P.N. Kumta, professor of materials science and engineering at CMU, said, “We chose TiN because of its electrical conductivity and mechanical strength, combined with electrochemical inertness to Li and chemical inerntness to both Li and Si. It seemed to be a very promising material, but of course we first had to find a suitable synthesis process for composites.” His group explored high-energy mechanical milling of Si and TiN, as this technique can generate amorphous, metastable, and nanophase structures.

Preliminary experiments showed that a 1:2 molar ratio of Si:TiN exhibited the best electrochemical properties. X-ray diffraction in combination with energy-dispersive x-ray analysis demonstrated that the composites consisted of nanocrystalline TiN with amorphous Si nanoparticles homogeneously distributed throughout the matrix after as little as 6 h of milling time. Increased milling times led to a decrease in the initial capacity of the composites.

“We are not sure yet what causes this decrease,” said Il-seok Kim of CMU. “It could be because the Si nanoparticles are embedded or enclosed by TiN during milling, which would prevent their reaction with Li.”

Composites obtained after milling for 12 h exhibited stable capacities of 300 mAh/g, indicating that a large fraction of Si is inactive (~44%). Although the gravimetric capacity is lower than that of conventional carbon, this value corresponds to a 30% higher volumetric capacity, reflecting the promising nature of the composites. Cycling data showed an irreversible capacity loss (~30%) during the first cycle.

“We are still trying to understand the exact reason for this loss. It could be due to the formation of a Li-containing passivation layer or surface oxidation,” said G.E. Blomgren. No cracks were present on the electrode surfaces after 30 cycles, and no changes in morphology were observed, indicating good microstructural stability.

Kumta said, “We have shown that Si/TiN nanocomposites are promising candidates as anodes for Li-ion batteries. But there is still a lot of optimization that needs to be done. We are currently working on detailed structural and electrochemical studies.”

Cora Lind

Scanning Tunneling Microscope Imaging of Cu on Cu(111) Yields Estimates of Surface-Mediated Interaction Potentials

A quantitative study of the long-range interaction between single copper adatoms on Cu(111) reveals significant changes in the growth of Cu/Cu(111) at low temperatures. As reported in the October 2 issue of Physical Review Letters, an international research team from Berlin University and the Paul Drude Institute for Solid-State Electronics in Berlin, Germany, and Chalmers University of Technology and Göteborg University in Sweden have experimented with a long-range interaction mediated by a two-dimensional nearly free electron gas. Their experiments were performed with a scanning tunneling microscope (STM) operated at low temper-
ature (9–21 K) using electrochemically etched tungsten wires for analyzing tips, in order to record the diffusion of single copper adatoms on the close-packed Cu(111) substrate. They prepared their samples by evaporating ~0.01 monolayer of copper at 15 K, a temperature at which single copper atoms are mobile, on Cu(111) substrates that were previously cleaned by several sputter and anneal cycles.

The researchers analyzed STM images showing two Cu adatoms separated by distances of up to 70 Å. More than 65,000 Cu spacing distances from a total of 3400 images were analyzed. The researchers established an oscillatory behavior of the potential energy, with a periodicity of \( \lambda_F/2 \), and an envelope that decays as \( 1/d^2 \) for large separation \( d \). The method used to determine the interaction potential was to extract it from the measured pair distribution, obtained from the time-dependence of the distance between two adatoms, correcting for geometrical effects inherent to the measuring process. The discrepancies between their experimental results and the previous theoretical descriptions of the phenomenon will help to re-evaluate the assumptions made as well as neglected terms in theoretically describing the potential energy between adatoms, and will also help to understand the growth of Cu on Cu(111) at low temperatures.

Claudiu Muntele

Nucleation and Growth Mechanism Causes Switching of Exchange Bias in Double-Superlattice System

At interfaces of ferromagnetic and antiferromagnetic films, exchange coupling results in a process called “exchange bias,” in which the ferromagnetic hysteresis loop shifts. Investigators at Argonne National Laboratory, studying the exchange bias in a “double-superlattice” system, have observed reversal of direction (“switching”) of the exchange bias. They attribute the switching mechanism to a breakdown into domains of the antiferromagnetically coupled (AF) superlattice by nucleation and growth, followed by complete reversal of magnetization in the layers. They reported this result, which challenges the uniform rotation model, in the October 2 issue of Applied Physics Letters.

Using the Fe/Cr(211) system for their study, researchers S.G.E. te Velthuis, J.S. Jiang, and G.P. Felcher combined a ferromagnetically coupled (F) superlattice consisting of 50 Å Fe and 20 Å Cr with an AF superlattice of 14 Å Fe and 11 Å Cr to form a double superlattice with the layer sequence \( \text{Fe}(50\,\text{Å})/\text{Cr}(20\,\text{Å})^{59}/[\text{Fe}(14\,\text{Å})/\text{Cr}(11\,\text{Å})^{20}] \). A 20-Å layer of Cr was sandwiched between the F and AF superlattices to provide a ferromagnetic intersuperlattice coupling. An artificial exchange bias (uniaxial anisotropy) was built into the system by epitaxially growing the sample onto a single-crystal MgO(110) substrate.

Magneto-optic Kerr effect measurements were performed around two critical values (~406 Oe and ~447 Oe) of the turning field in the field loop, \( H_{\text{min}} \). The hysteresis loop obtained for the ~406 Oe measurements is narrow, with a bias around ~38.5 Oe; the magnetization in the F superlattice reverts to its original orientation at ~33.6 Oe. However, with \( H_{\text{min}} \) at ~447 Oe, the F superlattice magnetization does not revert to its original orientation until 40.7 Oe, indicating that the AF superlattice has reversed its direction. Polarized neutron reflectivity measurements confirm the reversal of the AF superlattice. The fact that the bias direction switches at a value of ~447 Oe, which is much lower than the 14-kOe field required to saturate the AF superlattice, or the 2-kOe field needed to initiate spin-flop transitions, indicates that a different mechanism is at work. A nucleation and growth scenario is consistent with these results; further work is under way to determine the field-dependence of the magnetic layer structure more precisely.

Tim Palucka

Thin Films of \( \alpha-\text{Al}_2\text{O}_3 \) Result from the Use of an Alternative Anhydrous Solution

Thin films of crystalline \( \alpha-\text{Al}_2\text{O}_3 \) are obtained through an innovative sol-gel process developed by Naoufal Bahlawane and Tadahiko Watanabe at the Kyushu National Industrial Research Institute in Japan. This process has the advantage of reducing the transformation temperature for \( \alpha-\text{Al}_2\text{O}_3 \), gaining more control over the final conditions of particle shape and size. Another advantage of this process is the accomplishment of a direct conversion to \( \alpha-\text{Al}_2\text{O}_3 \) with the use of an anhydrous solution.