

### Nanocrystalline Alumina Synthesized with Use of Egg Whites

Nanocrystalline ceramic particles are often prepared using sol-gel techniques, wherein precursor solutions containing metal ions are transformed to an amorphous gel by a change in the pH, temperature, or ionic strength of the solution. S. Dhara of the Materials Science Centre at the Indian Institute of Technology in Kharagpur has demonstrated a novel

approach to sol-gel processing. Rather than producing the gel by treating the precursor solution, the metal nitrate is dispersed in an aqueous matrix of egg whites (ovalbumin). Heating this simple mixture leads to well-crystallized, nano-sized powders of  $\gamma$ -alumina.

As reported in the July issue of the *Journal of the American Ceramic Society* (p. 2003; DOI: 10.1111/j.1551-2916.2005.00382.x), the amorphous gel was formed by first mix-

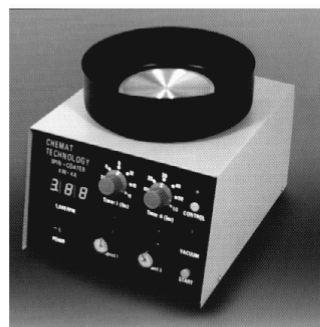
ing fresh egg whites in water, then adding aluminum nitrate dropwise to obtain a 3:2 ratio of  $\text{Al}(\text{NO}_3)_3$  precursor-to-ovalbumin solution. The mixed solution then is allowed to gel by resting for 2 h and is subsequently dried for 10 h at 80–150°C. The result is a yellowish mass that is then crushed into a fine powder.

X-ray diffraction (XRD) analysis showed that the powder is initially amorphous and that 2 h heat treatments crystallized the material into  $\gamma$ -alumina. Crystalline alumina peaks were observed for annealing temperatures as low as 330°C, although diffracted peak intensities increase at higher temperatures. These results were supported by differential thermal analysis (DTA) curves showing exothermic peaks at 330°C, 560°C, and 630°C. These peaks indicated crystallization of the precursor and subsequent degradation of the ovalbumin.

Particle size analysis using XRD and transmission electron microscopy (TEM) confirmed that these powders are nanocrystalline. Crystallite sizes were estimated to be in the range of 15–25 nm using the Debye-Scherrer formula for x-rays and in the range of 15–80 nm from TEM micrographs. Thus, said Dhara, ultrafine ceramic particles can be obtained from a gel matrix of metal ions dispersed in an aqueous solution of egg whites.

ANDY FRANCIS

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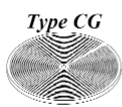
### Adjustable Speed

#### Stage 1

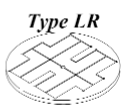
500 to 2500 rpm  
2 to 18 seconds

#### Stage 2

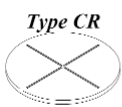
1,000 to 8,000 rpm  
3 to 60 seconds



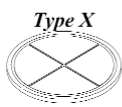
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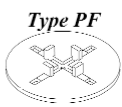
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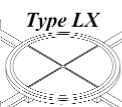
Type CR



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Type PF

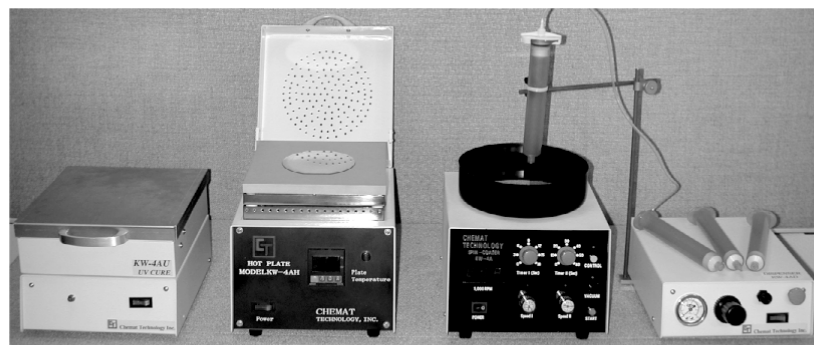


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### SB Explains CNFET Performance Dependence on Diameter and Metal Contact

Due to exceptional electrical properties and high carrier mobility, there has been significant interest in exploring the potential of carbon nanotubes (CNTs) as building blocks in future applications of nanoelectronics, such as the use of carbon nanotube field-effect transistors (CNFETs) as Schottky Barrier (SB) devices. However, CNFETs have yielded large on-current ( $I_{on}$ ) variations ranging from  $10^{-5}$  A to  $10^{-8}$  A with similar device geometries, and there has been no clear conclusion as to the origin of these variations. Recently, Z. Chen from the IBM T.J. Watson Research Center in New York, J. Knoch from the Institute of Thin Film and Interfaces and the Center of Nanoelectronic Systems in Jülich, Germany, and their colleagues have demonstrated that the optimum performance of the CNFET devices depends upon a combination of the diameter of a single-walled CNT and the type of metal contact and have produced a model that accounts for the observed current variations.

As reported in the July issue of *Nano-Letters* (p. 1497, DOI: 10.1021/n10508624), the researchers fabricated over a hundred

CNFET devices on *p*-doped Si substrates covered with 10 nm of SiO<sub>2</sub>, with a channel length of 300 nm, using three different source/drain metals (palladium, titanium, and aluminum) in direct contact with nanotubes, which had diameters of 0.6–1.5 nm. The Si substrate was used as the back gate

while the SiO<sub>2</sub> was the gate dielectric. Single-walled CNTs grown by laser ablation were used for this study. The researchers correlated the device on-currents with the nanotube diameters and observed that *p*-type Pd-CNFETs (dia. >1.4 nm) have the highest  $I_{on}$  and Al-CNFETs have

the lowest  $I_{on}$ , which follows a trend that would be expected from the clean-metal work function, but the researchers indicate that their results are not “directly correlated with the macroscopic clean surface work function values.” Furthermore, the researchers observed larger variations in

### Effects of Size and Surface Coating on Cytotoxicity of Nickel Ferrite Explored

Nanoparticles have attracted great interest recently for nanotechnology applications and, in particular, for biomedical applications, such as drug delivery and as biosensors. However, one area of concern for nanoparticles is toxicity and the consequent health effects of the particles, especially for *in vivo* applications. While it is clear that particle size and surface coatings affect chemical and physical properties of nanoparticles, corresponding effects on toxicological properties are as yet unknown. G.M. Chow, H. Yin, and H.P. Too of the National University of Singapore have unraveled some of the toxicological effects of nickel ferrite nanoparticles, both in terms of particle size as well as the presence of oleic acid as a surface coating. Their results will be reported in the October issue of *Biomaterials*; the article is currently available online (DOI: 10.1016/j.biomaterials.2005.02.036).

The researchers used ball milling to prepare uncoated nickel ferrite nanoparticles in two diameters, 150 nm ± 50 nm and 10 nm ± 3 nm. Using a polyol method, the research team also prepared samples with one or two layers of oleic acid coating, making the particles hydrophobic (see Figure 1). Cytotoxicity of the nanoparticles was measured using mouse neuroblastoma (Neuro-2A) cells and through use of a specific test that measures succinate dehydrogenase mitochondrial activity. In cytotoxic conditions, cell membranes were compromised and distinct cell shapes were no longer visible.

The size of uncoated nickel ferrite nanoparticles was found not to be a significant factor for cytotoxicity, since there were no toxic functional groups on the particle surface, although smaller particles were slightly less toxic than larger ones. The uncoated particles have a hydrophilic surface. When oleic acid molecules were present as a monomer, they were also not cytotoxic. However, when they were present as micelles or when they were coated on the nanoparticles, cytotoxicity was clearly observed. Particles with one layer of oleic acid (hydrophobic –CH<sub>3</sub> functional group) were more cytotoxic than particles with two layers of oleic acid

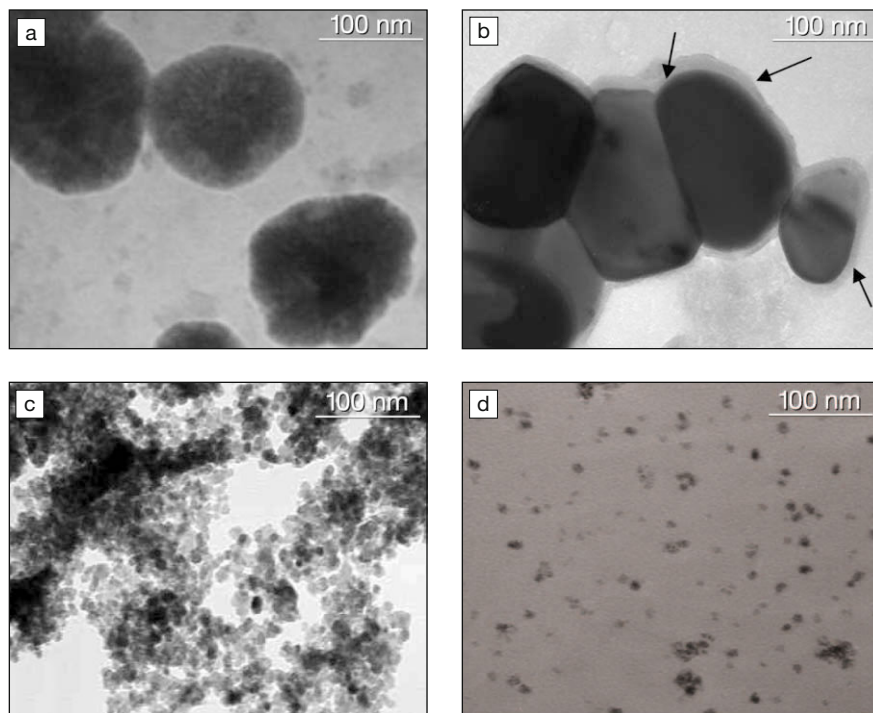


Figure 1. Transmission electron micrographs of nickel ferrite particles: (a) large particles (150 nm ± 5 nm diameter) prepared by ball milling; (b) large particles prepared by the polyol method; (c) small particles (10 nm ± 3 nm diameter) prepared by ball milling; and (d) small particles prepared by the polyol method. Arrows in (b) indicate the surfactant layer. Reprinted with permission from *Biomaterials* **26** (2005), p. 5818; DOI: 10.1016/j.biomaterials.2005.02.036. © 2005 Elsevier.

(hydrophobic –COOH functional group). The researchers said, therefore, that a hydrophobic surface was more cytotoxic than a hydrophilic surface. The coated nanoparticles also had aligned oleic acid molecules, which resulted in greater cytotoxicity.

The smaller particles were found to be less toxic than the larger ones in medium concentrations (20–100 µg/ml) of the oleic acid for both one- and two-layer surface coverage. For higher concentrations, small and large particles were equally cytotoxic. This size effect could be related to the surface energies and surface interaction areas that were size-dependent, said the research team. The surface energy of small particles is higher than that of large particles. This may have resulted in

different effects on surfactant absorption and conformation altering the chemical reactivity of the surfactant. Also, the effective interaction area of a large particle with a cell is greater than that for a small particle, thus resulting in a stronger stimulus on the cells, said the researchers.

The broad conclusion of the study, said the research team, is that the effects of intrinsic size and surface properties, both dependent on the synthetic and processing conditions, need to be cautiously considered in evaluating the biosafety of nanoparticles. In terms of future studies, the group is planning to investigate the interfacial interactions of nanoparticles and cells, particularly the mechanisms of cell degradation and death.

GOPAL RAO

$I_{on}$  for smaller nanotubes than for larger diameter nanotubes for all three types of metal contacts. To account for their results, the researchers developed a model that "describes the CNFET as a gated nanotube channel in contact with two reservoirs that are a result of the weak coupling between the metal and the nanotube in the overlap region," thus incorporating a tunneling barrier as part of their extended SB model.

By comparing other sets of data from known reports of CNFETs fabricated from CNTs grown by arc discharge and chemical vapor deposition methods with the results of their laser-ablation-grown CNFETs, the researchers confirmed that the local contact quality is not the major contribution to the observed current variation using nanotubes of the same length and that  $I_{on}$  is independent of the nanotube growth source. The results of the extended SB model agreed well with the data from this study as well as the data from the previous experiments. Qualitative band diagrams for CNFETs further showed that the injected current decreases exponentially with increasing barrier height, which is inversely dependent on the nanotube diameter. Therefore, the researchers con-

cluded that the variation in nanotube diameter is the main cause of the variations in current from device-to-device.

KINSON C. KAM

### Heterogeneous Gold-Based Catalyst Reduces CO in Reformate PEFCs

In the past few years, the automotive industry has undertaken significant efforts to develop electric vehicles powered by polymer electrolyte fuel cells (PEFCs). These fuel cells employ an electrochemical process to oxidize  $H_2$  while providing electricity and water as reaction by-products. The main fuel for PEFCs is either stored  $H_2$  or a reformate fuel, which is a hydrogen-rich gas mixture obtained after conversion of an organic fuel such as natural gas in a catalytic reformer. However, the performance of PEFCs is hindered by residual CO that is usually present in reformate fuel. CO reduces the capability of the Pt anode of ionizing hydrogen, which is the start of the electrochemical process. P. Landon of Cardiff University, A.A. Herzing of Lehigh University, S.E. Golunski of the Johnson Matthey Technology Centre, and their colleagues have now developed a Au/ $Fe_2O_3$

catalyst to remove CO from reformer fuel. They reported in the May issue of *Chemical Communications* (DOI: 10.1039/b505295p) their process of testing the catalyst in the actual conditions of temperature and gas mixture composition seen in PEFCs.

The researchers said that changes in the coprecipitation and calcination procedure influenced selectivity and performance of the catalyst. After calcination at temperatures lower than  $550^\circ C$ , the catalyst oxidized both  $H_2$  and CO. Calcination for 3 h at  $550^\circ C$  resulted in selective oxidation of CO, and calcination at higher temperatures destroyed the catalyst activity. Transmission electron microscope observations of the catalysts obtained at the different calcination temperatures revealed extensive grain growth, suggesting that the consequent reduction of surface area is the cause of the reduction of activity at calcination temperatures higher than  $550^\circ C$ , said the researchers. Furthermore, Au (4f) x-ray photoelectron spectra for samples calcined at different temperatures revealed the formation of metallic gold particles at temperatures higher than  $550^\circ C$ , which are inactive for CO and  $H_2$  oxidation, the re-



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