantum states of the atom exchange wave packets following another application of the carrier beam; then one of the internal state's wave packets is excited to a coherent state by the displacement beam force, causing the state of the atom to correspond most closely to that of Schrödinger's cat.

Day Receives UMR's 1996 Presidential Award

Delbert Day, Curators' Professor of Ceramic Engineering at the University of Missouri—Rolla (UMR), has received the University of Missouri System's 1996 Presidential Award for Research and Creativity. The award, which includes a \$15,000 stipend for research, is intended to recognize a UM faculty member for a sustained record of nationally and internationally prominent research or creativity.

Day is internationally recognized for his work on the structure and properties of inorganic glasses. He conceived, along with Gary Ehrhardt of UM—Columbia, the idea of using glass microspheres to irradiate malignant tumors. The therapy, which is currently undergoing trials for patients with kidney cancer, results in increased life expectancy with none of the discomforting side effects associated with radiation or chemotherapy. Day is also co-inventor of "glasphalt," a road-paving substance that includes waste glass. He holds 35 patents dealing with glass microspheres, ceramic dental materials, refractories, oxynitride glasses, and transparent composites.

Day earned his PhD degree in 1961 from The Pennsylvania State University and joined the UMR faculty in 1961. □

1996 MRS Publications Catalog Supplement is available at 412-367-3003