

MRS

# Meeting Scene...

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## XVIII International Materials Research Congress 2009

August 16-20, 2009  
Cancun, Mexico



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### DAY 4

WEDNESDAY, August 19

The [XVIII International Materials Research Congress 2009](#) (IMRC 2009) completed its fourth day of activities including the fourth plenary talk of the meeting by Guozhong Cao, the final evening poster session, and the continuing exhibit. The two major events of the day were the Women in Science Luncheon and the Conference Banquet in the evening.



*Conference Banquet*

### CONTENTS

- [Plenary Lecture - Guozhong Cao](#)
- [Technical Sessions](#)
- [Women in Science Luncheon](#)
- [Scenes from the Conference Banquet](#)

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MRS President Shefford Baker (l)  
and MRS-Mexico President Luis  
Enrique Sansores Cuevas (r).

#### PLENARY LECTURE - GUOZHONG CAO Engineering Nanomaterials and Interfaces for Energy Conversion and Storage



Chemical composition, crystal structure, and defects or impurities all play important roles in determining properties, and thus performance, of the devices that much of our modern-day lives depend on. Guozhong Cao (University of Washington, USA) focuses on the engineering of micro- and nano-structures through surface and interface chemistry to improve the performance of dye sensitized solar cells (DSC), lithium ion batteries, mesoporous carbon supercapacitors, and coherent hydrogen-storing nanocomposites.

"We cannot imagine [what our modern lives would be like] if we didn't have energy," Cao said, referring to the current energy crisis. Solar cell technology continues to evolve: the first generation, based on Si p-n heterojunctions first commercialized 50 year ago, remains the reigning champion of best efficiency. Third generation technology mainly includes dye sensitized solar cells (DSC) and organic photovoltaics (oPVs). While the efficiencies of DSCs remain low at 11%, they may reduce costs, detrimental environmental impacts, and the energy-intensive production in comparison to first-generation technologies. State-of-the-art DSCs are based on a layer of highly porous TiO<sub>2</sub> nanocrystallites (10-20 nm in diameter) 10 microns thick to match the diffusion length of electrons in the host TiO<sub>2</sub>, with a surface monolayer of dye molecules. However, engineering micron-scale particle aggregates and surface chemistries can

enhance light scattering in DSCs to improve photon capture and carrier harvest. Towards this end, Cao investigated structures providing a direct pathway for electron transport by fabricating submicron-sized aggregates of ZnO and TiO<sub>2</sub> nanocrystallites and nanotubes. This hierarchical structure was shown to increase cell efficiency by 3.5% over a commercial sample. To ensure that improved efficiency is indeed due to light scattering, Cao annealed the cell to reduce particle aggregation to form nanocrystallites, and found that the current density was cut in half, proving his theory. Cao also investigated the thickness dependence with his microstructure, and determined that a 40% reduction in thickness can achieve the same efficiency while reducing cost, weight, and fill factors. SEM and XRD indicated unstable ZnO particle surfaces, so surface chemistry was modified during synthesis to stabilize the particle surface, enhancing the efficiency to 6.2%. The employment of atomic layer deposition (ALD) to put down TiO<sub>2</sub> on the ZnO surface produced a 20% efficiency enhancement. Cao also investigated aggregate TiO<sub>2</sub> nanotube photoelectrodes, and found that they produced DSCs with 10% efficiency.

Ideal Li-ion battery performance includes large storage capacity, fast transport kinetics, and cyclic fatigue resistance. All of these performance parameters can be addressed by engineering the interfaces between electrodes and electrolyte at the nano-scale. Nanostructured electrodes can have larger surface areas for easier redox reactions, shorter diffusion distances for faster charge transport, and easier dilation for enhanced energy storage capacity. Cao focused on synthesizing crystal structures and crystallinity in the form of nanorods, core-shell nanocables, and nanotubes to achieve these characteristics. Template-assisted electrochemical fabrication produced pore diameters of 50-400 nm and densities of 10<sup>9</sup>/cm<sup>2</sup> for the synthesis of V<sub>2</sub>O<sub>5</sub> nanorods and V<sub>2</sub>O<sub>5</sub>-Ni core-shell nanocables, of which Cao showed the core-shell nanocables to have higher specific energy and power, as well as lower cyclic degradation. Next, template-less electrochemical growth by was presented, producing structures with large voids and porous walls of stacked nanoparticles. Cao found the storage capacity of these structures to be 148 mAh/g, well exceeding the theoretical limit.

Finally, Cao briefly reviewed his recent work on porous carbon supercapacitors and coherent nanocomposites for H<sub>2</sub> storage, continuing the trend of micro- and nano-structure engineering for enhanced materials properties. Carbon cryogel supercapacitors require fast kinetics (rapid charging and discharging) and minimal cyclic degradation. Cao introduced an electric double layer capacitor to increase the surface area and decrease diffusion distance, and observed significant changes in electrochemical properties by modifying the porous microstructures. He found that, while smaller pores give larger surface areas, the pores must be accessible to ions, and the pore dimensions can be manipulated by altering the surface chemistry. Hydrogen offers zero point-source emissions, is compatible with highly efficient fuel cells, and is naturally abundant, motivating the need for solid-state hydrogen storage materials for compact, safe energy storage solutions. Coherent carbon cryogels present thermal management and mechanical integrity, and may address challenges about dehydrogenation temperature, reversibility, and rapid phase changes required for ideal storage materials.



MRS Executive Director Todd Osman and Olivia Graeve of Alfred University.

#### WOMEN IN SCIENCE LUNCHEON



Dr. Gabriel Guerro (National University) has made quite an academic journey, some of which she shared during the 5th annual Women In Science luncheon. She began by noting that science has no gender and that there is no science "for women." A good idea in science should be respected, regardless of its origin. Despite these ideals, she notes, some discrimination against women and third-world scientists persists. Guerro began with some general information about the status of women in science in Mexico. In Mexico, women comprise 33% of the Research National System (2007), 20% of the Mexican Academy of Science, and 33% of scientific research at the UNAM. Participation of women in science is growing. Guerro is currently serving on an evaluating committee at CNYN-UNAM where, for the first time, there is an even number of men and women on the committee. In a survey, the National Association of Universities in Mexico found that when comparing the number of men and women involved in graduate work across a variety of disciplines, there are an even number in natural science and health sciences, more women in humanities and education, and more men in engineering and technology.



Guerro was born in Puebla, back when it was a small city, near Mexico City. She attended the Alexander von Humboldt German school, where her father was a teacher in math and physics. She decided that she wanted to study chemistry, and entered the University of Puebla. She was encouraged by her physical chemistry professor to go to the new Metropolitan University in Mexico City to prepare her bachelor's thesis under Prof. Ricardo Gomez. Guerro considers this "the first significant event that marked the development of my scientific career, but also the first big problem." After some difficulty in convincing her father to let her go, she presented her thesis and received her bachelor's degree. She continued on to obtain a masters at UAM and a PhD in Strasbourg, France, both degrees on scholarship from CONACyT. She presented her thesis with a cast on her left leg -- two month prior, she had been hit by a car while commuting to her lab by bicycle. Guerro spent the next three years in the Catalysis Group in the chemistry department at UAM-I. In 1985, received an invitation from the director of the Instituto de Fisica to create a catalysis group there, which she accepted. In 1991, she was invited to join CONTACyT as director of graduate programs and scholarships, the first woman with a PhD to occupy a high level position at CONACyT, but left this position in 1993 to return to the Instituto de Fisica.



Guerro's research focuses on catalytic nanostructured materials, mostly metal and oxide nanoparticles, to understand the relationship between catalytic performance and materials properties for applications such as hydrogen production and purification, CO oxidation, and greenhouse gas elimination. "I can hardly imagine my life outside science. I have been lucky. But what I have accomplished in the result of hard work, honesty, and true commitment to my work and my students. Troubles are always there. What is important is to put passion in your work, no matter what is your gender." While taking questions, Guerro also noted that young women are being involved more and more in scientific careers, likely due to the successful diffusion of knowledge to encourage young people to pursue scientific careers. She noted that challenges facing the Mexican female scientist include male-dominated evaluation committees and difficulties associated with publication when all authors have Latin surnames. Guerro was drawn to chemistry because, although she had "a lousy chemistry" teacher at a young age, the field continued to intrigue her and her cousin (who is a chemist) encouraged her studies.

## TALKS

### Symposium 1. Nanostructured Materials and Nanotechnology

#### *Nanoparticle Synthesis and Assembly from Atomistic Simulation Studies*

Nanoparticles are involved in 75% of today's chemical manufacturing processes (tires, toners, food, pharmaceuticals, waste, cosmetics, etc.), and controlled synthesis of these nanoparticles is critical for commercial success. Nanoparticles can be synthesized in a liquid phase, allowing for good control of particle size control but low production rate, or in a vapor phase, allowing for high productivity rates but has significant difficulties in controlling particle size. T. Hawa (University of Oklahoma) investigates synthesis variables on the effects of final nanoparticle physical properties by molecular dynamics simulations.

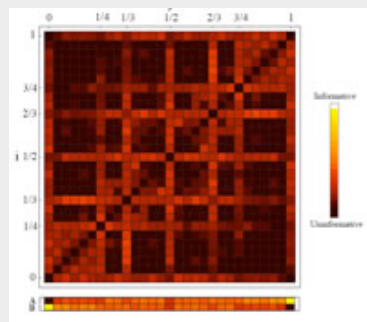
Vapor synthesis begins with a chemical precursor vapor that eventually breaks down and nucleates nanoparticles on a substrate. Final particle size is a balance particle-particle interactions: coalescence, where two particles combine to form one larger particle; and collision, where two particles collide to form one agglomerate. Hydrogen passivation is a common technique to tie up dangling bonds on the surface of nanoparticles and thus reduce their reactivity, and Hawa seeks to understand the effects of passivation on the balance of coalescence and collision during particle synthesis. Particle interaction is proportional to the contact surface area, and this data can be extrapolated to approximate the critical approach energy for the reaction. If the particle energy is lower than the critical reaction energy, then the particles will not react; if the energy is higher, then they will. Hawa also found that increasing particle temperature and decreasing particle size increases the probability of particle reaction. An equation for the coalescence time was obtained, and Hawa finds that hydrogen stays on the surface of the particle throughout coalescence. Effects of hydrogen passivation on the dynamics of sintering of individual particles and particle aggregates, as well as the stability of nanoparticles facets (from which the final particle shape can be predicted) were also investigated and compared to experimental results in the literature.

Nanoparticle assembly is one of the largest challenges in nanotechnology-based device development, and Hawa investigated electrostatic-directed assembly, both experimentally and by modeling. Electrostatic-directed assembly, where substrates have charged patterns that attract oppositely-charged particles, is a common and robust assembly technique. Hawa employed a GaAs substrate with regions of Si- and Zn-doping which formed p-n junctions. When a voltage was applied across the junction, charged particles were observed to collect on oppositely-charged regions of the substrate. Particle trajectory over like-charged substrate regions was modeled to determine the effect of the electric field to control particle deposition location.



### Symposium 2. Theory and Computer Simulation of Materials

#### *The Prediction of Crystal Structure by Combining Machine Learning Knowledge Methods with First Principle Energy Methods*



Novel chemistry can result in crystal structures that we can't currently predict-- "This is a serious problem," comments Gerbrand Ceder (Massachusetts Inst. Tech.). Crystal structures are difficult to predict because there are many possible structures competing for dominance, in addition to an inherently complicated energy landscape. Current prediction techniques find the ground-state crystal structure by beginning with an energy model and then directly optimizing the coordinates to select the system with the minimal ground state energy; yet, physics is only used in the initial energy model, whereas injecting physics into the energy landscape can allow smarter searching through statistical learning. Ceder employs structure assignments from large experimental databases to "learn" the underlying physical rules that governs crystal structures, and then captures this knowledge in a mathematical model by building a probability density for structures to coexist in a chemical system-- in other words, systems with the same underlying physics. Finally, he uses this knowledge-optimized model to predict crystal structures through density functional theory (DFT), which he showed to be accurate in predicting crystal structures ~97% of the time for 80 binary systems.

Ceder used his mathematical model to compute a candidate list for the crystal structure of  $\text{AgMg}_3$  which is known as a compound with an unknown structure. Cu3P-type structure, an uncommon structure that occurs only five times in all binary

alloys, was computed to be the most likely structure. Cross-validation predictions were run on more than 5000 structures, showing 95% confidence that the correct structure is on the knowledge-optimized 5-member candidate list, from which DFT calculations can be run. For ternary oxides, 15 crystal structures are required on the candidate list to reach 95% confidence, and  $K_2MgO_2$  was shown as an example. Errors in the ICSD database were also presented, such as CsIO<sub>3</sub>: Ceder predicts that the correct crystal structure is not perovskite, as listed in the ICSD database, but in fact a perovskite-related crystal structure whose ground state is 300 meV lower. With the power to rapidly predict crystal structures, Ceder reinvestigated computationally all binary and ternary systems, and found 3999 new compounds that can be used for a variety of applications, such as one that is currently being investigated as a radiation detector. Finally, results from the model were extrapolated to predict the existence of 2800 so-far undiscovered ternary oxide crystal structures.

#### *The accuracy of first principle methods in predicting thermodynamic properties of metals*

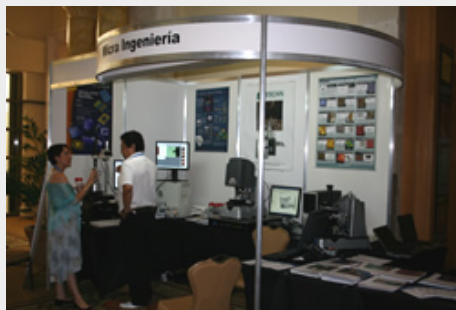
T. Hickel (Max Planck Intitut, Germany) introduced the Ni-Mg-Ga system to illustrate the shape-memory effect through a martensitic phase transition, where ferromagnetic phase is stable below 376K, as a motivation for computing thermodynamic properties by ab-initio predictions. He employed a standard quasiharmonic approximation with density functional theory and convergence tests, and showed that the typical numerical accuracy for a calculated free energy was 1 meV. Phonon dispersion, free energy, expansion coefficients, and heat capacity trends were compared to those from Calphad experimental data for Cu, Ag, Rh, and Pd. Hickel noted the importance of the inclusion of electronic excitations in Rh and Pd for improved heat capacity prediction accuracy. Next, magnetic Fe was investigated, requiring the consideration of magnetic excitations through a Random Phase Approximation to predict the free energy. Al had excellent agreement with Calphad for all properties except for heat capacity at higher temperatures. For this, Hickel included all known contributions (quasiharmonic, vacancies, electronic, and anharmonic) and simplified the calculation to reduce computation time, finding that only the vacancy contribution can explain the observed discrepancy with experiment. Ni-Mg-Ga was then investigated, with focus on discrepancy in the phonon dispersion for the austenite cubic phase. Hickel found that the martensitic transition has a vibrational origin and the premartensitic transition has a magnetic origin. To match experimental data, reduction of magnetization was necessary to stabilize the austenite cubic phase.



#### Symposium 4. Materials Characterization

##### *Spectroscopic and Thermodynamic characterization of a collapsing gas bubble*

In this poster, N. Navarrete (Instituto de Ingenieria) investigated the luminescence of a conical bubble collapse. Cavitation is the formation of, and activities of, cavities in a liquid, and the collapse of such cavities can have enormous consequences in a variety of materials systems and processes (emulsification, degassing). Initial bubble dimensions are controlled by inlet gas pressure through a U-tube. The gas was a mixture of argon and xenon, the bubble formed from liquid 1, 2-propanediol with small variable quantities of sulfuric acid to alter the chemical properties at the meniscus interface. A high-energy, quick flash of light (1.3 mJ, 30-1000 microseconds) was collected and analyzed by a spectrometer. Navarrete observed line emissions from OH, Na<sup>+</sup>, K<sup>+</sup> and Swan Lines, suggesting that light emission is due to chemiluminescence and by collisions between gas molecules and free electrons generated by the chemical reaction.



#### Symposium 8. Fracture Mechanics

##### *Scaling In Concrete Fracture Surfaces*

Concrete is a humble but complex material, said M. Hinojosa of the Universidad Autónoma de Nuevo León. The microstructure of concrete is interesting in that it contains a variety of random features over a wide range of length scales, which is useful for testing the scaling properties of fracture surfaces. The fracture surface in any material is the trace left behind as the crack front advances through a heterogeneous medium. Failure in concrete occurs by the culmination of progressive damage involving complex interactions between multiple defects and microcracks. In the present work, specimens of concrete were prepared and broken in bending and compression tests. The authors characterized the macro and microstructure, and carried out fractographic as well as self-affine analysis of the fracture surfaces using optical, electron and atomic force microscopy. Stylus profilometry and a 3D scanner were used to record height profiles. The scaling properties were investigated using the Hurst method and the heightheight correlation function, including the values and statistical distributions of roughness exponents which tended to fluctuate around 0.82 which is considered as universal. However, there was not a single local roughness exponent. Possible relationships among the scaling parameters and the characteristic sizes of the microstructural elements were also investigated. The fracture of cement is obviously very complex, and the roles and contributions of different

microstructural elements need to be determined.

#### Symposium 11. Composite and Hybrid Materials

##### *Growth of Diamond Films From Tequila*



Mexico is of course well known for Tequila, the most popular alcoholic drink here. Javier Morales of Universidad Autónoma de Nuevo León and UNAM-Querétaro described (in Spanish) an unusual use for Tequila - creating nanocrystalline diamond films. It so happens that Tequila has a carbon:oxygen:hydrogen ratio that lies within the diamond growth region of the C-H-O phase diagram. Tequila, which is made from the agave plant, has about a 38-43% alcohol content. Morales and his co-workers used pulsed liquid injection chemical vapor deposition (PLICVD) to form the diamond films on silicon and stainless steel substrates. The liquid Tequila was heated to 280 °C to transform it into a precursor gas. In a reaction chamber, the gas was heated to 800 °C for the deposition, resulting in solid diamond crystallites of about 100-400 nm. As is to be expected, nucleation and growth were a function of the surface quality of the substrates. While the technique works, the question is why use Tequila? The answer is that even very cheap Tequila appears to work well and this could be an inexpensive method to produce diamond films. The work has received significant interest in the press such as [this report from the BBC](#).

[\[View relevant paper at Arxiv\]](#)

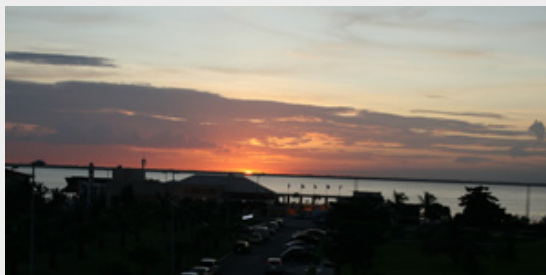
#### Symposium 13. Advances in Semiconducting Materials

##### *Catalysts in the form of thin films of TiO<sub>2</sub> with Au nanoparticles*

Environmental pollution by volatile compounds is a major international problem, motivating the development of photocatalytic materials to decompose volatile pollutants by simple light exposure. TiO<sub>2</sub> is a photocatalyst for oxidation of organic compounds in water and air, but is difficult to separate from a liquid solution, so Flor Palomar (University of Nuevo Leon) used a dip-coating method to deposit a thin film onto a solid substrate. TiO<sub>2</sub> films were deposited by dip-coating a glass substrate in a sol-gel at a constant speed, followed by an annealing step. Au nanoparticles (NPs) have been reported to enhance the photocatalytic activity of TiO<sub>2</sub>, so Au NPs were sprayed over the TiO<sub>2</sub>-coated substrate. Au NPs were obtained from HAuCl<sub>4</sub> solutions, where the NP diameter can be tuned by the solution pH. Optical microscopy proves that Palomar was able to optimize the dip-coating conditions to obtain high-quality films with few cracks. X-ray diffraction confirms an anatase phase of the TiO<sub>2</sub> films. UV-Visible spectroscopy determined the bandgap of the films to be approximately 3.6 eV, in agreement with published values. Atomic force microscopy shows that Au NPs were 15-25 nm in diameter. Photocatalyst tests indicate that the TiO<sub>2</sub> film decomposes methylene blue more completely than Au NP-enhanced TiO<sub>2</sub> composites (95% versus 70% decomposition upon UV irradiation, respectively), which Palomar attributes to the blocking of photocatalytic-active sites of TiO<sub>2</sub> films by Au NPs.

##### *A study of piezoresistive coefficients of carbon fiber cementitious composites under shear loads*

Earthquakes produce shear load failures in buildings, but the level of damage below visible cracks is unknown. Carlos Vinajera Reyna (Autonomous University of Yucatan) is working to create a smart building that can assess the internal shear loads that may lead to disastrous failures with carbon fiber-reinforced concrete. Although modern concrete has been in use since 1824, carbon fiber-reinforced concrete only became available in 1988, and utilized as an internal stress probe in 1996. Carbon fibers embedded in the concrete act as piezoelectric sensors, transmitting mechanical stresses as changes in electrical resistance along the fibers. Vinajera experimented with electrical fluxes along the x, y, and z directions through concrete blocks. Resistivity was found to vary under both compression and shear stresses in a linear fashion in all three directions, with maximum sensitivity when measured across the width of the block. Results show that initial cracks can be sensed long before plastic behavior and final failure. Vinajera is working to develop MEMS devices to interface directly with the carbon fibers, enabling wireless sensing.

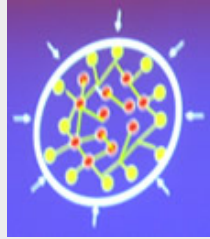


#### Symposium 11. Beams and Materials: Ion Beams

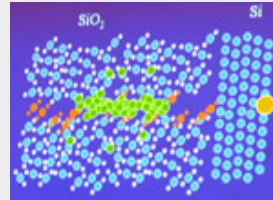
##### *Ion Beam Formation And Modification Of Elemental Metallic Nanoparticles*

In the first talk in symposium 20, D.J. Sprouster of the Australian National University described the formation and modification of elemental metallic nanoparticles (NPs) using ion beams, focusing on the application of synchrotron-based techniques to characterize finite-size effects and ion-irradiation-induced changes in NP structure, shape and size. Crystalline metal NPs, such as Co, Cu, Pt and Au, with diameter ~10 nm or less can be easily formed by ion implantation and thermal annealing. X-ray absorption spectroscopy (XAS) measurements were used to show that the non-negligible surface area to volume ratio reduces the average coordination number and bondlength yet enhances structural disorder. Ion irradiation in either the nuclear or electronic energy loss regimes is an effective means of modifying the structure, shape and/or size of elemental metallic NPs.

Sprouster first discussed the effects of "low" ion energy, which in this study is in the 9 MeV



regime. At low ion energies where elastic stopping processes dominate, irradiation of embedded metallic NPs can induce a crystalline-to-amorphous phase transformation. The enhanced sensitivity of NPs to ion irradiation results from greater structural disorder in the crystalline unirradiated state combined with the presence of an amorphous matrix to stabilize the amorphous phase induced in the NP by irradiation, according to Sprouster. These measurements of the inter-atomic distance distribution of amorphized NPs are consistent with theoretical predictions for that of a bulk amorphous metal.

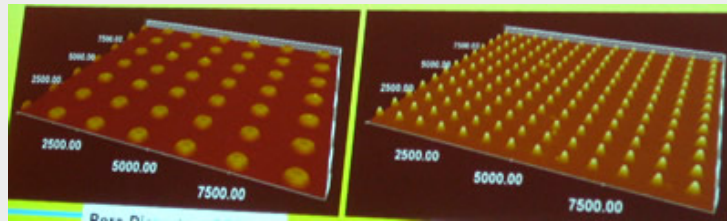


On the other hand, high energy ion irradiation, wherein inelastic stopping processes dominate, yielded a spherical-to-ellipsoidal shape transformation in embedded metallic NPs above a threshold diameter. NPs of lesser size remained spherical upon irradiation and after initially increasing in diameter, these smaller NPs progressively dissolved in the matrix. The larger deformed ellipsoidal NPs aligned with their major dimension parallel to the incident ion beam direction. Using small angle x-ray scattering measurements, the minor dimension was shown to saturate at an electronic-energy-loss-dependent value while the major dimension exhibited a broad distribution consistent

with that of the unirradiated spherical NPs. The threshold diameter for NP elongation was comparable to the saturation value of the minor dimension. This was correlated with the diameter of the molten ion track induced in the matrix by electronic energy loss. The results point to a transformation mechanism driven by NP melting followed by flow of molten material confined by the ion track.

#### *Ion Beam Nanopatterning Of Metal Nanoparticles Towards Plasmonics*

Naoki Kishimoto (National Institute for Materials Science, Japan) described the use of ion beams to pattern metallic nanoparticles on the nanoscale. One major motivation for this is plasmonics. Metal nanoparticles embedded in insulators have attracted attention for plasmonic applications. For future plasmonic devices, spatial control of nanoparticles, on a micro- or nano-scale, is vital. Ion beam-based techniques offer possibilities for robust spatial control of nanoparticles, especially for immiscible binary systems. Since ion implantation is inherently good at depth control of solutes or nanoparticles, additional lateral control may lead to 3D control of nanoparticles.



There are several issues relating to patterning of nanoparticles, divided into the nanoparticle formation process and spatial distribution. Kishimoto described self assembled 2-D distribution of nanocrystals for 60keV  $\text{Cu}^+$  implanted a- $\text{SiO}_2$ . Surface plasmon band tuning can be achieved by varying the ion species or by changing the substrate. He then discussed nanopatterning of radiation-induced swelling of crystalline  $\text{SiO}_2$  substrate using a mask. Si stencil masks with various pore diameters were formed and ion irradiation was carried out with the masks on the  $\text{SiO}_2$ . An anodic porous alumina mask was also formed and used. The researchers demonstrated successful patterned implantation of nano-sized periodic spots, down to 50 nm in diameter. The porous alumina mask was subjected to radiation-induced deformation, enhanced by electronic excitation effects. It was demonstrated that the metal-nanoparticle composites fabricated by the ion implantation showed pico-sec response of nonlinear optical susceptibility. In summary, ion beam-based methods are promising for 3-D control of nanoparticles, by the addition of lateral control using ion implantation. Also, masked implantation appears to be one the feasible options for lateral control.

#### SCENES FROM THE CONFERENCE DINNER





ABOUT THE MEETING SCENE

- The Meeting Scene reports are compiled and edited by [Dr. Gopal Rao](#), Web Science Editor, Materials Research Society. Contributors include Megan Brewster (Massachusetts Institute of Technology) and Tara Washington (University of Florida).



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