Nanoparticle Shapes Defined by Capping-Polymer/Platinum-Ion Ratio

Researchers at the Georgia Institute of Technology have created specific shapes and sizes of colloidal platinum nanoparticles, a development that could lead to advances in the field of catalysis. "Catalytic reactivity depends on the size and the shape of the nanoparticles, and therefore the synthesis of well-controlled shapes and sizes of colloidal particles could be critical for these applications," according to a paper published in the June 28 issue of *Science*.

The researchers altered the ratio of the concentration of the capping polymer material to the concentration of the platinum cations. The capping polymer material—in this case, sodium polyacrylate—wraps around the particles to stop their growth and make them soluble in water, but does not affect their chemical reactivity.

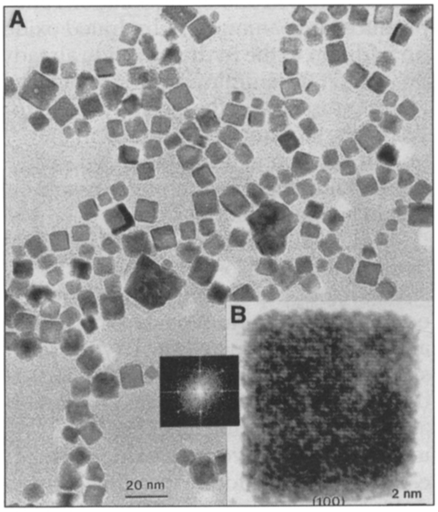
To create colloidal samples for the study, the researchers synthesized platinum nanoparticles in a liquid solution at room temperature, introducing argon and hydrogen gases. The latter served to reduce the platinum ions into neutral atoms in the process of making the nanoclusters. Three different samples were used, each with a different concentration of the capping polymer. All other factors, such as the salt and pH levels, the solvent used, and the temperature, were kept constant.

The researchers observed several geometric shapes, including tetrahedral, cubic, irregular-prismatic, icosahedral, and cubo-octahedral forms. The distribution of the shapes was dependent on the ratio of capping polymer material to the platinum cation. The first sample had a ratio of polymer concentration to platinum salt of 1:1, and contained 80% cubic particles. The second sample had a ratio of 5:1 and was dominated by tetrahedral shapes. It also had small percentages of polyhedral and irregular-prismatic particles. Sample three, with a ratio of 2.5:1, contained a mixture of tetrahedral, polyhedral, and irregular-prismatic particles.

Researchers will now explore the mechanisms of the process at the molecular level to understand how it works. This will include detailed studies of how such solution properties as pH, ionic strength, viscosity, and temperature affect shape distribution.

Mostafa A. El-Sayed, principal investigator for the collaborative project that includes researchers from California and Germany, said, "It is known that catalysis on metal surfaces depends on the face of the metal crystal used. When nanoparticles of certain shapes are used, it is expected that their catalytic activities will vary from one another—and, most likely, from metal crystal surfaces—as they have edges and corners that clean-polished crystal faces do not have."

Since a significant number of the atoms of nanoparticles are located on the surface, where catalysis takes place, nanoparticles are expected to be much more effective in catalysis per gram than larger crystals, according to El-Sayed.



(A) Low-magnification transmission electron microscope (TEM) image of polymer-capped platinum nanoparticles from a solution with a 1:1 polymer to platinum cation ratio, showing the size and shape distribution of the cubic particles. (B) High-resolution lattice image of a cubic platinum particle. (Inset) The Fourier transform of the lattice image gives the optical diffractogram of the particle. (Photo courtesy of Science.*)*

Mechanical Properties of Carbon Nanotubes Measured by Thermal Vibrations

Under transmission electronic microscopy (TEM), researchers have observed thermal vibration in carbon nanotubes at increasing temperatures, from room temperature to 800°C, giving an effective Young's modulus of 3.7±0.2 TPa. The researchers, M.M.J. Treacy and T.W. Ebbesen from NEC Research Institute and J.M. Gibson from the University of Illinois—Urbana, took large bundles of carbon nanotubes and shredded the tip of the bundle, making a brush of roughly horizontal isolated nanotubes. They clamped the nanotubes at one end. At room temperature the free end was blurry, blurring further at increasing temperatures. Near the clamped base, the nanotubes were ~15 nm across; at the free tips, however, the width of one was ~30 nm at 300 K and ~40 nm at 600 K. The blurred tips represent thermal vibration, which is confirmed through various equations as published in the June 20 issue of *Nature*. According to the researchers, the measured Young's modulus is highest for the thinner nanotubes, indicating that these nanotubes could be ideal for carbon-fiber-reinforced materials.

Superdeformed Strontium, Yttrium, Zirconium, Niobium Nuclei Detected

Physicists from Oak Ridge National Laboratory (ORNL) and their collaborators have detected short-lived, rapidly rotating, football-shaped nuclei of strontium, yttrium, zirconium, and niobium.

The nuclei are typically synthesized by bombarding a nickel-58 target with beams of silicon-28 or sulfur-32 ions. Occasionally, some ions strike the target nucleus nearly head-on and fuse with it with little spin. However, many ions hit the target nucleus off-center and produce fused compound nuclei that rotate rapidly. As a spinning nucleus drops down from its excited state, it releases its excess energy, first by emitting neutrons, protons, and alpha particles, then gamma rays. The gamma rays are particularly effective in carrying away the excess energy of rotation. In superdeformed nuclei, the gamma-ray energies drop with decreasing spin. The regularity of the energies of these gamma rays and the time it takes for the nucleus to emit them are used by physicists to determine how deformed these nuclei are. Measurements of lifetimes of these superdeformed states indicate that these fast-rotating nuclear tops last for a femtosecond before they jump to their next lower state.

Cyrus Baktash of ORNL's Physics Division said, "Working with our collaborating groups from Washington University, Lawrence Berkeley National Laboratory, the University of Pittsburgh, and Florida State University, we have detected 10 cases of superdeformed nuclei that have atomic masses in the range of 80 to 90. Our discoveries span four different elements: strontium, yttrium, zirconium, and niobium. They are the fastest-spinning nuclei yet observed."

In the 1960s, it was discovered experimentally that nuclei of actinides, such as plutonium-240, assume elongated shapes and have a good chance to break up, or fission spontaneously, into two fragments. Theorists proposed that rapidly rotating

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nuclei of certain groups of lighter elements could take on the same football shape. Subsequent calculations identified groups most likely to show this effect—several elements with atomic masses of 190–210, 150–160, and 80–90. The measured properties of these nuclei are in reasonable agreement with the predictions of theory.

Benzene-Thermal Process Produces Nanocrystalline GaN at 280°C

Through a benzene-thermal process, researchers at the University of Science and Technology of China synthesized nanocrystalline (30 nm) GaN. Using benzene as a solvent, the researchers combined Li_3N with $GaCl_3$ under pressure in an autoclave at 280°C, a much lower temperature than traditionally used. The sample was heated for 6–12 h, cooled to room temperature, washed with ethanol to remove LiCl, and then dried in a vacuum at 100°C for 2 h. The results consisted of hexagonal cells of GaN with lattice constants a = 3.188 Å and c = 5.176 Å, which are near values previously reported. The researchers reported, however, in the June 28 issue of *Science*, that "an unusually strong (002) peak in the pattern indicates a preferential orientation of [001] in nanocrystalline GaN." Other areas of the sample showed a rocksalt structure with a = 4.100 Å at 37 GPa. The photoluminescence spectrum of the sample showed emission at 370 nm, constant with bulk measurements.

ORNL Develops Self-Aligned Plastic Microspectrometer

A microspectrometer has been developed at Oak Ridge National Laboratory (ORNL) that sorts light according to wavelengths and detects a variety of chemicals. The plastic device contains multiple precision surfaces that diffract light that enters the unit through an aperture consisting of an optical fiber input. This fiber is attached to a fiber optic connector that is positioned directly onto the entrance surface so, unlike other spectrometers, no alignment is required.

"What separates this unit from others on the market is the fabrication technology that allows construction of low-cost, high-performance, completely alignment-free systems," said Slo Rajic, principal developer and a member of ORNL's Engineering Technology Division.

Another key to the microspectrometer is the ultraprecise single-point diamond turning fabrication technology developed at ORNL. This technique involves precision machining that produces optics-quality surfaces that need little polishing.

The microspectrometer can be inserted in the filler tube of an automobile's gasoline tank and configured to detect the octane of gas. Configured differently, to detect other portions of the spectral range, the device can be used as a laser warning receiver, for plasma diagnostics or for wavelength division multiplexing for fiber-optic telecommunications systems. The device can also be used for non-invasive blood chemistry analysis, environmental monitoring, industrial process control, chemical warfare detection, and aircraft corrosion monitoring.

Silver-Wire-in-Tube, BSCCO-2223 Powder Sustains High Current Density

Researchers at Argonne National Laboratory and the University of Pittsburgh have developed a "silver-wire-in-tube" manufacturing technique that consistently produces superconducting wires with "critical currents" greater than 100,000 A/cm^2, which is the value needed for practical

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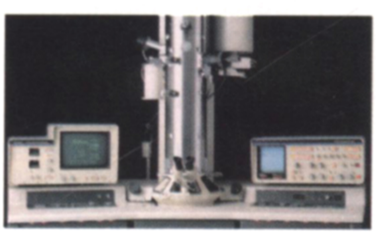

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applications. The technique is based on a key refinement to the "powder-in-tube" method for making wire from high-temperature superconductors. The powder-in-tube method seals high-temperature superconducting powder inside a silver tube, then draws and rolls it into a wire. The result is a silver-clad ribbon of high-temperature superconducting material.

"The key was learning which part of powder-in-tube wire actually carries the supercurrent," said N.G. Eror of the University of Pittsburgh's Department of Materials Science and Engineering.

In 1994, Argonne and the University of Pittsburgh, following up on University of Wisconsin research, found that supercurrent in powder-in-tube wires made at Argonne flowed almost exclusively through a thin interface, the layer of superconductor closest to the silver casing. The interface had the right grain structure and alignment. Since the remainder of the superconductor carried no significant amount of current, the researchers wanted to eliminate it. Ceramists inserted a silver wire into a silver tube and filled the space in between with BSCCO-2223 powder, a high-temperature superconductor made from bismuth, lead, strontium, calcium, copper, and oxygen. When cooled to about 110 K, it superconducts. After the wire was drawn and rolled, virtually all the BSCCO lay along the silver-superconductor interface. The result was a thin layer of superconductor 1–2 μm thick that consistently provided current densities of 100,000 A/cm^2. The powder-in-tube wire could consistently carry only 20,000–30,000 A/cm^2.

Co Wrapped in Graphite-like Carbon Suggests Potential Use for Ultrahigh-Density Magnetic Recording Media

Researchers at NIT Interdisciplinary Research Laboratories in Japan have reported in the June 27 issue of *Nature* that hcp cobalt nanocrystals wrapped by several layers of graphite-like carbon was fabricated from Co-C codeposited film annealed at 300°C. To encapsulate cobalt nanocrystals in graphite-like carbon, the researchers placed a 60 × 60 mm cobalt target adjacent to a 60 × 60 mm carbon target, then guided a 1.5 keV Ar ion beam with a diameter of ~30 mm around the boundary between the targets, thereby controlling the Co-C composition. After the process of ion-beam sputtering, the films were lamp annealed in a vacuum at $<1 \times 10^{-5}$ torr. The researchers used a vibration sample magnetometer to measure the in-plane magnetic properties at room temperature.

To examine the magnetic properties and smooth surfaces of the film required for ultrahigh-density recording media, the researchers deposited a 38-nm-thick film on a Corning 0211 glass substrate, repeated the sputtering process, and annealed the film in a vacuum for one hour at 250°C, 300°C, and 350°C, respectively. After annealing for one hour at 350°C, the saturation magnetization was measured at 550 emu cm^{-3} and coercivity (H_c) was measured at 370 Oe. While an H_c of 2,500 Oe is needed for future ultrahigh-density recording media, the researchers said that their film exhibits high potential for improvement and future use "because H_c is sensitive to many structural parameters, such as orientation, shape, internal stress, and defects of the Co nanocrystals, so there is still room for optimization."

Low-Temperature "Glue" Binds Ceramic Composites

Scientists at Ames Laboratory have developed a "glue" suitable for joining parts made of a class of ceramic materials called CFCCs. They consist of silicon carbide fibers embedded in a silicon carbide matrix. The fibers give the composite toughness that the single-phase ceramic lacks.

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The glue forms a strong joint at temperatures well below the onset temperature of the fiber degradation reaction. The main ingredients in the glue are a silicon-bearing polymer and a low-melting-point alloy of aluminum and silicon in powder form. The alloy gives the mixture the ability to form a strong joint at comparatively low temperatures. The polymer breaks down into silicon carbide and excess carbon when it is heated, and the aluminum alloy powder melts, reacts with the carbon to form more silicon carbide, and forms small islands of aluminum oxide and aluminum boride within the silicon carbide. These islands act much like the fibers in the composite material, toughening the joint.

Unlike other glues, this can be prepared outside a chemical fume hood because it includes no solvent. The viscosity of the polymer depends on the size of its molecules; because the glue is made with oligomers, it naturally has a paste-like consistency and does not have to be thinned with solvent. Without solvent, far less gas is released during curing, and the resulting joint has fewer trapped bubbles and much higher strength, at least double that of joints made by current techniques.

Streetman Named Engineering Dean at UT Austin

Ben G. Streetman, a pioneering researcher in the field of microelectronics and a prize-winning educator in electrical and computer engineering, has been named dean of the College of Engineering at The University of Texas—Austin. The appointment became effective on September 1.

Streetman, director of UT Austin's Microelectronics Research Center, is a science and technology advisor to industry and government, and was instrumental in attracting the microelectronics industry to Central Texas. As an engineering professor at UT Austin since 1982, he has been recognized on several occasions for teaching excellence and has mentored numerous students. His teaching and research interests include semiconductor materials and devices, radiation damage and ion implantation, molecular beam epitaxy, transcient annealing, deep level impurities and defects in semiconductors and multilayer heterostructures. He is the author of *Solid State Electronic Devices* and has co-published more than 250 technical articles. Streetman is a member of the National Academy of Engineering.

After receiving his PhD degree from UT Austin in 1966, Streetman served on the faculty of the University of Illinois at Urbana-Champaign before coming to the University.

a-SiN Coating by Vacuum-Bagging Protects Polymer-Matrix Composites

James K. Sutter of the Lewis Research Center has determined that a-SiN coatings protect polymer-matrix composites (PMCs) against thermo-oxidative degradation. He studied the effects of two types of fabricated a-SiN coatings on a substrate of polyimide-matrix/graphite-fiber composite made from 12 plies of graphite-fiber tape permeated with PMR-II-50 oligomer. In one group, the coating was fabricated by a simulated autoclave vacuum-bagging technique. Samples were coated with a-SiN and aged in air with uncoated samples for 500 hours at 371°C. The uncoated samples lost much more weight than did the coated samples, indicating that the coating protects PMCs against thermo-oxidative degradation.

In the other group in which the a-SiN coating was fabricated by compression molding, the coated sample lost about as much weight as did the uncoated sample. According to a report in the July issue of *NASA Tech Briefs: The Design/Engineering Technology Digest*, "the vacuum-bagged composite surface was resin-rich and free of the exposed fibers and larger craters that are prevalent on compression-molded surfaces." Oxidative weight loss occurs because the exposed fibers provide conduits for the diffusion of oxygen along fiber/matrix interfaces into the matrix.

NIST Issues 1996 STEP Grants

The National Institute of Standards and Technology (NIST) announced four cost-shared grants to build manufacturing assistance and modernization programs aimed at smaller manufacturers in California, Mississippi, and Washington. The awards are part of the State Technology Extension Program (STEP), managed by NIST's Manufacturing Extension Partnership (MEP). STEP grants provide matching funds (the grantee provides at least 50% of the cost of the proposed project) to qualified proposals from state governments and public/private, nonprofit organizations acting with the approval of state governments.

The new extension programs are San Diego Manufacturing Extension Center, Inc. (San Diego, California): federal funding: $65,953; matching funding: $75,678; Small Manufacturers' Institute (Sacramento, California): federal funding: $99,999; matching funding: $101,000; Mississippi Enterprise for Technology (Stennis Space Center, Mississippi): federal funding: $99,999; matching funding: $123,356; and Washington State Department of Community, Trade, and Economic Development (Olympia, Washington): federal funding: $95,809; matching funding: $102,000.

These grants are for planning regional or statewide extension programs for small and medium-sized manufacturers. Projects could include defining industry needs, assessing existing extension resources, or developing a statewide strategic plan for manufacturing extension.

Roy Receives Ellis Island Honor

Rustum Roy, Evan Pugh Professor of the Solid State and Professor of Science, Technology, and Society at The Pennsylvania State University, received the Ellis Island Medal of Honor in a gala black-tie ceremony on May 19 in the Ellis Island Museum. Roy was nominated by the Indian community for his scientific achievements and science policy activities. His scientific research reported in over 600 papers has been directly connected to several new materials which have entered the market place, from sol-gel ceramics to Corningware to diamonds.

Roy arrived in the United States in 1945 and joined the faculty at The Pennsylvania State University in 1946. Among his societal memberships are the U.S. National Academy of Engineering, the Royal Swedish Academy of Engineering Sciences, The Engineering Academy of Japan, and the Indian National Science Academy.

Unlikely Union of K and Ni Achieved Under High Pressure

Potassium and nickel have bonded with each other to form a compound, according to research published in the July 5 issue of *Science*. Using pressures as high as those deep within the Earth to form the potassium/nickel compound, Penn State Assistant Professor of Chemistry John V. Badding and his colleagues demonstrated that pressurized potassium functions like a new element with chemical properties like a transition metal.

"Alkali elements like potassium don't form compounds with transition elements like nickel at normal pressure because their size and electronic structure are so incompatible," Badding said.

The chemists produced the compound by first compressing potassium and nickel powder in a diamond-anvil cell to 31 GPa, then heating it with a laser to about 2,500 K. They confirmed the formation of the resulting compound with a single-wavelength x-ray-diffraction device. Potassium buckles under these pressures, collapsing by a factor of five. Its single outermost valence electron, which controls bonding, deforms from the spherical *s* orbital shape typical of the alkali elements to the smaller-volume, four-leaf-clover-pattern, *d* orbital characteristic of the transition element.

"A single *d*-electron is an extraordinary valence configuration that we just don't find in any of the other elements," Badding said. When potassium's outermost electron transforms to the *d*-orbital state, potassium sheds its alkali character and starts behaving like a transition element, enabling its bonding with nickel.

"Nickel's electron configuration changes much more slowly under pressure because it is relatively incompressible, so nickel stays in its primarily d-electron configuration while potassium changes completely," Badding said.

The research may help geophysicists understand the composition of the Earth's core, which contains primarily iron or an iron/nickel alloy plus some unidentified lighter element or elements.

Westwood Receives Acta Metallurgica J. Herbert Hollomon Award

The seventh annual Acta Metallurgica J. Herbert Hollomon Award has been awarded to Albert R.C. Westwood, Vice President of Research and Exploratory Technology at Sandia National Laboratories. The award will be presented to him in Cincinnati, Ohio on October 8, 1996 at the Annual Awards Dinner of ASM International. The award recognizes outstanding contributions to understanding the relations between materials technology and society, and/or contributions to materials technology that have had major impact on society. Westwood was selected in recognition of his leadership in numerous commissions and committees advising U.S. national and state governments, as well as for the consequences of his own research and that conducted under his direction as manager.

Westwood's principal research contributions have been on the mechanisms of liquid metal embrittlement and stress corrosion cracking, and the discovery and explanation of chemomechanical effects in nonmetallic solids (the effects of adsorbed species on near-surface dislocation mobility).

His current or recent professional responsibilities include Chair of the Public Information Advisory Committee (National Academy of Engineering); Chair of the National Research Council's Committee on Global Aspects of Intellectual Properties Rights in Science and Technology; President of The Metals, Minerals, and Materials Society (TMS); President of the Industrial Research Institute; Member of the Board of Directors of the U.S. Civilian Research and Development Foundation; Member of the Visiting Committee to the National Institute of Standards and Technology; Member of National Critical Technologies Panel for the White House Office of Science and Technology Policy; and Member of Advisory Committees to Massachusetts Institute of Technology, Georgia Institute of Technology, and the Universities of Colorado, Florida, and Maryland.

The Acta Metallurgica J. Herbert Hollomon Award was established in memory of Hollomon and his dedication to promoting positive societal consequences of science and technology. The award consists of a Steuben glass sculpture on a suitably inscribed base, a certificate, and a cash honorarium. □

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