

Nanostructure Synthesis and Fabrication Room Plaza Exhibit - Session NS-SuP

Nanostructures Synthesis and Fabrication Poster Session

NS-SuP-1 Refractive Index and Bandgap Variation in Al₂O₃-ZnO Ultrathin Multilayers Prepared by Atomic Layer Deposition, Javier López Medina, CONACYT - Centro de Nanociencias y Nanotecnología - UNAM, Mexico; E Solorio, H Borbón, F Castillon, R Machorro, Centro de Nanociencias y Nanotecnología - Universidad Nacional Autónoma de Mexico, Mexico; N Nedev, Universidad Autónoma de Baja California, Mexico; M Farias, H Tiznado, Centro de Nanociencias y Nanotecnología - Universidad Nacional Autónoma de Mexico, Mexico

This research focuses on the study of the refractive index and bandgap behavior in ultrathin multilayer films of Al₂O₃-ZnO bilayers grown via atomic layer deposition (ALD) technique on Si(100) substrates. The multilayer configuration stack consists in alternate layers of constant thickness Al₂O₃ (2 nm) and varying thickness ZnO films in order to obtain a total thickness of ~100 nm. A set of 10 samples based on bilayers with various 2:X thickness ratios were prepared, where X refers to the ZnO layer thickness. X is proportional to the number of cycles (N) of the ZnO precursor, varying from 1 to 100. The sample morphology was studied via Atomic Force Microscopy and the results show that the surface roughness of the multilayers varies from 0.2 to 1.2 nm, as the ZnO layer thickness increases. In all cases, the roughness values remain below 2% of the total thickness of the multilayer. The refractive index $n(\lambda)$ and optical bandgap, E_g , of each multilayer sample were studied via spectroscopic ellipsometry (SE). A General Oscillator optical model was utilized to fit the experimental data in order to obtain the total thickness, refractive index and absorption coefficient. Cross-sectional mode scanning electron microscope images verified the multilayer total thickness and corroborated the accuracy of the optical model. The refractive index varies significantly from values close to the Al₂O₃ refractive index when the bilayer thickness is small, up to values corresponding closely to ZnO for thicker bilayers. The refractive index, as a function of bilayer thickness, varies between 1.63 and 2.3, for $\lambda \approx 370$ nm (UV region), showing high sensitivity. In addition, the optical bandgap energy, E_g , determined using the Tauc model, decreases when the bilayer thickness increases, with a maximum variation of $\Delta E_g \sim 1.6$ eV. These results reveal that the refractive index and optical bandgap of Al₂O₃-ZnO material can be modulated systematically as a function of the bilayer thickness. Such behavior is of great importance for optoelectronics applications, in particular for the development of devices with response in the UV spectral range.

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NS-SuP-2 Controlled and Selective Etches for Gate All-Around Device Fabrication, Subhadeep Kal, J Smith, N Mahanty, Y Su, C Pereira, A Masden, P Biolsi, T Hurd, Tokyo Electron

In scaling beyond the 5nm technology node there is an impetus for gate all-around (GAA) device architecture. A basic requirement for GAA is the formation of silicon-germanium (SiGe) and silicon (Si) nanowires (NW). Fabricating either Si or SiGe NW (also known as NW release) requires an extremely selective, isotropic and precise SiGe (shown in Fig.1, step 1) and Si etch, respectively. After the Si NW release (or SiGe etch), the SiN gate spacer is formed around the Si NW, making a continuous and sealed gate spacer (Fig.1, step 2). In the case of a partial NW release (Fig.1), the SiGe etch must be controlled with an accuracy of >5Å since this recess will effectively define the gate spacer thickness in the area above and below the Si NW after spacer etch (SE)/liner etch (step 3, Fig.1). Here we highlight why a precise etch control is essential: (1) if the SiGe recess is too small, the reformed gate spacer thickness will be under specification and will result in capacitance problems between gate and adjacent source/drain bar metals. (2) If the SiGe recess is too much, the reformed gate spacer will penetrate into the replacement gate and will decrease the amount of gate

metal wrapping around the nanowire and will impact gate functionality. Once only the silicon wire is protruding through the seamless gate spacer (Fig.1, step 3), SiGe:B can be grown from the ends of the silicon wires protruding through the gate spacer (Fig.1, step 4). In addition to the above requirements, etch selectivity towards to the gate and low K material around the gate (not shown in Fig 1) is also preferable. Therefore, a process flow enabled with extremely high selective etches, where the selectivity is a function of film properties and/or etch chemistry is a quintessential advantage. In this article, we demonstrate the significance of such selective etches for Si NW formation step (Fig.1, step 1) and corresponding SiGe NW fabrication.

Fig.1 (step 1), shows the process performance of a selective gas phase SiGe etch for Si NW (SiGe etch) formation. Research is ongoing to explore further how these techniques can be optimized together to obtain a straight SiGe and Si etch front. Similar etch techniques has also proved to be essential for full NW release, where the requirements are selective, isotropic and complete etch of Si or SiGe stacks. In addition we will also show an alternate selective etch technique to fabricate corresponding SiGe NW (Si etch) formation.

NS-SuP-3 Wafer-Scale Synthesis of High-Quality and Few-Layer WS₂ Films on Si/SiO₂ Substrates, Yung-Ching Chu, National Chiao Tung University, Republic of China; C Jong, NARLabs, Republic of China; Y Ho, National Chiao Tung University, Republic of China; P Lu, UCLA; C Zhang, National Chiao Tung University, Republic of China; H Hsu, ITRI, Republic of China; Y Tu, National Chiao Tung University, Republic of China; J Woo, UCLA; E Chang, National Chiao Tung University, Republic of China

Due to their attractive properties for next-generation electronic and optoelectronic devices, two-dimensional (2D) layered transition metal dichalcogenides (TMDs) materials such as MoS₂, WSe₂ and WS₂ have attracted a great deal of attention recently. Among these 2D TMDs materials, few-layer tungsten disulfide (WS₂) is particularly attractive for electronic applications due to its potential high hole mobility. The theoretically predicted room temperature mobility of WS₂ is 1,103 cm²-V⁻¹-s⁻¹, much higher than that for MoS₂ (304 cm²-V⁻¹-s⁻¹) and WSe₂ (705 cm²-V⁻¹-s⁻¹) [1]. In addition, few-layer WS₂ with a direct band-gap about 2.0 eV is suitable for electronic and optoelectronic devices. While remarkable methods for other TMDs large area preparation have been reported, large area synthesis of WS₂ remains a challenge.

In this study, a novel scalable method of realizing wafer-scale well controlled WS₂ films is proposed and demonstrated. The synthesis of WS₂ films start by depositing tungsten trioxide (WO₃) films on 4-inch heavily doped silicon substrate with 80-nm SiO₂ cap layer by e-gun evaporation. These films were then converted into few-layer WS₂ via sulfurization with hydrogen sulfide (H₂S) at elevated temperatures. Figure 1(a) shows that a uniform WS₂ film is achieved, fully covering the 4-inch substrate wafer. The WS₂ film thickness can be controlled by adjusting the e-gun evaporated tungsten oxide film thickness, which was confirmed by cross-sectional transmission electron microscopy (TEM). Figure 1(b) shows the cross-section TEM image for 1 nm WO₃ film converted to a continue WS₂ film. The inset is a high resolution TEM showing 2~3 layers of WS₂. Both Raman and photoluminescence (PL) spectra of the samples were acquired using a Raman microscope system with a laser excitation wavelength of 532 nm. Raman spectrum (Figure 1(c)) indicates that the as-synthesized WS₂ thin films have two characteristic peaks of the in-plane vibrational mode, E_{2g}, at about 353 cm⁻¹ and the out-of-plane vibrational mode, A_{1g}, at about 418 cm⁻¹. PL spectrum of as-synthesized WS₂ (Figure 1(d)) shows the major PL peak (A) at approximately 1.98 eV associated with the direct band-gap transition at K point. The weak PL peak (I) associated with the indirect band-gap transition.

In conclusion, a promising method for the synthesis of wafer-scale high quality WS₂ on insulating substrates that could enable batch fabrication of 2D electronic and optoelectronic devices were proposed. TEM, Raman and PL were performed on these samples to demonstrate their excellent material qualities.

Reference

[1] W. Zhang et al., *Nano Research*, vol. 7, 1731-1737, 2014

NS-SuP-4 Towards Producing Bulk Monolithic Core/Shell Nanocomposites, Boris Feigelson, J Wollmershauser, K Manandhar, U.S. Naval Research Laboratory

With designed bulk nanostructured solids, one could potentially combine properties that are mutually exclusive in a single bulk material, and, as a result, dramatically improve the desired performance. However, a major

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research challenge and roadblock is how to produce 3D nanostructured materials consistently with the required phases arranged in designated spatial order that are at the same time fully dense without porosity and detrimental phases. Known state-of-the-art techniques for producing bulk nanostructures cannot simultaneously meet all these requirements. As a result, the inherent properties of such bulk monolithic nanostructured materials are greatly unknown and unexplored.

We developed an Enhanced High Pressure Sintering (EHPS) process to consolidate oxide, metal, and semiconductor nanoparticles into 3-dimensional monolithic nanostructured materials. EHPS incorporates stringent environmental control and utilizes high pressures to break agglomerates while simultaneously exploiting the increased pristine surface potential of nanoparticles for surface-energy-driven densification without microstructural changes. Using this approach, monolithic nanocrystalline transparent ceramics with grain size below 30 nm are demonstrated. Such ceramics exhibit a 30% increase in hardness over a corresponding order of magnitude reduction in grain size and suggest that Hall-Petch type (strengthening via grain size reduction) relations exist in ceramics at least down to 25nm [1].

Core/shell nanoparticles offer fundamentally new means for nanostructured solids design and tailoring basic properties of these artificial materials. To provide flexibility in core/shell nanoparticles design, a particle atomic layer deposition (pALD) reactor was incorporated in the EHPS facility. The new setup allows to controlling environment during all stages of the nanoparticles processing, atomic layer deposition and sintering. Spinel/alumina core/shell nanoparticles were the first material system tested for producing the first core/shell ceramic nanocomposite. Alumina shell was grown on spinel nanoparticles, and then core/shell nanoparticles sintered under pressure without exposing to atmospheric air. The developed nanocomposite ceramics demonstrated better hardness and fracture toughness than pure nanocrystalline spinel.

[1] Wollmershauser, J. A.; Feigelson, B. N.; Gorzkowski, E. P.; Ellis, C. T.; Goswami, R.; Qadri, S. B.; Tischler, J. G.; Kub, F. J.; Everett, R. K., *Acta Materialia*, 69, 9-16 (2014).

NS-SuP-5 Nanolaminate Copper Barriers of Ru/TaN_x Thin Films by Inductively Coupled Plasma Enhanced Atomic Layer Deposition, Bo-Heng Liu, W Cho, C Kei, National Applied Research Laboratories, Republic of China

Ru/TaN nanolaminate barriers and Cu interconnect were sequential directly grown on dual damascene nano-porous dielectrics by inductively coupled plasma enhanced atomic layer deposition (PEALD). Ru/TaN thin films were successfully deposited by home-built PEALD using [Ru(EtCp)₂] and Ta(NC₂H₆)₅ as precursor with high energy reductive Ar/H₂ and Ar/O₂ plasma. The substrate temperature of Ru/TaN thin films were 300 and 250 °C with 500 cycles to PEALD process. The N₂ purge time and plasma power were held on 5 s and 400 W. Ru/TaN thin films were analyzed by X-ray photoelectron spectroscopy (XPS: Perkin Elmer PHI 670) and HRTEM (JEOL JEM-2100F). The resistivity was measured by using Hall effect measurement system (Accent/HL 5500PC). Fig. 1 shows the XPS of Ru 3d spectra deposited by PEALD. The energy states of 3d_{5/2} (at 280.2 eV) and 3d_{3/2} (at 284.3 eV) indicated metallic Ru. However, the lower intensity peaks at 281.2 eV and 285.9 eV are from RuO₂. Growth rate of Ru the films was increased significant from 0.01 to 0.1 nm/cycle by using dc biased PEALD. The resistivity of Ru thin films prepared by PEALD was 40 μΩ cm. The growth rate of TaN thin films was 0.05 nm/cycle as the PDMAT pulse time is more than 2 s. Cross-section HRTEM image in Fig. 2 shows that the Ru/TaN nanolaminate barriers were formed. In summary, Ru/TaN nanolaminate barriers were prepared by using Ru(EtCp)₂ and Ta(NC₂H₆)₅ precursor with the aid of the high energy reductive Ar/O₂ and Ar/H₂ plasma in ITRC PEALD system.

NS-SuP-6 On the Possibility of the Development of Vicinal Superlattices in Quantum Wires on Semiconductor Low - Index Surfaces, Victor Petrov, Russian Academy of Science, Russian Federation

As is well known, vicinal superlattices (VSLs) are realized in 2D electron systems on semiconductor high-index Miller surfaces. The existence of SL effects in VSLs is due to the appearance in these systems of a new crystallographic translation period in the plane of quantum wells $A \gg a_0$ (a_0 is the lattice constant). A new translation period A produces minigaps (MGs) in the energetic spectrum of the particles in these systems and, as a result, different SL effects. At the present time, all these VSLs are developed only in 2D systems. At the same time, it is known that

superlattice effects should be maximal in quantum wires (QWR) when the SL period A appears along the axis of the QWR.

In this work we suggest a new method of development of VSL in QWR on semiconductor low-index surfaces. For this purpose we suggest to orient the axis of the QWR at the necessary angles to the basic translation vectors on a low-index surface. In this case in the QWR the new basic translation period along the axis of the QWR $A \gg a_0$. Thus, the period A along the surface in the one-dimensional VSL is selected by the orientation of the QWR on the low-index surface. If the QWR is realized in the MOS system with the use of a narrow gate then the orientation of the wire will be determined simply by the appropriate orientation of the gate. The analytic expressions of the new periods A were obtained as a function of the angles which determine orientation of the QWR for the different low-index surfaces GaAs and Si. The positions of MGs in the one-dimensional k-space were determined. It is should be noted that in the region of the particle wave function localization in the QWR there are many crystallographic planes which form a SL energetic spectrum of the particle. Illustrative estimates of the magnitude of the MGs for the QWR of the rectangular cross-section made in the weak coupling approximation demonstrate their dependence on the geometric parameters of the cross-section, on the period A as well as on the crystal potential.

NS-SuP-7 Rational Design of Hyperbranched Nanowire Systems for Tunable Superomniphobic Surfaces Enabled by Atomic Layer Deposition, Ashley Bielinski, M Boban, University of Michigan, USA; *Y He*, University of Pittsburgh; *E Kazyak, D Lee*, University of Michigan, USA; *C Wang*, Pacific Northwestern National Laboratory; *A Tuteja, N Dasgupta*, University of Michigan, USA

The hierarchical assembly of semiconductor nanostructures to form heterogeneous material systems has the potential to advance a range of technologies including electronics, optics, sensors, mechanical resonators, and energy conversion. Semiconductor nanowire (NW) arrays have already been successfully applied in these fields, but challenges such as lack of deterministic control of feature size, shape, and position limits the development of more complex geometries. One example, hyperbranched NWs, have been synthesized for a variety of applications, but the ability to tune the morphology along a spectrum at each level of hierarchy requires further development. Here we demonstrate surface and interface modification via atomic layer deposition (ALD) to control the fabrication and bottom-up solution growth of ordered hyperbranched NW systems.

Existing techniques for the assembly of complex nanostructures generally rely on either nanoscale patterning, or solution based processes. The patterning techniques while slow and costly on planar substrates, often become impossible on high aspect ratio surface geometries. The solution-based approaches offer scalability and lower cost, but the results are often disordered and difficult to tune. ALD is an enabling technique for the control of low-cost, scalable hydrothermal NW synthesis. This process relies on atomic-scale precision of conformal films deposited on nanowire arrays, for which ALD is an ideal process.

We start by demonstrating the control of ZnO NW array properties such as density and orientation by tuning the crystallographic orientation, roughness, and surface stress of ALD films used to seed the NW nucleation.[1] We then show how ALD can be used to overcome the challenges that arise when transitioning from simple NW arrays to complex branched structures. Sub-monolayer deposition of inert blocking layers were used to reduce NW density independent of orientation in order to make room for subsequent branched NW growth. ALD interlayers were then used to prevent epitaxial ALD growth of subsequent seedlayers on the single crystalline NWs. These techniques were all combined to fabricate hyperbranched NW arrays. The array properties were controlled with ALD at each level of hierarchy to produce superomniphobic (repellent to high and low surface tension liquids) with tunable contact angles for different liquids.[2]

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(2) Bielinski, A. R.; Boban, M.; He, Y.; Kazyak, E.; Lee, D. H.; Wang, C.; Tuteja, A.; Dasgupta, N. P. *ACS Nano* **2017**, 11 (1), 478–489.

NS-SuP-8 Designing Low Density Foams by ALD Templating, Monika Biener, Lawrence Livermore National Laboratory

Low density foams with precisely controlled architectures, compositions and densities have many promising applications in the fields of energy storage, catalysis, biomaterials, and high energy density physics. Here, atomic layer deposition offers a unique opportunity for rapid on-demand

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development of functional nanoporous bulk materials by applying the principle of templating to material systems for which robust synthesis strategies have already been developed. I will summarize recent progress made in synthesis and characterization of ALD-derived ultralow density target materials that are important for inertial confinement fusion (ICF) experiments. As porous substrates we use Polystyrene beads deposited by electrophoretic deposition, additively manufactured 3D micro lattices and nanoporous gold. Nanometer-thick metal oxide ALD coatings (ZnO, Ta₂O₅, Al₂O₃) provide the desired functionality and often dramatically improve the mechanical properties of the porous substrate. Even only nanometer thick coatings can be mechanically so robust that the substrate can be removed without collapse of the structure. The resulting materials can have air-like densities which, for example, enable the realization of brighter X-ray sources and promise to improve the performance of ICF targets.

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NS-SuP-9 Fully CMOS-Compatible Synthesis and Photodetector-Integration of Ultrathin, Parallel-Aligned ZnO Nanowire Arrays by Infiltration Synthesis, *Chang-Yong Nam, A Stein*, Brookhaven National Laboratory

Semiconductor nanowires with reduced diameters enable high-performance chemical sensors and photodetectors owing to their large surface-to-volume ratios and enhanced surface band bending. Synthesis of nanowires and their device integration by CMOS (complementary metal-oxide-semiconductor)-process-compatible methodologies can be extremely powerful for various technological applications of nanowires. Here we report fully CMOS-compatible synthesis and ultraviolet (UV)-photodetector-integration of ultrathin (~30 nm diameter), parallel-aligned, polycrystalline ZnO nanowire arrays by infiltration synthesis, a type of inorganic-organic hybridization technique derived from atomic layer deposition. The ultrathin ZnO nanowire array was generated by infiltrating diethylzinc (DEZ) and water vapors into lithographically patterned polymer nanowire template made of a negative-tone resist SU-8. The integrated ZnO nanowire array photodetectors featured ultralow dark currents <20 fA unchanging with the number of nanowires, photocurrent on-off ratios over 10⁶ leading to >120 dB linear dynamic range, and super-linear photocurrents causing unusually increasing photodetector performance parameters for a higher incident light power. Considering the temperature-dependent field-effect transistor characteristics of the ZnO nanowire arrays, we explained the observed super-linear photoconductivity by the new type of photo-thermionic emission mechanism involving the reaction of chemisorbed oxygen and photo-generated holes at grain boundaries. The developed rationale provides guidelines for utilizing polycrystalline semiconductor nanostructures for photodetectors and other sensors. The demonstrated synthesis and fabrication methods based on the infiltration synthesis have potentials for CMOS-integration of nanowire sensor devices and circuitries.

NS-SuP-10 Monodispersed, Highly Interactive Facet Oriented Pd Nanograins Grown by ALD onto Electrospun Polymeric Nanofibers, *Kugalur Ranjith, A Celebioglu*, Bilkent University, Turkey; *H Eren*, Delft University of Technology, Netherlands; *N Biyikli*, Utah State University; *T Uyar*, Bilkent University, Turkey

We present a membrane form of templates for the growth of Pd nanograins through the atomic layer deposition (ALD) on the electrospun polymeric nanofibers such as Nylon 6,6, PAN and Polysulfone. Under the selective atmosphere, the stable flexible polymeric nanofibers were utilized as a template and the Pd nano grains were made to interact with the polymer surface and decorated on it. Utilizing the template avoided the aggregation of the nanograins during growth and initiated mono dispersive nature of the Pd nanostructures immobilized with the size of 2 nm. Template form of Pd decorated nanofiber network exhibited an effectualness towards the reduction of 4-nitrophenol into 4-aminophenol through hydrogenation process. Even under low loading capacity of Pd nanoparticle, effective catalytic performance was exhibited which was ascribable to the exposure of single crystalline highly interactive (111) planes with high surface area on the nanofiber surface. With the aid of Pd nanograins decorated polymeric fibers, the catalytic reduction can be finished within 35 mins, even when the content of Pd ions was as low as 2 wt%. Pd nanograins exhibited thermal stability and structural integrity even after 3 consecutive reactions. Improvisation of the catalytic performance on Pd loading in different polymeric fibers was evidently a resultant of the higher surface area of the nanofibers with higher surface interaction. The

method opens up possibilities for synthesizing decorative metal nanostructures on functional polymeric fibers based membranes for multifunctional applications.

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