

Nanometer-scale Science and Technology Room 2016 - Session NS-ThA

Nanoscale Material Processing

Moderator: S. Jesse, Oak Ridge National Laboratory

2:00pm NS-ThA1 Controlled Chemical Mechanical Polishing of Polysilicon and Silicon-Dioxide for Nanostructures, *V. Joshi, A.O. Orlov, G.L. Snider*, University of Notre Dame

In this paper, we report experimental results of silicon dioxide (SiO₂@sub 2@) and polysilicon chemical mechanical polishing (CMP) to produce nanoscale features with very smooth surfaces. The sizes of the features polished ranged from 20 to 500 nm. Nanostructures were defined by e-beam lithography and reactive ion etching (RIE). For polysilicon polishing, the nanostructures were defined in ZEP-520A (positive tone e-beam resist) and pattern was transferred to the oxide substrate through RIE (CHF₃@sub 3@/O@sub 2@) plasma. These etched nanostructures (70 nm deep trenches) were conformally filled with LPCVD polysilicon, and polished using a Logitech CDP system. The slurry used was Semi-Sperse P1000 from Cabot Microelectronics. Similarly for oxide CMP study, nanostructures were patterned on Si substrate using HSQ (negative tone e-beam resist) as the etch mask. These nanostructures in Si (~100 nm height) were covered with thick layer of PECVD SiO₂@sub 2@. Polishing was done to planarize the sample and expose the Si structures. CMP-1150 slurry from Ektech was used for oxide CMP. The polished structures were studied using SEM (cross-sections) and AFM (surface). We report controllable CMP to realize ~20 nm thick layers after polishing. The RMS roughness of the polished surfaces was ~ 0.3 nm. Better control of the CMP process (2 nm/min removal rate) was demonstrated by using diluted slurry or pure DI water as the CMP slurry.

2:20pm NS-ThA2 Properties of Carbon-Based Nanostructures Grown by Low-Pressure Plasma Enhanced Chemical Vapor Deposition, *J.B.O. Caughman, L.R. Baylor, R.A. Kisner, K. Korsah*, Oak Ridge National Laboratory

The role of the plasma in the growth of carbon-based nanostructures is being determined by relating plasma conditions to the physical and electrical properties of carbon nanofibers and nanosheets. Nanofibers have a cylindrical cone-like structure while nanosheets have a planar sheet-like structure. Forests of vertically aligned nanofibers and nanosheets have been grown using an inductively coupled plasma source operated from 30 to 100 mTorr. The plasma is composed of hydrogen and either acetylene or methane as the carbon source. The plasma conditions are determined by using mass spectroscopy and optical emission spectroscopy. Dilute mixtures of carbon containing gases in hydrogen lead to well-formed nanostructures, while excessive carbon in the plasma leads to an increase in amorphous carbon deposition. The presence of a nickel catalyst layer prior to deposition leads to nanofiber growth, while no catalyst leads to nanosheet growth. Substrate bias plays an important role in the formation of nanofibers (by controlling the physical etching component during deposition), while having a detrimental effect on nanosheet growth. Field emission from the material, as measured with a scanning probe, is found to be strongly dependent on the details of the nanostructure. Sparse forests or well defined nanosheet edges give the lowest threshold fields. Details of the effect of plasma properties and substrate conditions (bias and temperature) on the electrical/physical properties of the nanofibers will be presented.* @FootnoteText@ *Oak Ridge National Laboratory is managed by UT-Battelle, LLC, for the U.S. Dept. of Energy under contract DE-AC05-00OR22725.

2:40pm NS-ThA3 Prospects and Processing of Soft Materials, *G.G. Malliaras*, Cornell University

INVITED

During the past two decades dramatic advances have been achieved in the performance of organic semiconductor devices. Light emitting diodes, transistors, solar cells, and several other devices can now be fabricated from organic semiconductors and are at various stages of commercialization. This fast-paced progress, which highlights the technological potential of soft materials, is largely a result of advances in our ability to deposit and pattern high quality films. To make this possible, traditional deposition and patterning techniques had to be adapted, and new ones had to be developed. Moreover, the integration of organic semiconductors with biologically-relevant materials, which share some of the processing challenges, promises the development of novel sensors with

unmatched capabilities. I will discuss recent advances in the processing of soft materials and the opportunities they have created.

3:20pm NS-ThA5 Synthesis of Suspended Single-Walled Carbon Nanotubes on Tips of Silicon Nanostructures Fabricated by Plasma Etching, *C.H. Weng, H.C. Su, W.Y. Lee, C.H. Tsai, K.C. Leou*, National Tsing Hua University, ROC

Suspended single-walled carbon nanotubes (su-SWNTs) have attracted a great deal of attention recently. This is because (1) the perturbation from substrates can be minimized to allow probing the natural properties of SWNTs; and (2) nano-devices using su-SWNTs exhibit enhanced performance. In this work, we present a method to synthesize su-SWNTs selectively suspended on tips of silicon based nanostructure (Si-n) templates formed by anisotropic hydrogen plasma etching. Conventional thermal chemical vapor deposition technique was employed to synthesize the SWNTs. Nano-sized islands or particles as formed from multi-layered thin-films (Mo/Fe/Al) at high temperature are served as the catalytic nanomasks (CNMs) during plasma etching, where the remaining CNMs left at tips directly become the catalysts for SWNTs growth. As revealed by characterizations using scanning electron microscopy and resonance Raman spectra, su-SWNTs form networks interconnecting ns-Si templates. This technique allows us to assemble two different nanomaterials, i.e., ns-Si and su-SWNTs, together as a building block for future possible applications. (Work supported by the grant from the National Science Council of the ROC.)

3:40pm NS-ThA6 Fabrication of Thin Silicon Nanodisk for Quantum Effect Devices Using Cl Neutral Beam Etching and Ferritin Iron Core Mask, *T. Kubota, T. Hashimoto*, Tohoku University, Japan; *M. Takeguchi*, National Institute for Materials Science, Japan; *Y. Uraoka, T. Fuyuki*, Nara Institute of Science and Technology, Japan; *I. Yamashita*, Matsushita Electric Industrial Co., Ltd. and Nara Institute of Science and Technology, Japan; *S. Samukawa*, Tohoku University, Japan

Miniaturization of semiconductor devices will require new fabrication processes to realize nanometer-scale structures without process damages. To realize a nanometer-scale structure, we used ferritin iron core (7nm in diameter) as a uniform and high-density template, and our developed neutral beam (NB) etching process for damage-free etching. We have fabricated "nanodisk", a nanometer-thick disk-shaped silicon structure. Thin oxide layer of about 1nm was grown on Si(100) wafer and then 4-nm-thick Si layer was deposited. The Si layer was etched by using Cl NB with ferritin iron core as an etching mask. There were two problems which had to be solved to fabricate thin nanodisk. (1) The native oxide layer on thin Si layer causes enlarging the diameter of nanodisk. (2) The oxide layer must be remained while Si layer has to be completely etched. To solve these problems, two step NB etching process was performed. High-energy neutral beam was used for the first step to breakthrough the native oxide and low-energy neutral beam was used for the second step to obtain high etching selectivity to underlying-SiO₂@sub 2@. To monitor the proceeding of atomic layer etching, XPS measurement was utilized. We could successfully observe the fabrication of silicon nanodisk (2nm in thick and 10nm in diameter) through cross-sectional TEM. Such nanometer-scale structure would be applied to quantum effect devices. A part of this work was supported by "Nanotechnology Support Project" of the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan. This study was supported by Leading Project of Ministry of Education, Culture, Sports, Science and Technology.

4:00pm NS-ThA7 Cool Plasma Functionalization of Nanodiamond Particles, *G.E. McGuire, M.A. Ray, O. Shenderova, W.M. Hooke, A. Martin, T. Tyler, G. Cunningham, V. Grishko*, International Technology Center

An atmospheric pressure dielectric barrier glow discharge (APDBGD) plasma system has been developed at ITC. It was demonstrated that the APDBGD plasma system is a powerful tool for the surface functionalization of nanodiamond particulates. Nanodiamond functionalization was performed in minutes using plasma discharges generated with nitrogen and fluorine containing gases. The chemical bonds formed between reactive species generated in the plasma and nanodiamond surfaces were confirmed by FTIR and XPS analysis. Following plasma fluorination, XPS analysis revealed a relatively high concentration of F on the nanodiamond surface, up to 4-6wt%. Based on FTIR spectra analysis of several types of the initial ND produced by different vendors, it was demonstrated that plasma treatment of ND results in removal of particular surface groups (such as OH- and C=O), as well as in the formation of a variety of carbon-fluorine types of bonding (such as CF, CF@sub 3@(CF@sub 2@), C=CF@sub 2@) depending on the surface chemistry of the initial ND. Stable

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colloidal solutions of anisole with 0.5% by mass of nanodiamond were formed using these fluorinated nanodiamond particles, which are intrinsically hydrophilic and otherwise easily sediment in non-polar organic solvents. The degree of functionalization with a specific surface group depends on the surface chemistry of the initial nanodiamonds as well as on the type of gas used for plasma generation. Particularly, the use of CF₄ gas results in more efficient nanodiamond fluorination than SF₆.

4:20pm **NS-ThA8 ZnCdSe Ternary Nanophosphors**, *H.-J. Lee*, University of Florida; *H. Yang*, Hongik University, South Korea; *S.Y. Seo*, **P.H. Holloway**, University of Florida

ZnCdSe quantum dots have been synthesized by a high temperature colloidal method using trioctylphosphine oxide (TOPO) solutions. Oleic acid complexes of Cd and Zn were used for the metal sources in reactions that produced air-stable nanoparticles of CdSe and ZnSe, respectively. Minimal alloying was achieved during a one-step synthesis process that led to a ZnSe shell on a CdSe core. Reaction temperatures below 250°C led to less alloying as evidenced by spectral shifts. The kinetics of alloying were slower at 250°C as compared to quantum dots synthesized at 320°C. Smaller nanocrystals were obtained at the lower growth temperature due to a smaller critical radius of nucleation, leading to a large nucleation rate. ZnCdSe nanorods were also synthesized from CdSe/ZnSe coreshell nanorods via a solution thermal alloying process at 270~280°C. CdSe nanorods were prepared using tetradecylphosphonic acid (TDPA)/TOPO surfactants, and a ZnSe shell was grown on CdSe nanorods at 180~190°C. These nanorods were characterized as a function of alloying time using X-ray diffraction, Raman, and transmission electron microscopy. The kinetics of alloying and properties of ZnCdSe nanophosphors will be discussed.

4:40pm **NS-ThA9 Multiphysics Modeling of Electrochemical Structuring Using Complex Tool Electrodes**, *J.A. Kenney*, **G.S. Hwang**, University of Texas at Austin

Electrochemical machining using ultra-short voltage pulses has been used to modify electrically-active substrates at the micron length-scale without causing thermal damage or affecting crystal structure, unlike other machining technologies. However, this technique suffers from relatively slow processing speeds. In order to increase the speed, the use of complex tool 'template' electrodes has been suggested. Here we examine the relationship between pulse duration and feature separation on the resulting substrates using a transient charging simulation of the behavior of the pulse coupled with a feature profile evolution simulation employing the level set method.

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