

# SYMPOSIUM KK

## Kinetics-Driven Nanopatterning on Surfaces

November 29 - December 2, 2004

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\* Invited paper

**8:30 AM \*KK1.1/JJ1.1/U1.1**

**Kinetic Critical Thickness for Quantum Dot Formation.** Yuhai Tu and Jerry Tersoff, IBM Watson Center, Yorktown Heights, New York.

In heteroepitaxial growth of strained layers, the growing layer often remains planar up to some "critical thickness", at which point three-dimensional (3D) islands form. This Stranski-Krastanov transition is of great practical importance. Many device structures require smooth planar layers, while conversely, 3D islands hold promise as self-assembled quantum dots. The transition from planar growth to islands poses a longstanding puzzle in the understanding of heteroepitaxy. As the misfit strain is reduced (by varying the deposited alloy composition), the "critical thickness" is observed to increase dramatically. Transition thicknesses as large as 10-30 atomic layers are seen for InGaAs on GaAs (001), and similar behavior is seen for GeSi on Si (001). We have simulated such strained-layer growth, using a continuum model that consistently accounts for the alloy compositional degrees of freedom. Under generic assumptions, we find behavior closely resembling that seen experimentally. The surface remains flat up to some thickness at which islands suddenly form. This apparent critical thickness depends sensitively on composition, but only weakly on deposition rate. Our results confirm the insightful proposal of Cullis et al., that the key factor controlling the transition is the continuous increase of surface composition due to intermixing during growth. However, the specific kinetic mechanism is unanticipated and surprisingly simple.

**9:00 AM \*KK1.2/JJ1.2/U1.2**

**Microscopic View of InAs Quantum Dot Growth and Overgrowth.** Klaus Kern, Nanoscale Science Department, Max Planck Institute for Solid State Research, Stuttgart, Germany.

Surfaces and interfaces not only determine to a large extent the properties of small-scale materials due to their high surface-to-volume ratio, they are also an ideal platform for the design, fabrication and device integration of nanostructures. Both top-down and bottom-up methods have been developed for the handling of matter at the molecular and atomic scale. The prime example for the bottom-up fabrication of nanostructures is the Stranski-Krastanov growth of semiconductor quantum dots (QD's), with the model systems Ge/Si(100) and InAs/GaAs(100). Although the self-organized growth of these nanostructures has been intensely studied for more than a decade and a number of important achievements have been obtained concerning their electronic and optical properties, a definite and coherent picture of the growth scenario is still missing. In this talk I will present a microscopic view of the growth and overgrowth of InAs quantum dots on GaAs(100) obtained by scanning tunneling microscopy. The atomic-scale experiments demonstrate that the shape and composition of the quantum dots are determined by the delicate interplay between thermodynamic and kinetic effects. Moreover, the STM measurements reveal that only two well-defined island shapes, pyramids and domes exist. Both structures are very similar to the island shapes observed in the Ge/Si system suggesting that pyramids and domes are universal island shapes, independent of specific material parameters. Also the overgrowth scenario that emerges from our measurements, in which the QD capping can be described as a backward transition from steeper domes to shallower pyramids, is material independent as long as the surface energy of the cap material is higher than that of the island material.

**9:30 AM \*KK1.3/JJ1.3/U1.3**

**Pattern Formation on Silicon and Silicon-on-Insulator.** Max G. Lagally, Department of Materials Science and Engineering, University of Wisconsin-Madison, Madison, Wisconsin.

The strain driven self-assembly of faceted Ge nanocrystals during epitaxy on Si(001) to form quantum dots (QDs) is by now well known. [1] We have also recently provided an understanding of the thermodynamic driving force for directed assembly of QDs on bulk Si (extendable to other QD systems) based on local chemical potential and curvature of the surface. [2] Silicon-on-insulator (SOI) produces unique new phenomena. The essential thermodynamic instability of the very thin crystalline layer (called the template layer) resting on an oxide can cause this layer, under appropriate conditions, to dewet, agglomerate, and self-organize into an array of Si nanocrystals. Using LEEM, we observe this process and, with the help of first-principles total-energy calculations, we provide a quantitative understanding of this pattern formation [3] and show how addition of Ge affects the energies and hence the pattern. The Si nanocrystal pattern formation can be controlled by lithographic patterning of the SOI prior to the dewetting process. The resulting patterns of electrically isolated Si

nanocrystals can in turn be used as a template for growth of nanostructures, such as carbon nanotubes. [4] We show that this growth is controlled by the flow dynamics, and possibly by electrostatics. Growth of heteroepitaxial films on SOI brings with it unique defect generation mechanisms, associated with the Si-oxide interface, that may, in fact, result in the elimination of patterns found for growth on bulk Si. We fabricate thin membranes and free-standing structures to investigate the effect of added uniaxial stress on adatom diffusion and the nucleation and coarsening of 2D and 3D structures on this surface. Finally, nanopattern formation on Si using etching techniques results in unexpected wetting behavior. Aspects of the work were supported by DOE and by NSF. In conjunction with B. Yang, F. Liu, P. Rugheimer, D. Savage, M. Roberts, P. Zhang, V. Joshkin, M. Marcus, and M. Eriksson. 1. Y.-W. Mo, D. E. Savage, B. S. Swartzentruber, and M. G. Lagally, Phys. Rev. Letters **65**, 1020 (1990). 2. B. Yang, F. Liu, and M. G. Lagally, Phys. Rev. Letters **92**, 025502-1 (2004). 3. Bin Yang, Pengpeng Zhang, M.G. Lagally, Guang-Hong Lu, Minghuang Huang, and Feng Liu, submitted to PRL. 4. B. Yang, M.S. Marcus, H. F. Tang, P.P. Zhang, Z.W. Li, B.J. Larson, D.E. Savage, J.M. Simmons, O. M. Castellini, M.A. Eriksson, and M.G. Lagally, submitted to Appl. Phys. Letters.

**10:30 AM KK1.4/JJ1.4/U1.4**

**KMC Computations of Strained Heteroepitaxy in 3 Dimensions.** Peter Smerka<sup>1</sup>, Giovanni Russo<sup>2</sup>, Leonard Sander<sup>1</sup> and Jason Devita<sup>1</sup>, <sup>1</sup>University of Michigan, Ann Arbor, Michigan; <sup>2</sup>University of Catania, Catania, Italy.

We study the evolution of strained heteroepitaxial films in 3 dimensions using an atomistic model due to Orr, Kessler, Snyder and Sander. This model consists of a SOS type model with nearest and next nearest neighbor bonds. The elastic effects are considered using a ball and spring model with nearest and next nearest neighbor bonds. The strain field in the substrate was deduced using an exact solution which can be efficiently evaluated using Fast Fourier Transforms. Our results show that when the elastic effects are small the films grows in a layer-by-layer fashion. However, when the elastic effects become strong we observe either mound formation or trench type structures depending on the deposition rate.

**10:45 AM KK1.5/JJ1.5/U1.5**

**The Effect of Inhomogeneous Diffusion on the Formation of Quantum Dots.** Christian Ratsch, Raffaello Vardavas, Xiaobin Niu and Russel Caflisch; Mathematics, UCLA, Los Angeles, California.

The simulation of the formation and self-organization of quantum dots is a major goal in modeling efforts of epitaxial growth. An anisotropic, spatially varying diffusion constant can lead to preferred island nucleation in certain regions. Such an anisotropic, inhomogeneous diffusion field can be achieved, for example, by burying defects (or other structures) underneath the surface: the inhomogeneous strain field leads to an inhomogeneous potential energy surface, and thus to an inhomogeneous diffusion field. We will show that under the right conditions, nucleation occurs preferentially along certain geometric lines, with all islands being approximately the same size. In addition, there are large denuded zones on the surface without any islands in it. Our results were obtained with an island dynamics model that employs the level-set technique. This approach is particularly well suited for this problem, as both, a spatially varying diffusion field, as well as microscopic events on vastly different time-scales can easily be implemented in our model, without extra computational cost.

**11:00 AM KK1.6/JJ1.6/U1.6**

**On the mechanisms guiding epitaxial island assembly on topographically patterned substrates.** Robert V. Kukta, Mechanical Engineering, Stony Brook University, Stony Brook, New York.

A method of self-assembling quantum dot domains that promises substantial flexibility and control is strained layer growth on topographically patterned substrates. Prior to growth, features are introduced on the substrate by a method such as lithography and ion-etching. These features guide morphological development by providing preferred sites for islands to nucleate and grow. While it is known from experiment that the arrangement of islands on patterned substrates is affected both by energetic and kinetic phenomena, the nature of these effects has not been well characterized. There have been few analytical efforts to understand where islands form and why and how they develop at particular sites. It is the purpose of this paper to offer some new insight. A continuum-level model is used where the free energy of system is assumed to consist of surface free energy and strain energy. Energetics is first addressed to understand which sites are most favorable for islands, depending on factors as substrate shape, lattice mismatch between the film and substrate materials, surface energy, and the amount of material deposited. The result is presented in a simple phase diagram. Then the kinetic process of deposition combined with morphological evolution by surface diffusion is simulated. It is found that configurations may arise

that appear to be neither stable nor metastable according to a simple energetic analysis; free energy is reduced as islands translate in certain directions. Yet even with a lengthy post-deposition anneal these configurations do not evolve kinetically. Such configurations are found to represent a novel metastable state wherein islands are locked in energetically unfavored sites because the kinetically pathway admitted by surface diffusion requires evolution through states of higher free energy. It is demonstrated that a critical island size exists for this metastability to occur. Small islands tend to be mobile and able to move to minimum-energy sites, whereas large islands tend to be immobile. This phenomenon results in islands affixed to the side walls of substrate surface features. Focus is limited to growth on small amplitude sinusoidal substrate features as studied in detail recently in a series of experiments by Zhong et al. [J. Appl. Phys. 93, 6258 (2003); Appl. Phys. Lett. 82, 445 (2003)]. Their experiments provide evidence of the novel metastability and also illustrate that both kinetics and energetics have a role in guiding the positioning of islands. Insight to the nature of these effects is obtained from kinetic simulations based on their experiments.

#### 11:15 AM KK1.7/JJ1.7/U1.7

**In-Situ Studies of Cu<sub>2</sub>O Nano-Island Formation on Cu (001).** Jeffrey A. Eastman, Paul H. Fuoss, Guangwen Zhou, Lynn E. Rehn, Peter M. Baldo, Loren J. Thompson and Dillon D. Fong; Materials Science Division, Argonne National Laboratory, Argonne, Illinois.

The early-stage oxidation behavior of the Cu (001) surface is being investigated at the Advanced Photon Source via in-situ x-ray diffraction techniques. Single crystal (001) Cu thin films grown on (001) SrTiO<sub>3</sub> substrates are oxidized to form crystallographically-aligned Cu<sub>2</sub>O nano-islands on the Cu surface. By measuring oxide island growth kinetics as a function of oxygen partial pressure, the thermodynamic limit between oxide growth and reduction has been determined for several temperatures and film thicknesses. We find the Cu/Cu<sub>2</sub>O phase boundary at much larger oxygen partial pressure than predicted by bulk phase equilibria, and also find that the temperature dependence of this phase boundary is smaller than expected. Large reversible changes in the oxide lattice parameter of up to 0.5% in response to controlled variations in oxygen partial pressure in the chamber are also observed, possibly indicating an unexpectedly large stoichiometric range in Cu<sub>2</sub>O nano-islands compared with bulk Cu<sub>2</sub>O. Significant differences are seen in the oxidation behavior of thin film and bulk single crystal samples, leading to the conclusion that substrate constraints are influencing behavior. The prospects for engineering these constraints to influence nanopattern formation will be discussed.

#### 11:30 AM KK1.8/JJ1.8/U1.8

**Orientation dependence behavior of self-assembled (In,Ga)As quantum structures on GaAs substrates.** Shahram Seydmohamadi, Zhiming M. Wang, Hong Wen and Gregory J. Salamo; Physics, University of Arkansas, Fayetteville, Arkansas.

The orientation of the substrate can play an important role in the formation of (In,Ga)As quantum structures grown on GaAs. In particular, the unique surface morphology of a high index substrate allows the substrate to act as a template for the growth of self assembled quantum wires (QWRs) and quantum dots (QDs). In this work, the formation of self assembled (In,Ga)As quantum structures are discussed as a function of crystallographic orientation of the GaAs substrates. We report on the effect of the orientation of a GaAs substrate on the evolution of strained (In,Ga)As quantum structures, along one side of the stereographic triangle from (100) towards (111)A, including (100) and (N11)A surfaces, where N is equal to 7, 5, 4 and 3. The samples were grown by UHV molecular beam epitaxy (MBE) and characterized by atomic force microscopy (AFM) and scanning tunneling microscopy (STM). For the same coverage of (In,Ga)As, a transition from zero dimensional (In,Ga)As QDs to one dimensional QWRs is observed as a function of the orientation of the substrate moving from (100) toward (311)A along the stereographic map. While (In,Ga)As QDs form on GaAs (100) and (711)A surfaces, we observe QWRs formation on GaAs (311)A and (411)A surfaces. In between, GaAs (511)A is covered by elongated (In,Ga)As QDs. When taken together with the observation of QDs on the GaAs (100) and (711)A surfaces and QWRs on the GaAs (311)A and (411)A surfaces, the texture observed on the GaAs (511)A surface is interpreted as observing a transition taking place from quantum dots to quantum wires on the (511)A surface. The overview picture then, is that a continuous transition from QD to QWR takes place along the mentioned stereographic direction. We will present an explanation for the transition from QD to QWR behavior along the stereographic triangle based on the changing surface morphology of the substrates.

#### 11:45 AM KK1.9/JJ1.9/U1.9

**Energetics of Trench Formation Around Ge/Si Quantum Dots.** Dhananjay Tulsiram Tambe and Vivek B. Shenoy; Solid

Mechanics, Brown University, Providence, Rhode Island.

At high growth temperatures, the misfit strain at the boundary of a Ge quantum dot on Si(001) is relieved by the formation of a trench around the base of the island. The depth of the trench has been observed to saturate at a level that depends on the base-width of the island. Using finite element simulations, we show that the self-limiting nature of trench depth is due to a competition between the elastic relaxation energy gained by the formation of the trench and the surface energy cost for creating the trench. Our simulations predict a linear increase of the trench depth with the island radius, in quantitative agreement with recent experimental observations.

SESSION KK2/JJ2/U2: Joint Session: Patterning and Steps on Surfaces  
Chairs: Jim Evans and Richard Vinci  
Monday Afternoon, November 29, 2004  
Room 210 (Hynes)

#### 1:30 PM \*KK2.1/JJ2.1/U2.1

**One-dimensional interfaces in Two-dimensional Materials Structures\*.** Ellen D. Williams<sup>1,2</sup>, Daniel Dougherty<sup>2</sup>, Chenggang Tao<sup>2</sup>, Oleksander Bondarchuk<sup>1,2</sup>, Theodore L. Einstein<sup>1,2</sup>, Michael S. Fuhrer<sup>2</sup> and Philip J. Rous<sup>3</sup>; <sup>1</sup>MRSEC, University of Maryland, College Park, Maryland; <sup>2</sup>Physics, University of Maryland, College Park, Maryland; <sup>3</sup>Physics, University of Maryland Baltimore County, Baltimore, Maryland.

Steps, island edges and domain boundaries are one-dimensional interfaces that serve as the locus of material transport, and as interfacial barriers for electron transport. These interfaces fluctuate under thermal excitation, with length and time scales that can be observed directly using scanning probe imaging. Quantitative characterization of these fluctuations using the tools of statistical mechanics yields energetic and kinetic parameters that can be used to predict evolution of structure under external driving forces (e.g. temperature gradient, growth or sublimation, electromigration). In addition, as the size of the bounded structure decreases into the nanoscale, the stochastic aspects of the fluctuations themselves become a significant component of the material properties. Scanned probe measurement of fluctuations, correlation, autocorrelation, survival and persistence, will be presented for steps (on Ag, Pb and C<sub>60</sub>/Ag) and domain boundaries (Pb/Si, Ag/Si and C<sub>60</sub>/Ag). The meaning of system size in designing, evaluating and using these results will be explained. The impact of the one-dimensional structures on electron flow will also be presented. Direct measurements of step fluctuations in the presence of an electromigration current density of up to 10<sup>5</sup> A/cm<sup>2</sup> will be shown and interpreted in terms of the limits on effective charge for mass displacement at the line boundary. Measurements of the noise and resistivity in electron transport will be shown and characterized in terms of structural fluctuations in a film near the percolation threshold. \* Different aspects of this work have been supported respectively by the DOE-BES, NSF-NIRT and NSF-MRSEC

#### 2:00 PM \*KK2.2/JJ2.2/U2.2

**A Model of Patterning Molecular Dipoles on Solid Surfaces.** Zhigang Suo, Harvard University, Cambridge, Massachusetts.

Abstract Adsorbed on a solid surface, a molecule can migrate and carry an electric dipole moment. A nonuniform electric field can direct the motion of the molecule. A collection of the same molecules may aggregate into a monolayer island on the solid surface. Place such molecules on a dielectric substrate surface, beneath which an array of electrodes is buried. By varying the voltages of the electrodes individually, it is possible to program molecular patterning, direct an island to move in a desired trajectory, or merge several islands into a larger one. The dexterity may lead to new technologies, such as reconfigurable molecular patterning and programmable molecular cars. This paper develops a phase field model to simulate the molecular motion and patterning under the combined actions of dipole moments, intermolecular forces, entropy, and electrodes. Slides of this talk will be available at [www.deas.harvard.edu/suo](http://www.deas.harvard.edu/suo) Z. Suo and W. Hong, PNAS 101, 7874 (2004). Y.F. Gao and Z. Suo, J. Appl. Phys. 93, 4276 (2003). W. Lu and Z. Suo, J. Mech. Phys. Solids, 49, 1937 (2001).

#### 2:30 PM \*KK2.3/JJ2.3/U2.3

**The Quasicontinuum Monte Carlo method for simulating surface growth.** Leonard M. Sander<sup>1</sup>, Jason Devita<sup>1</sup> and Peter Smereka<sup>2</sup>; <sup>1</sup>Physics, University of Michigan, Ann Arbor, Michigan; <sup>2</sup>Mathematics, University of Michigan, Ann Arbor, Michigan.

We have developed an algorithm for treating growth on surfaces which treats the adatoms as a continuous fluid, and the islands and steps as

collections of discrete particles. The method gives an accurate account of shot-noise fluctuations by converting adatoms to solid one atom at a time. For the case of irreversible growth we can treat fractal island shapes, multilayer growth, the effect of Ehrlich-Schwoebel barriers, and nucleation. The algorithm can, in favorable cases, be faster than Kinetic Monte Carlo. For growth near equilibrium we need to treat attachment and detachment from the islands on the same footing. This is much more delicate than the irreversible case. We will discuss the issues that arise, and how we deal with them.

### 3:30 PM KK2.4/JJ2.4/U2.4

#### Continuum Theory of Interacting Steps on Crystal Surfaces in (2+1) Dimensions. Dionisio Margetis<sup>1</sup> and Robert V. Kohn<sup>2</sup>;

<sup>1</sup>Mathematics, M.I.T., Cambridge, Massachusetts; <sup>2</sup>Courant Institute of Mathematical Sciences, New York University, New York, New York.

The difference-differential equations of step motion on crystalline surfaces below the roughening transition temperature are formulated for closed interacting steps of reasonably arbitrary shape in (2+1) dimensions. Among the major kinetic processes considered are the diffusion of adatoms across terraces, and the attachment and detachment of atoms at the step edges, with inclusion of the Ehrlich-Schwoebel barrier. Basic ingredients of the formulation are (i) the approximate solution of the Laplace equation for the adatom concentration on each terrace within the quasistatic approach via separation of the space variables into "fast" and "slow" ones and application of asymptotics, and (ii) an analytical expression for the step chemical potential for a sufficiently wide class of step interactions, which include local elastic dipole-dipole interactions as a special case. The continuum limit of the discrete step-flow equations is examined in detail, with particular emphasis on the case with next-neighbor interactions. This limit yields a nonlinear partial differential equation (PDE) of fourth order for the height profile, which accounts for both step stiffness and step-step interaction energy. The PDE is compared to the one derived from the standard surface-free energy considerations and is shown to reduce to the latter under certain local conditions on the step interactions. The role of longitudinal currents, which are parallel to the steps, is discussed.

### 3:45 PM KK2.5/JJ2.5/U2.5

#### Formation of Ridges on Patterned Mesas and Their Role in Evolution of Step Arrays on Mesas. Kee-Chul Chang and Jack M. Blakely; Materials Science & Eng, Cornell University, Ithaca, New York.

Mesa structures fabricated on Si(111) surfaces have been experimentally found to develop step arrays with large spacing of the order of a micron or more after annealing at temperatures where sublimation becomes important. These are caused by transient ridges around the edges that initially develop during annealing and form a barrier to step motion before eventually breaking down. This produces a step distribution that can be approximated as an array of steps of the same sign with a few wide terraces. The analysis of the evolution of such an array of steps presents new aspects from those associated with cases where the step train consists of nearly evenly spaced steps. We have done computer simulations using one dimensional Burton, Cabrera and Frank (BCF) theory with attachment-detachment and step-step repulsion for this configuration. The results suggest that under conditions where there is significant motion of the entire train of steps due to evaporation, the widely spaced terraces will persist for a long time. If we include a significant Ehrlich-Schwoebel effect, where the incorporation rate for adatoms from the lower terrace is higher than that for the upper terrace, the steps redistribute to form step density waves that become stable after a short time. With no Ehrlich-Schwoebel effect the steps tend to become uniformly spaced. The predicted distributions will be compared with the experimentally measured ones.

### 4:00 PM KK2.6/JJ2.6/U2.6

#### Self-ordering of Nanofacets on Vicinal SiC(0001) Surfaces and Its Application to Heterogeneous Nanostructures. Satoru Tanaka, Kazunori Terada, Tomoyuki Miyamoto, Masahiro Fujii and Ikuo Suemune; RIES, Hokkaido Univ., Sapporo, Japan.

Surface nanostructures by taking advantages of self-ordering or self-organization have been attracted much attention in nanofabrication of semiconductor device structures. Periodically and spontaneously ordered semiconductor surfaces reveal significantly fine and spatially uniform patterns, which are beyond the lowest limit of electron beam lithography. Formation of step/terrace structures is typically observed on vicinal surfaces. Technologically important substrates such as Si and GaAs have been studied, both for device applications and for understanding of basic surface physics. Silicon carbide is an important compound semiconductor, which possesses polymorphism and a variety of superior characteristics such as wide band-gap and significantly electronic properties. It is thus a promising

semiconductor for the next generation of high power and high frequency electronic devices. Initially, we have investigated step/terrace configurations appeared on vicinal SiC(0001) surfaces after high temperature H<sub>2</sub> annealing and found that self-ordered nanofacets, consisting of pairs of (0001) and (11-2n), were generated [1]. The origins of such periodic nanofacets were discussed in terms of equilibrium surface phase separation and surface elasticity theory followed by Marchenko et al.[2]. Note here that the periodicity of such nanofacets on 4H and 6H-SiC surfaces is superior to other materials such as Si and GaAs. We believe this is due to its polytypic nature. In this presentation we report such ordered nanofacet formation under various etching conditions and discuss possible control of surface nanostructures and also show an application to heterogeneous epitaxial system such as GaN nucleation on SiC. [1] H. Nakagawa, S. Tanaka, I. Suemune, Phys. Rev. Lett. 91, 226107-1(2003) [2] V. I. Marchenko and A. Y. Parshin, Sov. Phys. JETP 52, 1 (1980).

### 4:15 PM KK2.7/JJ2.7/U2.7

#### The Controlled Self-Assembly of Nano-Structures by the Activated Stranski-Krastanow Transition Method. Cheng-hsin Chiu, Z. Huang and C. T. Poh; Department of Materials Science, National University of Singapore, Singapore, Singapore.

The growth of nano-crystalline islands on the surface of Stranski-Krastanow (SK) film-substrate systems by self-assembly is a promising technique for device applications. A critical issue in realizing the self-assembly technology is to control the sizes, the locations, and the shapes of the nano-structures. In this talk we propose that the controlled self-assembly of nano-islands can be achieved by a novel scheme, namely the activated Stranski-Krastanow transition (ASKT) method. The basic idea of the ASKT method is to make simple patterns on a flat surface of the SK system in a special thickness range and then anneal the system to activate the self-assembly of the patterns into islands. The method can control the island locations, and by varying the geometry of the patterns, the method has the capability to produce different island structures. For example, changing the size, the aspect ratio, and the height of a rectangular pattern, the ASKT method can yield a two-by-two island array, a square ring with a single or multiple rims, and multiple wires. In addition, the feature length of the structures can be a small fraction of the pattern size, and the length can be tailored by adjusting the mismatch strain in the film without reducing the pattern size. Most importantly, the instability of the nano-structures against coarsening and shape transition, a common phenomenon that causes serious problems in device applications, can be avoided in the ASKT method. The theory of the ASKT method is presented in this talk together with three-dimensional numerical simulation that demonstrates the potential capability of the ASKT method.

### 4:30 PM KK2.8/JJ2.8/U2.8

#### Stability and Evolution of Nano-ripples on Crystalline Surfaces. Ashwin Ramasubramanian and Vivek B. Shenoy; Division of Engineering, Brown University, Providence, Rhode Island.

The relaxation of nano-ripples on metallic and semiconductor surfaces is studied using a nonlinear continuum approach that accounts for the formation and interaction energies of surface steps as well as the Schwoebel barrier at step-edges. This method is used to model recent experimental studies of relaxation of a Cu(001) surface. The relaxation process is in the attachment-detachment kinetic regime at the temperatures of interest here. Ripples are seen to decay with the formation of step-free regions or facets which indicates that the line-tension of the steps plays an important role in the decay behavior. Although the ripples have a dominant spatial frequency or wavelength, our studies find that the decay behavior is not same as that of a sinusoid of a single wavelength. The inherent nonlinearity of the evolution equations leads to significant coupling between the modes in the vicinity of the dominant wavelength. Numerical calculations that account for these coupling effects are in very good agreement with the experimental observations.

### 4:45 PM KK2.9/JJ2.9/U2.9

#### Dislocation Driven Surface Dynamics on Solids. Sanjay V. Khare<sup>1,2</sup>, S. Kodambaka<sup>1,2</sup>, W. Swiech<sup>2,1</sup>, K. Ohmori<sup>1,2</sup>, I.

Petrov<sup>2,1</sup> and J. E. Greene<sup>1,2</sup>; <sup>1</sup>Department of Materials Science and Engineering, University of Illinois, Urbana, Illinois; <sup>2</sup>Frederick Seitz Materials Research Laboratory, University of Illinois, Urbana, Illinois.

Using low-energy electron microscopy, we investigate the near-equilibrium dynamics of surface-terminated dislocations. We observe, in real time, the thermally-driven (1500-1700 K) nucleation and shape-preserving growth of spiral steps rotating at constant temperature-dependent angular velocities ( $\omega(T)$ ) around cores of dislocations terminating on TiN(111) in the absence of applied external stress or net mass change. We measure  $\omega$  as a function of spiral geometry, N<sub>2</sub> partial pressure, annealing time, and temperature.

We find that  $\omega$  is independent of the local environment and ambient, and decreases linearly with time. From the temperature-dependent  $\omega$  data, we obtain an activation barrier of  $4.9 \pm 0.3$  eV for the growth of spirals. This phenomenon, attributed to point-defect migration from the bulk to the surface along dislocation lines, is both qualitatively and quantitatively different from step curvature-driven surface dynamics and "standard" Burton-Cabrera-Frank (BCF) spiral growth.<sup>1</sup> Our results demonstrate that dislocation-mediated surface roughening can occur even in the absence of deposition or evaporation, and provide fundamental insights into mechanisms controlling nanostructural stability. We expect that this process is general and that it occurs in other materials. <sup>1</sup>S. Kodambaka, S.V. Khare, W. Swiech, K. Ohmori, I. Petrov, and J.E. Greene, *Nature* 429, 49 (2004).

SESSION KK3: Poster Session  
 Chair: Peter Anderson  
 Monday Evening, November 29, 2004  
 8:00 PM  
 Exhibition Hall D (Hynes)

### **KK3.1**

**Curvature Dependent Surface Relaxation on Si by Low-Energy Ion Beam Erosion.** Frank Frost, Bashkim Ziberi and Bernd Rauschenbach; Leibniz-Institute for Surface Modification, Leipzig, Germany.

During the last years it has been shown that low-energy ion beams are a versatile tool for nanostructuring of various surfaces via self-organized pattern formation caused by ion beam erosion. Additionally, ion beam erosion can be used as an alternative process for surface smoothing and the preparation of ultra-smooth surfaces. Provided that in the evolution of surface topography different relaxation mechanisms dominate over roughening (e. g., caused by curvature dependent sputtering) smoothing of initially rough surfaces can occur. In this work, the surface smoothing of Si surfaces by Ar<sup>+</sup> ion beams (ion energy  $E_{ion} \leq 2000$  eV) was analyzed. Atomic force microscopy (AFM) has been used to systematically investigate the topography evolution of the surfaces with respect to different process parameters (ion energy, ion incidence angle, erosion time, sample rotation). From the AFM measurements the surface roughness was quantitatively characterized by the first order (rms roughness) and second order (power spectral density - PSD) statistical quantities. Based on the time evolution of these roughness parameters the relevant surface relaxation mechanisms responsible for surface smoothing have been discussed. Especially, it is shown that (i) low-energy Ar<sup>+</sup> ion beam erosion of Si surfaces leads to various surface topographies, (ii) smoothing dominates for normal and near-normal ion incidence, (iii) if smoothing occurs, the minimum achievable surface roughness is limited by atomic noise, (iv) for low-energy Ar<sup>+</sup> ion beam erosion of Si surfaces ballistic drift (atomic transport parallel to surface) and ballistic diffusion are the dominant relaxation mechanisms, (v) secondary sputter effects caused by backscattered projectile ions and sputtered Si atoms have a great impact on the topography evolution, and (vi) smoothing by atomic ballistic drift is the most efficient smoothing process at short lateral length scales at normal and near normal ion incidence, respectively. In conclusion it will be shown that ion beam smoothing is suitable for the polishing of technological relevant Si surfaces down to 0.1 nm root mean square (rms) roughness level showing a great promise for large-area surface processing, which is essential for many advanced optical applications (e.g., x-ray optics, components for the extreme ultraviolet lithography).

### **KK3.2**

**Formation of Aligned Microfibers via Self-Assembling Silica Nanocolloids.** Valery Shklover, Lab of Crystallography, ETH Zentrum, Zuerich, Switzerland.

Controllable assembling of ordered low-dimensional nanostructures and microstructures built of nanosized building blocks is of interest for numerous applications in optics, optoelectronics and sensorics. The aim of this work, which was intended as part of more general study of the use of self-assembling in design of new nanodevices, were (a) fabrication of macroscopic colloidal arrays with shear flow self-assembling method, (b) study of influence of symmetry of crystallization cell environment on the structure of self-assembled arrays, and (c) study of influence of heat treatment on the structure of self-assembled arrays. We tried also to establish the peculiarities of crystallography of obtained nano- and microarrays in relation to the process conditions and symmetry of crystallization cell, which is one of the ways to ensure controllability and reproducibility of the assembly products. Self-assembling spherical colloidal SiO<sub>2</sub> nanoparticles using shear-flow leads to the formation of microfibers, aligned into planar arrays (laminae). The examination of the SEM patterns clearly indicates the hexagonal packing of the SiO<sub>2</sub> spheres

and ABC stacking sequence of silica particles and ccp structure (space group Fm3m, packing density 0.74), observed in many colloidal crystals. In situ X-ray study of high-temperature annealing of microfibers indicates the formation of mixtures of silica polymorphs with the high temperature beta-cristobalite and tridimite as major components. Microfibers heated in the middle temperature zone exhibit graded morphology with degree of coalescence of amorphous nanoparticles, changing along the microfiber length. The termination of microfibers and laminae can be described in terms of crystallographic faces of corresponding close packed structures, which in their turn are preset by the kinetic conditions of crystallization. The obtained 2D structure, consisting of nearly parallel micrometer-sized arrays, stabilized by drying, could be characterized by three levels of organization: (1) Crystallinity of constituent SiO<sub>2</sub> nanoparticles. According to TEM data, primary SiO<sub>2</sub> colloids and SiO<sub>2</sub> particles, partially coalesced in the middle heating zone are amorphous. (2) Structure of self-assembled microfibers. The microfibers exhibit the ccp packing of the SiO<sub>2</sub> nanospheres, usual for self-assembled nanocrystalline arrays, with the unit cell constant of the corresponding cubic cell nm, the interplanar separation in direction normal to the substrate surface nm, and position of the minimum in transmission spectrum for normal incidence to (111) face as nm (UV range), where refraction index is calculated for packing density of 0.74 and refraction index of SiO<sub>2</sub> of 1.46. (3) Structure of self-assembled laminae. The performed study is a part of the elaboration of strategy (which could require both self-assembly and template-assisted steps) towards bottom-up approach to design of nanostructure arrays for technological applications.

### **KK3.3**

**Dichroism of Thin Films Containing Aligned Ag Nanoparticles Prepared by Multi-source Dynamic Oblique Deposition.** Wataru Maekita, Motofumi Suzuki, Kazuaki Kishimoto, Kaoru Nakajima and Kenji Kimura; Department of Engineering Physics and Mechanics, Kyoto University, Kyoto, Kyoto, Japan.

It is well known that hybrid films prepared by the oblique codeposition of metals and dielectric materials have nanostructure in which metallic nanoparticles embedded in the birefringent medium. Since the plasma resonance of free electrons in the nanoparticles depends on the dielectric constants of the surrounding medium, the hybrid films show the anisotropy in the optical absorption (dichroism). In addition to this, if we control the shape of nanoparticles, the dichroism will be significantly enhanced due to the plasma resonance in which the electrons moving along the shorter and longer axes of the elongated metal nanoparticles resonate at very different frequencies. In this work, we try to embed elongated metal nanoparticles in the birefringent medium by using multi-source dynamic oblique deposition (MS-DOD), in which both deposition and azimuthal angles of substrate are controlled during deposition. In order to prepare the birefringent medium, SiO<sub>2</sub> was deposited on a glass substrate by vacuum evaporation at room temperature at a certain deposition angle between 70°–82°. The glass substrate was rotated 180° periodically during deposition. This deposition technique is known as so-called serial bideposition (SBD). Extreme atomic shadowing and limited adatom diffusion work together to produce prolate bundle on the SiO<sub>2</sub> surface. On this SBD template surface, Ag was deposited from a separated source at the large deposition angle of around 70°–80°. Since the deposition of Ag was performed at RT, it was expected that Ag stuck on the top of the bundle of SiO<sub>2</sub> and that the aligned and elongated Ag nanoparticles were formed. Optical transmittance of the samples was measured with a single-beam spectrophotometer in the wavelength range between 300 and 1500 nm. It was found that the samples have the large dichroism in the near IR region and that the direction of absorption axes coincides with longer axes of prolate template surface of SiO<sub>2</sub> and Ag nanoparticles. Anisotropic thin films including aligned metal nanoparticles will be applied for the thin film polarizer which can be fabricated on any surfaces without pre- and post-treatment such as lithography and heat treatment. This work was supported by The 21st Century COE Program "Center of Excellence for Research and Education on Complex Functional Mechanical Systems" and by The Mazda Foundation's Research Grant.

### **KK3.4**

**Studies of Si Surface Morphology Evolution during Ar<sup>+</sup> Ion Bombardment.** Gozde Ozaydin<sup>1</sup>, Ahmet Serkan Ozcan<sup>1</sup>, Yiyi Wang<sup>1</sup>, Justin Hotchkiss<sup>1</sup>, Karl F. Ludwig<sup>1</sup>, Randall L. Headrick<sup>2</sup>, Hua Zhou<sup>2</sup> and Charles R. Eddy<sup>3</sup>; <sup>1</sup>Physics, Boston University, Boston, Massachusetts; <sup>2</sup>Physics, University of Vermont, Burlington, Vermont; <sup>3</sup>Naval Research Laboratory, Washington, District of Columbia.

A systematic study of Si surface evolution during Ar<sup>+</sup> ion bombardment is reported. Real-time grazing incidence small-angle x-ray scattering (GISAXS) measurements were performed at the National Synchrotron Light Source of Brookhaven National

Laboratory. Ex-situ atomic force microscopy was also used to provide real-space information. Si (100) samples were bombarded at ion energies ranging from 300 to 1000 eV. For normal-incidence sputtering at room temperature, the development of correlated structures with two different characteristic length scales was observed. The shorter length scale features ( $\dots$ ) coarsened with time but had a typical spatial wavelength of 30 nm at all energies examined. The longer characteristic length, however, increased with energy for a given sputter erosion time. Studies performed on Si (111), Si (110) and amorphous samples gave similar results, suggesting that crystalline Si surfaces become amorphous during ion sputter erosion at room temperature. To study the temperature dependence of the surface evolution, Si (100) samples were bombarded with 500 eV ions at temperatures ranging from 25 – 500 C. At temperatures above 300 C, the dots disappeared and the roughness increased significantly, suggesting that, at the ion fluxes used, there may be a crossover from ion-induced surface amorphization at low temperatures to a crystalline surface at high temperatures. The time evolution of surface morphology during off-axis ion bombardment of Si (100) at room temperature was also studied. In some parameter regimes, two sets of perpendicular ripples were observed with distinctly different wavelengths.

### **KK3.5**

**The constrained growth and patterned distribution of nc-Si from a-SiNx/a-Si:H/a-SiNx: mechanism and experiments.** Kunji Chen, Kai Chen, Liangcai Wu, Wei Li, Yongjun Zhang and Xinfan Huang; Nanjing University, Nanjing, China.

Low-dimensional semiconductor systems, such as nanocrystalline silicon (nc-Si), are currently being studied for the purpose of new applications in both nanoelectronic and photoelectronic devices. The size and position control of uniform nc-Si grains is the key technology for the device application. We propose a model on constrained growth in a-SiNx/a-Si:H/a-SiNx sandwich structures to make uniform nc-Si grains and a method for getting patterned nc-Si distribution. The model of the constrained growth of nc-Si has been studied in accordance with the classical thermodynamic theory. Firstly, we discuss quantitatively the effect of the interface and shape of the nc-Si on the crystal growth in relation to the Gibbs free energy. During the growth processes, when the grain shape transforms from sphere-like (diameter < a-Si thickness) to disc-like shape (diameter  $\approx$  a-Si thickness), the interfacial energy between nc-Si and a-SiNx will influence the grain growth which induces the increase of the nc-Si growth free energy and finally causes the growth to halt. From our model, we determine the critical a-Si sublayer thickness of 34nm for the constrained crystallization growth and interpret the increase of the crystallization temperature for the ultra thin a-Si sublayer. Some experiments have been designed and carried out to examine the model. A series of samples of a-SiNx/a-Si:H/a-SiNx with a-Si sublayer thickness from 2nm to 40nm were annealed under different annealing conditions using both thermal (finance) annealing and laser annealing. The results show that the mean size of nc-Si grains is controlled by annealing conditions and the a-Si sublayer thickness and the critical a-Si sublayer thickness is between 20nm and 40nm which is coincidence with the theoretical value estimated from our model. We employ the method of laser interference crystallization carried out by using the phase-shifting grating mask (PSGM) to fabricate nc-Si with the two-dimensional (2D) patterned distribution. The results of atomic force microscopy, cross-section transmission electron microscopy and high resolution transmission electron microscopy show that Si nanocrystallites are formed and selectively located in the discal regions within the initial a-Si:H layer, which are patterned with the same 2D periodicity of 2.0  $\mu$ m as the PSGM. The diameter of each discal region is about 250 nm and the height is about the same as the thickness of a-Si:H layers. These results demonstrate that the present model and method is promising to fabricate various patterned nc-Si arrays for device applications simply by changing the geometry of the mask

### **KK3.6**

**Topography Evolution of Si(001) Substrate Fabricated by Ar+ Ion Beam Sputtering.** Hyung Seok Kim, Ju-Hyung Suh and Chan Gyung Park; Materials Science and Engineering, Pohang University of Science and Technology (POSTECH), Pohang, Kyungbuk, South Korea.

Ion beam sputtering is a widespread experimental technique, used in a large number of applications, such as thin film deposition, sputter etching, substrate cleaning and surface analysis. Recently, it has been reported that ion sputtering produced nanopatterns and quantum dots on Si surface under certain conditions. The ion sputtered nanopatterns are very important for the development of optoelectronic and quantum devices because of the low cost and efficient mass production of nanometer scale surface structures. For improved control of the ion beam sputtering, it is required that effects of process parameters on the formation characteristics of surface

nanostructures should be quantitatively understood. In the present study, for the fabrication of nanopatterns, Ar+ ion beam sputtering was performed on Si(001) varying process parameters, such as ion energy, flux and incident angle. Si(001) wafers were irradiated with an Ar+ beam from a 3cm Kauffman ion gun and atomic force microscopy was performed in tapping mode. Ion sputter-etching is the successful fabrication method for uniform nanometer scale patterns, such as ripples and 3-D islands on the Si(100) substrates. The 3-D island was formed in the condition of low ion beam flux and inclined incidence angle (60°). As ion beam flux increased, the ratio of ripples on etched Si surface increased, in contrary, the ratio of 3-D islands decreased. Topography of Si substrate was significantly dependent on ion beam flux and incident angle, however, independent on ion beam energy and etching time. It was considered that the formation of 3-D islands was induced by local perturbation of surface geometry and the ion beam density and incident angle were crucial factors for controlling the morphology of Si substrates.

### **KK3.7**

**Abstract Withdrawn**

### **KK3.8**

**Fabrication of High Aspect Ratio Nanoscale Pit Using Carbon Nanotube Probe.** Norikazu Arima and Akihito Matsumuro; Department of Micro/nano System Engineering, Nagoya University, Nagoya, Aichi, Japan.

Fabrication of a high aspect ratio nanoelectromechanical systems (NEMs) and nanodevices (below 50 nm) is an indispensable challenge as microelectromechanical systems shrink towards the nanoscale. However, at present, the technology to build NEMS is very limited and a great deal of improvements is required in key areas such as lithography. Here, we focus our attention on a fabrication technique that makes use of the scanning tunneling microscope. Additionally, we tried to establish a fabricating method for a nanoscale pit with a high aspect ratio by using carbon nanotube (CNT) as the probe. The CNT probes used in this study is a tungsten wire of 0.3 mm and multiwall carbon nanotube of 20-30 nm diameter. The CNT can be attached to the apex of tungsten probe by pulling-up process from CNT dispersion liquid. Tungsten probe was prepared by electrochemical etching in aqueous sodium hydrate solution and the tungsten probe was pulled from CNT dispersion liquid with a bias voltage. Effective length of the CNT probe was more than 300 nm. The nanostructures produced were Au thin films on mica substrates that were prepared by magnetron sputtering. Fabrication was carried out in an ambient pressure and room temperature. The fabricating conditions were bias voltage of 1-5 V, tunnel current of 1-8 nA and fabricating time of 10-90 s, and the optimum conditions for high aspect ratio fabrication were examined. The results of our experiment show that a threshold value exists for the fabrication of the pits between 1 V and 2 V. The depth and diameter of the pit increased with the increase in the bias voltage and tunnel current, respectively. Consequently, a bias voltage of 3 V and tunnel current of 4 nA were found to be the optimum conditions for a high aspect ratio nanoscale pit fabrication. In changing the fabricating time, depth of the pit increased with the increase in fabricating time, with a little change in the diameter of the pit. This demonstrates that CNT probes can be useful for fabricating structures without changing the diameter of nanoscale CNT probe.

### **KK3.9**

**Reconstruction of the Hydrogen Bonds in Protein-Drug Nanocomposite Prepared by Infrared-Pulsed Laser Deposition.** Sanshiro Nagare<sup>1</sup>, Jo Sagawa<sup>2</sup> and Mamoru Senna<sup>2</sup>;

<sup>1</sup>Technical Development, Nara Machinery Co., Ltd., Tokyo, Japan;

<sup>2</sup>Faculty of Science and Technology, Keio University, Yokohama, Japan.

Nanostructured protein-drug composites for a novel drug delivery system were deposited by infrared-pulsed laser deposition (IR-PLD). Composite targets were prepared by mechanically mixing the powders or chemically mixing the solutions. Species and pressure of the background gas during deposition were varied in order to examine the extent of coalescence of ablated clusters and hence the kinetics of film growth on the substrate. For this purpose, AFM observations and in situ monitoring of the film growth by a QCM sensor were performed. Mechanical interaction between the ablated clusters and gaseous molecules results in the momentum loss of the ablated clusters, so that the deposition rate increases with decreasing the molecular weight and pressure of the background gas. Ablation parameters such as laser fluence and pulse frequency were varied systematically to control the degree of vibrational excitation of the target. Varying the degree of vibrational excitation results in partial reconstruction of the hydrogen bonds between the target protein and drug, which are analyzed by the chemical shift of FT-IR. Target preparation also significantly affects the reconstruction of the hydrogen bonds and hence, the nanostructure of the protein-drug composite after PLD.

### **KK3.10**

**Nanostructure Analysis of Protein Thin Film Prepared by Wet and Dry Process.** Ichiro Taketani<sup>1</sup>, Sayuri Nakayama<sup>1</sup>, Sanshiro Nagare<sup>2</sup> and Mamoru Senna<sup>1</sup>; <sup>1</sup>Faculty of Science and Technology, Keio University, Yokohama, Japan; <sup>2</sup>Technical Development, Nara Machinery Co., Ltd., Tokyo, Japan.

Protein (mainly silk fibroin) thin films were deposited on polyethylene and Si(100) substrates by a wet or dry process. The former was performed by dip or spin coating and the latter, by pulsed laser deposition (PLD). Microstructures of the films, i.e. film thickness, surface morphology and crystallinity, were examined by AFM, SEM and thin film XRD. Secondary structure ( $\beta$ -sheet,  $\alpha$ -helix or random coil) of the films was examined by FT-IR. In order to quantify nanopattern formation on surface of films, we not only evaluated the average surface roughness, but also the average wavenumber (number of the peaks per unit length) from AFM images of the cross sections of films. The film made by the wet process is lower in the population of particles above 20 nm in height than that by the dry process. Emphasis is laid on comparison of nanopattern formation of the films made by both processes. We also varied the substrate and examined the interaction between the film and substrate of the interface. By the chemical shift of FT-IR after post treatment with methanol, Si(100) was found to have higher affinity with the protein film compared to polyethylene, due to the interaction between the protein and the surface silanol groups.

### **KK3.11**

**Protein-Drug Interaction in the Nanocomposites Prepared by UV and IR Pulsed Laser Deposition.** Jo Sagawa<sup>1</sup>, Sanshiro Nagare<sup>2</sup> and Mamoru Senna<sup>1</sup>; <sup>1</sup>Faculty of Science and Technology, Keio University, Yokohama, Japan; <sup>2</sup>Technical Development, Nara Machinery Co., Ltd., Tokyo, Japan.

Protein (Bovine Serum Albumin)-drug nanocomposites were deposited on Si(100) by pulsed laser deposition (PLD) at two wavelengths, infrared (1064nm) and ultraviolet (266nm). We used uniformly dispersed mixture of protein and drug as a target. Primary structure of the protein was analyzed by matrix laser desorption ionization time of flight mass spectrometer (MALDI-TOF-MS). Effects of electronic and vibrational excitation by UV and IR laser respectively, on the nanostructure of the composite were examined. We analyzed the secondary structure of the protein and the interaction with drug using FT-IR. The interaction between protein and drug, especially to view of the reconstruction of hydrogen bond, was examined. We observed surface morphology of the thin films using AFM and TEM, and studied the effects of ablation processes on the nanostructure of the surface. Protein targets without a drug were used for comparison to study the effects of conjugation with drug on the nanostructure and preservation of primary and secondary structures of the protein.

### **KK3.12**

**Kinetics of Linear Defect Formation in Gallia-Doped Rutile.** Nathan H. Empie and Doreen Edwards; NYS College of Ceramics, Alfred University, Alfred, New York.

The diffusion of Ga<sub>2</sub>O<sub>3</sub> into the surface of single crystal [001] rutile leads to the insertion of beta-gallia subunits along {210} planes of the parent rutile structure. These linear defects introduce hexagonally shaped tunnels, approximately 2.5 angstroms in diameter, normal to the [001] surface. Because these tunnels may serve as highly reactive sites for the attachment of macromolecules, we are exploring the application of these linear defects for creating nanostructures. The current work investigates the kinetics of defect formation and the factors that affect defect periodicity and orientation. Gallium oxide was applied to the surfaces of [001]-oriented TiO<sub>2</sub> single-crystal substrates via a sol-gel spin-coating process using different gallium-containing precursors. Thermal treatments were systematically varied to obtain different defect surface structures. Defect orientation and the distance between these rows of defects was characterized via tapping mode atomic force microscopy.

### **KK3.13**

**Photo Etching of Polyamide Thin Film by TiO<sub>2</sub> Micro Wire Prepared Using Phase Separation-Selective Leaching Method.** Yoshiaki Sone, Keishi Nishio and Atsuo Yasumori; Department of Materials Science and Technology, Tokyo University of Science, Noda-shi, Chiba, Japan.

The techniques for the solid surface modification have been studied extensively in order to apply to the fabrication of micro or nano devices and biochips. The present method for patterning is mainly photolithography and it employs photo-mask and photo-resist film accompanying with UV light illumination in a particular condition. Currently, electron beam and X-ray lithography are also examined for high resolution patterning, however, these equipments require further particular environmental conditions during process and may damage

the thin film and/or substrates due to high energy beam. On the other hand, TiO<sub>2</sub> is well known to exhibit various properties accompanied with photocatalytic reaction such as self-purification, self-antibacterial, and self-cleaning. In those reactions, organic substances are decomposed by strong oxidation owing to some oxygen radicals which generate from TiO<sub>2</sub> under UV light irradiation. Thus, photo etching of organic resist films by use of a thin film pattern of TiO<sub>2</sub> have been examined already. We have reported that the photo etching of hard and chemically inactive surfaces of organic thin films such as polyamide and DLC in the atmosphere by use of porous TiO<sub>2</sub> materials which were prepared by phase separation-selective leaching method. In this method, the high temperature melt in TiO<sub>2</sub>-SiO<sub>2</sub> binary system is held in its stable immiscibility regions in order to make the melt separate into two liquid phases. Subsequently, a TiO<sub>2</sub> porous material is formed by quenching of the phase separated melt and selective leaching of SiO<sub>2</sub> glassy matrix by alkaline aqueous solution, because the melt-quenched material consists of TiO<sub>2</sub> rich phase comprising nanometer-scale fine TiO<sub>2</sub> particles and SiO<sub>2</sub> rich phase as glassy matrix. In this method, it is easy to control the shape of the material such as a micro wire by deformation of the melt during quenching. In this study, TiO<sub>2</sub> porous micro wire was prepared by phase separation-selective leaching process, and the micro photo etching of the surface of a polyamide thin film was examined by use of the obtained wire under UV irradiation in order to apply to the patterning. This technique has the advantage; that is, direct pattern drawing in the atmospheric condition and TiO<sub>2</sub> micro wire can be used repeatedly and no photo-mask and photo-resist are utilized. Therefore, this technique has a potential to expand into novel patterning procedures.

### **KK3.14**

**Abstract Withdrawn**

### **KK3.15**

**Micro- and Nano-Structured P(VDF-TrFE) Copolymer and Terpolymer Films: Fabrication and Characterization.** Shihai Zhang<sup>1</sup>, Ziqi Liang<sup>2</sup>, Qing Wang<sup>1,2</sup> and Q.M. Zhang<sup>3,1</sup>;

<sup>1</sup>Materials Research Institute, The Pennsylvania State University, University Park, Pennsylvania; <sup>2</sup>Materials Science and Engineering Department, The Pennsylvania State University, University Park, Pennsylvania; <sup>3</sup>Electrical Engineering Department, The Pennsylvania State University, University Park, Pennsylvania.

Ferroelectric P(VDF-TrFE) copolymers and its derivative electrostrictive terpolymers exhibit electromechanical properties attractive for a broad range of microdevices. However, because of the incompatibility of the conventional photolithography technology, few microdevices have been developed based on these active polymers. Recently, we investigate the ink-jet printing and micro-stamping for fabricating micro- and nano-structured devices for these polymers. By properly controlling and modifying the surface energy, ink-jet printing of solution of P(VDF-TrFE) copolymer and electrostrictive terpolymer can produce structure at size below 30 microns. Making use of dewetting of these polymers in a surface energy structured surface, sub-micro sized features can be produced easily. It is also shown that these micron and nano-sized P(VDF-TrFE) copolymer and terpolymer films exhibit high ferroelectric or electrostrictive response.

SESSION KK4: Misfit Stress and Nanopatterning  
Chairs: George Gilmer and Enge Wang  
Tuesday Morning, November 30, 2004  
Room 308 (Hynes)

### **8:30 AM KK4.1**

**Trench Formation and Intermixing in Ge/Si(100) Islands.** Philippe Sonnet<sup>1</sup> and Pantelis C. Kelires<sup>2</sup>; <sup>1</sup>LPSE, CNRS, Moulhouse, France; <sup>2</sup>Physics Department, University of Crete, Heraklion, Crete, Greece.

The main strain relief mechanisms operating at high temperatures in Ge/Si(100) heteroepitaxy are trench formation and intermixing. Trenches are grooves of missing material which form around dome islands and large hut clusters, they have an anisotropic shape, and extend into the Si substrate. The details of their morphology are well analyzed, but the physical origin behind their formation is not so clear. Perhaps, the most complicated issue concerns the relation of trench formation and intermixing. The relative importance of these two effects for stress relaxation is not transparent. For example, the rates at which trenches nucleate and intermixing takes place at the various stages of growth are not known, even qualitatively. A related question, which has not been addressed, is when the initial stage of trench formation takes place. Here, we present recent work [1] which shed light onto these issues and unraveled the physical origin of trench formation. It is based on atomistic Monte Carlo simulations of stress buildup and relief in Ge dome islands on Si(100). The stress

field is mapped using the tool of local atomic stresses. The empirical potentials of Tersoff are used for the interatomic interactions. By choosing an appropriate growth model, and by monitoring the stress evolution as the island grows layer by layer, it is found that a trench is most likely being formed halfway during growth. The primary driving force for this phenomenon is the reduction of the concentrated stress below the edges of the island, but not the need to provide Si into it, as is widely believed. By considering intermixing/alloying at the interfacial region, it is also shown that initially the trench nucleation rate should be faster than the rate at which intermixing alloys the wetting layer and the island. Fast, initial intermixing would depress the driving force for trench nucleation. However, once the trench is formed, subsequent intermixing through it is enhanced, and compensates for the stress in the island. This analysis also provides an opportunity for a critical comparison of different growth models. [1] P. Sonnet and P. C. Kelires, *Appl. Phys. Lett.* 85, XXX (2004).

#### 8:45 AM KK4.2

**Morphological evolution of Ge island arrays assembled on Au-patterned Si(100).** Jeremy Robinson<sup>1,2</sup>, Andrew M. Minor<sup>2</sup>, Velimir Radmilovic<sup>2</sup>, J. Alexander Liddle<sup>2</sup> and Oscar D. Dubon<sup>1,2</sup>, <sup>1</sup>Materials Science and Engineering, University of California at Berkeley, Berkeley, California; <sup>2</sup>Lawrence Berkeley National Laboratory, Berkeley, California.

Ge on Si has been a model system to study self-assembly processes in misfit heteroepitaxial systems. The 4.2% lattice misfit between Ge and Si leads to growth via the Stranski-Krastanov (S-K) mode, which is characterized by a transition from initially layer-by-layer to 3D-island growth. In the growth of Ge on Si(100), Ge islands evolve in a random distribution and develop into a rich array of morphologies, including the so-called huts, domes, and super-domes. Here we present a new approach to control Ge island shape and position. We have achieved one and two-dimensional alignment of Ge islands grown by molecular beam epitaxy on Au-patterned Si(100). Arrays of Au dots are patterned by e-beam evaporation of 1nm of Au through a stencil mask. The dot size and spacing as well as the equivalent Au-layer thickness are observed to affect Ge island evolution and size distribution. For a square array of Au dots, a two-dimensional square lattice consisting of thousands of Ge islands less than 100 nm in height extending over hundreds of microns has been realized. Surprisingly, the Ge islands grow at the centers of the squares formed by the Au-dot arrays. Unlike the reported bimodal distribution of huts and domes formed on unpatterned Si(100), islands grown in the Au-patterned region are square-based truncated pyramids (TP) with {111} side-, {110} corner- and (100) top-facets. The morphological evolution of Ge islands with increased Ge coverage can be broken down into three growth regimes. At the lowest coverages (1.5 to 4ML-Ge), small lens-like islands form. At intermediate coverages (4 to 10 ML-Ge), these islands transform into fully developed truncated pyramids with an aspect ratio of approximately two to one (base to height). Cross-sectional transmission electron microscopy reveals that TP islands contain misfit dislocations. We have not detected the presence of Au in TP islands using electron dispersive x-ray spectroscopy. At higher coverages (10 to 50ML-Ge) TP islands nucleate additional facets (e.g. {113}) and transform into more superdome-like structures. Germanium huts and domes are observed in regions of the substrate where there is no Au even as truncated pyramids are forming in the Au-patterned region. These differences in island shape give clear indication of different island coarsening behaviors in these two regions. Unlike other substrate patterning techniques that require extensive substrate-to-substrate templating, the approach presented here is a fast, effective and versatile method to manipulate island growth kinetics and thereby direct island assembly over large areas.

#### 9:00 AM \*KK4.3

**Heteroepitaxial Self-Assembly of Higher-Complexity Structures By Combining Growth Control with Nanopatterning.** Jerrold A. Floro<sup>1</sup>, Joel L. Means<sup>1</sup>, Jennifer L. Gray<sup>2</sup>, Nitin Singh<sup>2</sup>, Robert Hull<sup>2</sup>, Dana Elzey<sup>2</sup>, Peter Sutter<sup>3</sup> and Chi-Hang Lam<sup>4</sup>; <sup>1</sup>Surface and Interface Sciences Department, Sandia National Labs, Albuquerque, New Mexico; <sup>2</sup>Department of Materials Science and Engineering, University of Virginia, Charlottesville, Virginia; <sup>3</sup>Center for Functional Nanomaterials, Brookhaven National Laboratory, Upton, New York; <sup>4</sup>Department of Applied Physics, Hong Kong Polytechnic University, Hong Kong, Hong Kong.

Strain-induced self-assembly during heteroepitaxial semiconductor growth can be used to produce quantum structures such as nanodots and nanowires. To rationally form more complex features, we manipulate the growth kinetics associated with molecular beam epitaxy of SiGe alloys on Si (001). Through use of limited growth temperatures, elevated deposition rates, and interspersed annealing steps, we can simultaneously self-assemble 4-fold quantum dot molecules, quantum dots, and high aspect quantum wires. These could potentially form the elements of nanologic circuitry schemes

based on quantum cellular automata. The behavior that determines the formation of quantum dot molecules and wires in our approach is the preferential self-assembly of pits instead of islands. In the first half of the talk, we will discuss the structure of quantum pits and their dynamic formation mechanism, based on AFM and in situ STM measurements, and kinetic Monte Carlo calculations. The second half of the talk will focus on directed self-assembly of quantum dot molecules using nanopatterns generated ex situ in the Si substrate by a focused ion beam. Periodic arrays of quantum dot molecules can be achieved over macroscopic distances, but the mechanism underlying the formation and placement process is non-trivial to understand. This work was partially supported by the DOE Office of Basic Energy Sciences. Sandia is a multiprogram laboratory of the United States Department of Energy operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

#### 10:00 AM \*KK4.4

**Abstract Not Available.** S. Williams

#### 10:30 AM KK4.5

**The Stress Driven Rearrangement Instabilities in Electronic Materials and in Helium Crystals.** Michael Grinfeld<sup>2</sup>, Pavel Grinfeld<sup>3</sup>, Haruo Kojima<sup>4</sup>, John Little<sup>1</sup>, R. Masutomi<sup>4</sup>, Per-Olof Persson<sup>3</sup> and Tsvetanka Zheleva<sup>1</sup>; <sup>1</sup>US Army Research Laboratory, Adelphi, Maryland; <sup>2</sup>US Army Research Laboratory, Aberdeen Proving Ground, Maryland; <sup>3</sup>Massachusetts Institute of Technology, Cambridge, Massachusetts; <sup>4</sup>Department of Physics, Rutgers University, New Brunswick, New Jersey.

It was demonstrated, on general thermodynamic grounds, that a flat boundary of non-hydrostatically stressed elastic solids is always (morphologically) unstable with respect to "mass rearrangement". The rearrangement can occur via a) melting-freezing or vaporization-sublimation processes at liquid-solid or vapor-solid phase boundaries, b) surface diffusion of particles along free or interface boundaries, c) adsorption-desorption of atoms in epitaxial crystal growth, etc. At present, many researchers believe that the Stress Driven Rearrangement Instability (SDRI) is a universal phenomenon over large length scales. This universality allows investigation of nano-scale SDRI effects in, say, semiconductor nano-technology by means of manifestations of the SDRI in macro-scale experiments with He4 crystals. We discuss the role of SDRI in the problems of solid nano-films epitaxy and low temperature physics. In particular, results related to the dislocation-free Stranski-Krastanov pattern of growth of semiconductor InGaAs quantum dots (QD) on GaAs substrate grown via molecular beam epitaxy are revealed as an evidence of the SDRI mechanism of formation of these nano-structures. The structural perfection, epitaxial quality, size, shape and density of the QD and their relationship to the growth parameters are discussed in terms of the SDRI theory. Then, we discuss our recent low temperature experiments to observe the solid-liquid interface of macro-crystalline He4 growing under nonhydrostatic stress. Theoretical analysis of the SDRI involves highly nonlinear equations allowing analytical methods only in the initial stage of unstable growth. At present, we are unable to explore analytically the most important deeply nonlinear regimes of growth. To avoid this difficulty we develop numerical tools facilitating the process of solving and interpreting the results by means of visualization of developing morphologies.

#### 10:45 AM KK4.6

**X Ray Diffraction Mapping of Strain Fields of SiGe:Si(001) Quantum Fortress.** Marina Leite<sup>1,2</sup>, Jennifer Gray<sup>3</sup>, Robert Hull<sup>3</sup>, Jerry Floro<sup>4</sup>, Rogério Magalhaes-Paniago<sup>5,1</sup> and Gilberto Medeiros-Ribeiro<sup>1</sup>; <sup>1</sup>Laboratorio Nacional de Luz Sincrotron, Campinas, Brazil; <sup>2</sup>Instituto de Física, Campinas, Brazil; <sup>3</sup>Department of Materials Science and Engineering, University of Virginia, Charlottesville, Virginia; <sup>4</sup>Sandia National Laboratories, Albuquerque, New Mexico; <sup>5</sup>Departamento de Física, Universidade Federal de Minas Gerais, Belo Horizonte, Minas Gerais, Brazil.

SiGe:Si(001) has been considered a model system for strained epitaxial growth. This assumption stems from the fact that only two elements are involved. Nevertheless, there is a wealth of morphologies that can be achieved given a particular set of growth conditions. Depending on kinetic and thermodynamic factors, a large number of interesting structures that may present quantum effects can be created. For instance, for the low growth temperature regime, hut pits can nucleate prior to island formation, and these faceted pits then act as precursors for pyramid formation. The faceting in these systems is comprised of (105) planes. Molecular Beam Epitaxy (MBE) of group IV semiconductors was used to explore the low temperature regime of SiGe alloys. Increasing film thickness (Si<sub>0.7</sub>Ge<sub>0.3</sub> at 550C and 0.9 Å/s), a sequence of three-dimensional metastable morphologies was observed. First, square pits were formed to relieve strain energy. Next, at the four upper edges of each pit, elongated islands of SiGe alloy



developed during growth. These structures are known as quantum fortresses (QFs) because of their four-walled shape. QF surface morphology and strain profile were studied using Atomic Force Microscopy (AFM) and X Ray Diffraction (XRD). AFM showed that QFs are uniform in size and shape and grow preferentially along the  $\langle 001 \rangle$  direction. It was also seen by AFM that QF inner walls consisted of  $\langle 105 \rangle$  facets. To examine QF chemical composition and buffer layer stress, angular [around (4 0 0) and (0 4 0) reflections] and radial XRD scans were performed at two different energies: 11.0 and 11.1 keV. Near the Ge K-absorption edge and 11.0 keV the scattering factor is notably different and the intensity variations measured are related to Ge concentration in the QF. XRD measurements showed lattice parameter ( $d$ ) variation from 5.362 to 5.485 Å, which brackets the Si lattice parameter (5.431 Å). Pits are initially formed to relax the strain in the film, probably increasing  $d$  on average in the local region. Smaller  $d$ , however, indicates lattice compressive strain, thus our XRD results point to the co-existence of relaxed and compressed strain in the QF's and substrate.

#### 11:00 AM KK4.7

**Strain-driven nucleation of Ge-islands ordered clusters on multilayered Ge/Si structures.** Riccardo Marchetti<sup>1</sup>, Francesco Montalenti<sup>1</sup>, Leo Miglio<sup>1</sup>, Giovanni Capellini<sup>2</sup>, Monica De Seta<sup>2</sup> and Florestano Evangelisti<sup>2</sup>; <sup>1</sup>INFN and L-NESS, Dipartimento di Scienza dei Materiali, University of Milano-Bicocca, Milano, Italy; <sup>2</sup>INFN and Dipartimento di Fisica, University of Roma Tre, Roma, Italy.

It has been experimentally observed that the growth of Ge islands on strain-modulated Si surfaces results, under suitable conditions, in the formation of nanometric clusters of ordered islands. We have investigated such phenomenon by large-scale atomistic simulations combined with experimental measurements. In particular, by using semiempirical Tersoff potentials, we have computed the strain field induced in a silicon matrix by mesa-shaped Ge islands. The resulting strain profile at the Si(001) surface has been calculated as a function of the capping-layer thickness, and used to build a suitable model describing the nucleation and the early stages of self-ordering for additional Ge dots grown on the capping layer. We show that such Ge islands tend to form dot clusters of various shapes which depend on the capping-layer thickness. The interplay between the width of the preferred nucleation regions and the island growth dynamics is also discussed. A good agreement between the model predictions and AFM measurements of CVD-grown multilayered Si/Ge structures is obtained in terms of island positioning within the cluster.

#### 11:15 AM KK4.8

**Buffer Layer Patterning of InAs/GaAs Quantum Dot Superlattices.** W. Ye, M. Reason, X. Weng and R. S. Goldman; Materials Science and Engineering, The University of Michigan, Ann Arbor, Michigan.

The vertical stacking of self-assembled quantum dot (QD) superlattices (SLs) is often explained by the preferred nucleation of islands at strain energy minima directly above buried dots. However, the mechanisms of lateral ordering of QDs are the subject of continued debate. For example, anisotropic lateral alignment of QDs has been observed in a number of systems, and long "chains" of laterally aligned QDs were recently reported. A significant remaining question concerns the effects of buffer layer patterning on this lateral QD alignment. For example, anisotropic "mounds" have been reported during the growth of GaAs films, but their formation has not been related to the alignment of QDs. Therefore, we have examined the patterning effects of buffer layers during the growth of QD SLs. Our QD SLs consisted of 2.6 ML InAs and 5 nm GaAs grown by molecular beam epitaxy at 500°C. Prior to QD deposition, GaAs buffer layers were grown under several different conditions, involving growth and/or annealing at 580°C and 500°C. For high temperature grown buffers, atomic force microscopy (AFM) reveals relatively flat surfaces with wide terraces. On the other hand, AFM reveals that low temperature grown buffers contain a high density of "mounds" elongated along the [1-10] direction. When single-layers of QDs are grown on either high or low temperature grown buffers, isotropic distributions of QDs are observed. Similar results are obtained for 5- and 10-period QD SLs grown on high temperature grown buffers. Interestingly, for 5- and 10-period QD SLs grown on low temperature grown buffers, preferential alignment of QDs along the [1-10] direction is apparent. This anisotropic QD alignment is enhanced as the number of QD SLs is increased. We propose a new mechanism for lateral QD alignment, which is based upon buffer layer patterning leading to undulated In-enriched GaAs spacer layers following the initial sets of QD SLs. Similar studies using focused-ion-beam and laser pre-patterned surfaces will also be presented. This work was supported in part by NSF, DOE, ARO, and NASA.

#### 11:30 AM KK4.9

**In-situ Monitoring of Formation and Overgrowth of InAs Quantum Dots.** Michael Yakimov, Vadim Tokranov, Gabriel

Agnello, Jobert van Eijsden and Serge Oktyabrsky; College of Nanoscale Science and Engineering, University at Albany - SUNY, Albany, New York.

With a goal of development of a high performance active medium for optoelectronic devices, formation of InAs self-assembled quantum dots (QDs) in GaAs/AlAs matrix was studied using in-situ methods: RHEED, Auger electron spectroscopy, along with post-growth techniques: TEM, photoluminescence and AFM. QD formation and evolution were analyzed with the emphasis on the role of AlAs or GaAs capping layers and shape engineering process. The observed benefits of a thin AlAs capping layer on top of QDs was found to result from the conformal coverage of pyramidal QDs, thus reducing the intermixing of InAs with the matrix. From the AFM measurements of the surface QDs and photoluminescence data, the QDs capped with AlAs appear to be larger than those grown in otherwise identical conditions but capped with GaAs. Auger electron spectroscopy was used to evaluate Indium redistribution during QDs capping and shape engineering process. Indium segregation coefficients were found to be almost identical for capping of a single monolayer of InAs by either AlAs or GaAs. We conclude that the QD size dependence on the chemistry of capping layer is primarily due to differences in Indium lateral diffusion on GaAs and AlAs surfaces. These differences are caused by two factors. Firstly, as we have previously shown<sup>1</sup>, diffusion coefficients of Indium adatoms on AlAs surface is much smaller than on GaAs. We believe this reduced diffusion coefficient inhibits transport of Indium between the dots capped by AlAs. Secondly, lower surface mobility of Aluminum adatoms results in conformal growth of AlAs overlayer on top of QDs, while GaAs growth on QD tip is much slower than between QDs. These arguments are supported by differences in RHEED pattern evolution during the overgrowth. Beneficial properties of the developed QD medium are demonstrated by evaluation of laser diodes that demonstrated unsurpassed thermal stability with maximum lasing temperature of 219 °C and a characteristic temperature of 380 K.<sup>2</sup> The QD structures can withstand two orders of magnitude higher defect density than quantum wells typically used in lasers.<sup>3</sup> 1 M. Yakimov, V. Tokranov, and S. Oktyabrsky, MRS Symp. Proc., 648 (2001) P2.6 2 V. Tokranov, M. Yakimov, A. Katsnelson, M. Lamberti, and S. Oktyabrsky. Appl. Phys. Lett., 83, (2003) 833. 3 M. Lamberti, A. Katsnelson, M. Yakimov, G. Agnello, V. Tokranov, and S. Oktyabrsky. MRS Symp. Proc., 799 (2004) Z5.36.

#### SESSION KK5: Misfit Stress, Dots and Nanowires

Chairs: Robert Hull and Zhenyu Zhang  
Tuesday Afternoon, November 30, 2004  
Room 308 (Hynes)

#### 1:30 PM KK5.1

**Island Electromigration on Si(001) Surface. 2D Monte Carlo Simulation.** Florin Nita<sup>1,2</sup> and Alberto Pimpinelli<sup>1</sup>; <sup>1</sup>LASMEA, Aubiere, France; <sup>2</sup>Institute of Physical Chemistry, Bucharest, Romania.

The island motion as result of a bias in surface migration of Si dimers was studied by kinetic Monte Carlo simulation, using a model that accounts for the decay of pyramidal nanostructures on Si(001) surface<sup>1,2</sup>. As for the nanopyramids decay, in the model we consider only the dimer diffusion events. The diffusion activation energy is given by:  $E_d = ED + n_{\perp}E_{a\perp} + n_{\parallel}|E_{a\parallel}|$ , where  $E_{a\perp}$  and  $E_{a\parallel}$  are the lateral interaction energies in perpendicular, respectively parallel direction relative to the dimer rows direction in the plain, and  $n_{\perp}$  and  $n_{\parallel}$  are the numbers of nearest neighbours in these two directions.  $ED$  is the energy barrier due to the interaction with the substrate and can have two different values,  $ED_{\perp}$  or  $ED_{\parallel}$ , depending on the dimer diffusion direction relative to the dimer rows direction in the substrate. The simulations were performed for different values of the diffusion bias. The island diffuse along the substrate and the speed of its centre of mass depends on the value of the bias. For the same sign of the bias, the direction of motion alternates on alternating reconstructed, (1x2) or (2x1), terraces. For the same orientation of dimer rows in the substrate, changing the sign of the bias, the island direction of motion changes with respect to the laboratory frame, but not with respect to the the sign of the bias. A good agreement was established between our simulation results and experimental data concerning the motion of silicon islands driven by DC current. [1] Decay Process of Pyramidal Islands on Si(001) Studied by Monte Carlo Simulation, Florin Nita, Alberto Pimpinelli, Hiroo Omi, 17emes Journées Surfaces et Interfaces - JSI 2003, Villeneuve d'Ascq, 29-31 January 2003, France [2] Scaling and crossovers in nano-island decay : a kinetic Monte Carlo study Florin Nita, Alberto Pimpinelli, Surface Science 551 (2004) 31-38

#### 1:45 PM KK5.2

**Surface patterns and morphological evolution of Titanium**

### Nitride islands in the pre-coalescence growth regime.

Panos Athanasios Patsalas and Stergios Logothetidis; Physics, Aristotle University, Thessaloniki, -, Greece.

In recent years the Titanium Nitride (TiN) research is focused to applications in electronics, such as diffusion barriers and ohmic contacts for GaN-based devices [1]. We employ in-situ Spectroscopic Ellipsometry (SE) to study the evolution of microstructure, stoichiometry and electronic properties of TiN grown on Si and GaN by reactive magnetron sputtering with varying ion irradiation conditions. In order to achieve sub-nm resolution for the SE analysis, we developed and validated the appropriate methods of analysis [2]. The in-situ SE study is complemented by thorough investigations of TiN surfaces using X-Ray Reflectivity (XRR) and Diffuse Scattering (XDS) and Atomic Force Microscopy (AFM). The SE and X-Ray results are compared and correlated with supporting Monte-Carlo simulations of the irradiation effects (SRIMTM) and surface diffusion of adatoms (SIMBAD<sup>TM</sup>). We found that the surface and interface morphology are sensitive to the mechanism of dissipation of the impinging ions. If the ion energy is enough to overcome the subplantation threshold (50 eV), the films are highly compressed and exhibit ultra-smooth surface and rough interface. On the other hand, when the ion energy is below the subplantation threshold the interface is atomically sharp and the surface morphology depends on the mobility and surface diffusion length of adatoms. However, in both cases these effects are dominant at the homogeneous growth. At the initial stages of nucleation and island growth the differences in the growth mode (island or layer) due to irradiation conditions are not pronounced and the thermodynamics of wetting of TiN on Si are prevailing factors. In this case, we identified a pure Volmer-Weber type of growth with four distinct stages. In the pre-coalescence growth regime (<12 nm) the TiN islands behave like isolated metal particles and manifest very strong quantum size effects in their optical and electronic properties. We model the SE data in order to identify the transport properties of conduction electrons; we determine the mean free path of conduction electrons of the TiN islands and we quantitatively correlate it with the island size and surface roughness (examined by XRR/XDS and AFM) for the various irradiation conditions. This correlation enables the identification of secondary scattering sites of the conduction electrons (in addition to island boundaries) and may provide information on the presence of structural defects into the islands. We finally investigate the evolution of island size and distribution during island growth with regards to the various irradiation and stress conditions. [1] S. Gautier, Ph. Kominou, P. Patsalas, et al, Sem. Sci. Technol 18, 594 (2003). [2] P. Patsalas, S. Logothetidis, J. Appl. Phys. 93, 989 (2003).

### 2:00 PM \*KK5.3

**Kinetic Limitations in Two- and Three-Dimensional Growth.** K.L. Man<sup>1</sup>, Wenxin Tang<sup>1</sup>, Hanchen Huang<sup>2</sup> and Michael Altman<sup>1</sup>; <sup>1</sup>Physics, Hong Kong University of Science and Technology, Kowloon, Hong Kong; <sup>2</sup>Mechanical Engineering, Rensselaer Polytechnic Institute, Troy, New York.

Advances in the fabrication of nanostructures and devices depend largely upon the degree to which one can understand and control the growth process. Growth shape and morphology will be affected, or even dictated, by kinetic limitations that may be present during growth. One such limitation, which has received a great deal of attention, occurs in growth at surfaces when there is a Schwoebel-Ehrlich (SE) diffusion energy barrier to atomic motion descending a monolayer height step. In this presentation, it will be shown how the impact of the SE barrier on growth morphology may be hindered in real systems by other factors such as step permeability and the relative importance of diffusion and step attachment, i.e., the rate limiting step. We also present evidence of an analogous 'three-dimensional' (3D) SE energy barrier to atomic diffusion across the ridge that separates two facets on a three-dimensional crystal. The 3D SE barrier stems from the reduced coordination at the ridge, and differences of the adatom formation energies on adjacent facets cause the 3D SE barrier to be asymmetric. Kinetically limited growth shapes of Ag crystallites on the Si(111) surface have been studied with low energy electron microscopy (LEEM) and diffraction (LEED). LEEM observations of the modification of growth shapes caused by codeposition of surfactants are consistent with modification of the 3D SE barrier caused by surface passivation. This suggests that nanocrystal growth shape may be manipulated by exercising control over the 3D SE barrier. The 3D SE barrier is also expected to be relevant to diffusion in the presence of multilayer height steps on surfaces, and is therefore also important for the development of film texture.

### 3:00 PM \*KK5.4

**Self-assembly of uniform metal nano-clusters and nano-platelets on Si(111) surface.** Qi-Kun Xue, The Chinese Academy of Sciences, State Key Lab for Surface Physics, Beijing, China.

Fabrication and understanding of nanoclusters and nanodots have become one of the most exciting research areas, as driven by their great potential applications in technology and scientific significance to bridge our understanding between molecular and condensed matter physics. A barrier hindering the development of this area has been the problem in fabrication of these nanostructures with uniform size, composition and spatial distribution. In this work, we solve the problem by a novel approach to coax metal atoms (Al, Ga, In, and Pb) into arrays of evenly-distributed identical clusters in which surface mediated magic clustering was used to achieve identical cluster size, while the Si(111)-7 x 7 reconstructed surface as a template for ordering the clusters. Furthermore, using the ordered Al cluster covered Si surface as a template, we could also be able to fabricate self-organized Co nanoplatelets with a singular height, quantized lateral sizes, and unique shape and spatial orientation. Despite their relatively small volume (only a few nm<sup>3</sup>), these nanomagnets exhibit an unusually high blocking temperature (>100 K). In this talk, I will discuss the formation mechanism, structural and magnetic properties of these nanostructures.

### 3:30 PM KK5.5

#### **Self-assembly of BaTiO<sub>3</sub>-CoFe<sub>2</sub>O<sub>4</sub> Nanostructures.**

Haimei Zheng<sup>1</sup>, L. Mohaddes-Ardabili<sup>1</sup>, Junling Wang<sup>1</sup>, L.

Salamanca-Riba<sup>1</sup>, D. G. Schlom<sup>2</sup>, Long-qing Chen<sup>2</sup> and R. Ramesh<sup>3</sup>; <sup>1</sup>Department of Materials Science and Engineering, University of Maryland, College Park, College Park, Maryland; <sup>2</sup>Department of Materials Science and Engineering, Pennsylvania State University, University Park, Pennsylvania; <sup>3</sup>Department of Materials Science and Engineering and Department of Physics, University of California, Berkeley, California.

BaTiO<sub>3</sub>-CoFe<sub>2</sub>O<sub>4</sub> nanostructures have been spontaneously formed during heteroepitaxial growth on a single crystal SrTiO<sub>3</sub> (001) substrate by pulsed laser deposition. The patterns of the nanostructures change dramatically as the film thickness increases. Above certain thickness (200 nm) films form well-organized CoFe<sub>2</sub>O<sub>4</sub> nano-pillar arrays embedded in a BaTiO<sub>3</sub> matrix. The lateral size of the pillars increased from 9 nm to 70 nm when the substrate temperature increases from 750 °C to 950 °C, which can be fitted to an Arrhenius behavior. Lattice mismatch strain, elastic modulus of the two phases and interface energy are considered to play an important role in the dynamic pattern formation of BaTiO<sub>3</sub>-CoFe<sub>2</sub>O<sub>4</sub> nanostructures. This work is supported by the NSF-MRSEC under contract No. DMR-00-80008.

### 3:45 PM \*KK5.6

**Self-Assembly and Shape Transitions of Epitaxial Nanowires and Strained Monolayer Islands.** Vivek B. Shenoy, Division of Engineering, Brown University, Providence, Rhode Island.

Several interesting shape transitions have been recently observed during the growth of submonolayer islands on lattice-mismatched substrates. These shapes, which allow relaxation of mismatch strain, include nanowires that are elongated along certain crystallographic directions with widths in the single-digit nanometer range, shapes that show concave boundaries in equilibrium and formation of highly ramified or branched structures during growth. In this talk, I will present a sharp interface model and a phase-field model to study the shapes (kinetic and equilibrium) of individual islands and stability and coarsening kinetics of monolayer island arrays. Our model includes the kinetics of adatom diffusion on the terraces and island edges, attachment kinetics to islands, substrate-mediated elastic interactions between the islands and anisotropies associated with the creation of island edges. Particular emphasis will be given to self-assembled growth of regular arrays of epitaxial nanowires that have potential applications as non-lithographically fabricated interconnects.

### 4:15 PM KK5.7

**Endotaxial Silicide Nanowires.** Zhian He<sup>2</sup>, David J. Smith<sup>3,1</sup>, Frances M. Ross<sup>4</sup> and Peter Bennett<sup>1,2</sup>; <sup>1</sup>Physics, Arizona State University, Tempe, Arizona; <sup>2</sup>Science and Engineering of Materials, Arizona State University, Tempe, Arizona; <sup>3</sup>Center for Solid State Science, Arizona State University, Tempe, Arizona; <sup>4</sup>T. J. Watson Research Center, Yorktown Heights, New York.

We demonstrate the growth of self-assembled nanowires (NWs) of cobalt silicide on Si (111), (100) and (110) substrates by deposition of Co onto a heated Si substrate. Island structures are determined using Atomic Force Microscopy, High-Resolution Transmission Electron Microscopy (TEM) in cross-section and in situ UHV-TEM in plan-view. Silicide NWs grow via an *endotaxial* (embedded epitaxy) mechanism, with coherent B-type interfaces along inclined Si planes, which breaks the symmetry of the surface, and leads to a long, thin nanowire shape. During growth, the length and width of the islands increase linearly with time in a fixed proportion that varies strongly with growth temperature, showing that the nanowire shape is

kinetically determined. It is expected that NWs could form in many other overlayer/substrate systems via this mechanism.

#### 4:30 PM KK5.8

**Nanowire growth using the annealing and pulsed laser deposition.** Hyun D. Park and Timothy P. Hogan; Electrical & Computer Engineering, Michigan State University, East Lansing, Michigan.

Nanowire growth via the annealing is a simple technique that primarily consists in raising the temperature of a given substrate predeposited with a suitable metal catalyst. Previously, there has been reported nanowire growth just from annealing where silicon oxide nanowires have been grown using gallium as the metal catalyst and the wafer itself as the source material. Recently, we have also been able to demonstrate the growth of nanowires by simple annealing using gold as the metal catalyst in the growth of silicon oxide nanowires as well as nanowires containing indium and antimony using silicon and InSb substrates, respectively. Despite the simplicity in the growth technique, the results in particular from the InSb substrate annealing have not been high quality, with evidence of tapering, variation in the chemical composition along the length of the nanowire, as well as crystal quality not quite reaching the desired single crystal. In order to resolve the shortfalls from the annealed results, a new growth technique using the annealing and pulsed laser deposition is found. InSb substrate as the nanowire source material and gold target as the metal catalyst were used to demonstrate the new technique. The new technique allows the precise control over the growth temperature thereby resulting in higher quality nanowires than obtained from simple annealing alone. The control over the density and the diameter of the nanowires is also shown. The results from the variation of gold deposits at the fixed growth temperature of 510 °C are shown with scanning electron microscope (SEM) and high resolution transmission electron microscope (HRTEM) images.

#### 4:45 PM KK5.9

**Growth of Amorphous SiO<sub>2</sub> Nanowires on Si using a Pd/Au Thin Film as a Catalyst.** Jose L. Elechiguerra<sup>1</sup>, Jorge A. Manriquez<sup>3</sup> and Miguel Jose-Yacamán<sup>1,2</sup>; <sup>1</sup>Chemical Engineering, The University of Texas at Austin, Austin, Texas; <sup>2</sup>Texas Materials Institute, The University of Texas at Austin, Austin, Texas; <sup>3</sup>Centro de Manufactura, Tecnológico de Monterrey, Monterrey, N.L., Mexico.

Nanowires of amorphous SiO<sub>2</sub> were synthesized by thermal processing of a (100) Si substrate at 1100 C in the presence of a nitrogen flow, and using a 15 nm thick Pd/Au film as a catalyst. The substrate itself was the only source of silicon for the nanowire growth. The nanostructures produced were characterized by high resolution transmission and scanning electron microscopy and by x-ray diffraction. The nanowire growth is consistent with the vapor-liquid-solid (VLS) mechanism, with particles of Pd<sub>2</sub>Si and Au(Pd) being observed to form from the reaction between silicon and the catalytic film, and to remain at the tip of the wires. The synthesized nanowires showed a well defined flower-like morphology which could be very interesting for lasing applications.

SESSION KK6: Ion Beam Patterning of Surfaces  
Chairs: Michael Aziz and Ramki Kalyanaraman  
Wednesday Morning, December 1, 2004  
Room 308 (Hynes)

#### 8:30 AM KK6.1

**Coupled Atomistic and Continuum Model for Generation of Surface Morphology in Silicon by Ion Bombardment.** Harley T. Johnson<sup>1</sup>, Nagarajan Kalyanasundaram<sup>1</sup> and Jonathan B. Freund<sup>2</sup>; <sup>1</sup>Mechanical & Industrial Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois; <sup>2</sup>Theoretical and Applied Mechanics, University of Illinois at Urbana-Champaign, Urbana, Illinois.

Semiconductor quantum dot arrays are promising for a wide range of applications in optoelectronics, nanoelectronics, biology, and quantum computing. Successful fabrication of regular, uniform lateral arrays of quantum dots by self-assembly methods continues to be an important problem. Recent work by several experimental groups has identified a possible new method for generating arrays of semiconductor quantum dots using a simple ion-bombardment process. The basic mechanism of rippling and sputter-induced surface instability in semiconductors has been understood for some time, but little work has been done to connect the continuum level mechanisms to atomistic scale surface features associated with the ion-bombardment process. This talk describes recent work to study the ion sputtering surface instability using a multiscale model that combines molecular dynamics and continuum surface evolution methods. The effects of individual ion

impacts are studied using a molecular dynamics analysis of medium energy (500eV-700eV) argon ion bombardment of silicon. Empirical potentials are used to model the argon and silicon; sputtering and stress results are in agreement with experimental observations. Both sputtering and ion-assisted surface rearrangement effects are considered, and the statistics are then input into a continuum equation describing the surface and strain energy driven diffusive mass transport active over much longer time scales. The surface profile is then calculated using an implicit time integration, with consideration of changing elastic fields as the surface shape evolves. The analysis is valid for the low temperature regime in which the surface remains strongly disordered. Results of the multiscale analysis are compared to the arrays of quantum dots grown experimentally by the ion bombardment method.

#### 8:45 AM KK6.2

**Ripple and Dot Pattern Formation on Silicon Surfaces by Low-Energy Ion Beam Erosion.** Bashkim Ziberi, Frank Frost, Thomas Hoeche and Bernd Rauschenbach; Leibniz-institute for surface modification, Leipzig, Germany.

Due to different roughening and smoothing mechanisms, a multitude of topographies can result from surface erosion by low-energy ion beams. Under certain conditions, sputtering can roughen the surface resulting in a pronounced topography evolution in some cases producing well-ordered patterns. The underlying mechanism for the evolution of regular nanometer structures with typical dimension < 100 nm, is a self-organization process caused by the interplay of roughening by curvature dependent sputtering and surface smoothing by different surface relaxation mechanisms (e.g., atomic transport stimulated by ballistic drift and diffusion processes). In this work, results for self-organized pattern formation on Si surfaces during low-energy Ar<sup>+</sup> ion beam erosion (ion energy ≤ 2000 eV) under normal and oblique ion incidence with and without sample rotation are presented. These self-organized patterns were analyzed by scanning force microscopy (AFM) and high resolution transmission electron microscopy (HRTEM). It will be shown that ordered nanodots are formed at room temperature for normal and oblique ion incidence angles in the investigated ion energy range. In the case of simultaneous sample rotation during ion beam erosion, dot patterns formed at oblique ion incidence angles between 70° and 80° with respect to the surface normal show a remarkably high degree of ordering comparable to dot patterns reported for different III/V compound semiconductors [1,2,3]. Without sample rotation, strikingly ordered ripple patterns are formed at ion incidence angles slightly deviating from normal ion incidence with a mean wavelength < 50 nm. The mean size and ordering of these nanostructures (dots, ripples) can be adjusted by various process parameters like ion beam energy and erosion time, respectively. In contrast to advanced lithographic methods and subsequent etching procedures for pattern production with structure sizes < 200 nm, which are complex technological processes, self-organized pattern formation by low energy ion beam erosion, is a cost-efficient 'bottom up' approach for the fabrication of nanostructures. With suitable broad beam ion sources, it is possible to produce large-area nanostructured surfaces. [1] S. Facsco, T. Dekorsy, C. Koerdt, C. Trappe, H. Kurz, A. Vogt, H. L. Hartnagel, Science 285 (1999) 1551-1553. [2] F. Frost, A. Schindler, F. Bigl, Phys. Rev. Lett. 85 (2000) 4116-4119. [3] F. Frost, B. Ziberi, T. Hoeche, B. Rauschenbach, Nucl. Instrum. Methods Phys. Res. B 216 (2004) 9-19.

#### 9:00 AM \*KK6.3

**Real-time monitoring of nanoscale surface morphologies during ion beam erosion.** Soenke Habenicht, <sup>1</sup>Philips Semiconductors Hamburg, Hamburg, Germany; <sup>2</sup>Institute of Physics, Georg-August University of Goettingen, Goettingen, Germany.

Using focused ion beam microscopy one is enabled to monitor in-situ the morphology evolution of solid surfaces during ion beam erosion on a nanometer scale in real-time. Within this technique - while scanning the ion beam over the exposed surface - the same Ga-FIB-ion beam is used for erosion of the surface via sputtering and microscopy simultaneously by using the secondary electron emission during implantation. This method, combining focused ion beam technology and secondary electron microscopy, has been used to monitor the evolution of nanoscale surface ripples - in this case on Si-surfaces - after oblique incidence of the ion beam and especially the ripple propagation over the surface and with increased erosion time. The physical background as well as the implications out of these experiments will be discussed.

#### 9:30 AM KK6.4

**Temperature Dependence of Sputter Ripple Formation on Si(111).** Ari-David Brown and Jonah Erlebacher; Materials Science and Engineering, Johns Hopkins University, Baltimore, Maryland.

The time evolution of ripple surface pattern formation during low

energy ion sputtering provides a sensitive probe of microscopic surface kinetics. In this study, we report the morphological evolution of one-dimensional self-organized arrays of Ar<sup>+</sup> ion etched sputter ripples form on heated Si(111). The time evolution of ripple formation was studied in situ using UV light scattering spectroscopy in a temperature range from 600-750 C. A number of interesting kinetic phenomena will be discussed including the observation of an amorphous to crystalline transition resulting from a competition between ion induced amorphization and solid phase epitaxial regrowth, and activation energies for mass transport.

**9:45 AM KK6.5**

**Self-Organized Ripple Morphologies during Focused Ion Beam Milling of Ge.** Stefan Ichim and Michael J. Aziz; Div. Engrg. & Appl. Sci., Harvard University, Cambridge, Massachusetts.

We report the use of the boundary between ion irradiated and unirradiated regions to template the lateral self-organization of nanoscale ripple-like morphological features that spontaneously evolve during uniform ion irradiation. Using uniform rastering of a 30 keV Ga<sup>+</sup> focused ion beam, up to eight periods of ripples have been templated to follow the boundary under the set of conditions explored. We report the dependence of the range of lateral templating on incident angle, ion dose, and boundary inclination with respect to the projected ion beam direction. We discuss the implications for the fundamental physics underlying the morphological instability.

**10:30 AM \*KK6.6**

**Nonlinear Stabilization Mechanisms in Amplitude Saturation During Sputter Ripple Formation on Silicon.** Jonah Erlebacher and Ari-David Brown; Materials Science and Engineering, Johns Hopkins University, Baltimore, Maryland.

Sputter rippling refers to the formation of regular surface patterns during glancing incidence energetic ion beam etching of surfaces, usually as a result of a competition between etching (from the ion beam) and capillary action (driving smoothing via surface diffusion). Many different kinds of morphologies are often observed, including ripples oriented parallel or perpendicular to the projected ion beam direction and "quantum dots" arranged in hexagonal or rectangular arrays. Theoretical analyses of ripple evolution have concentrated on the initial stages of the surface instability leading to pattern formation, and the details associated with the non-linear mechanisms leading to amplitude saturation and pattern stabilization remain a subject of active interest. The Si(111) surface is a single component surface with isotropic diffusion kinetics; for these reasons, this system provides a useful probe of surface evolution without complicating effects of compositional inhomogeneities and anisotropic diffusion. Our examination of the Si(111) surface indicates that step fluctuation dynamics may play an important role in the long-term time evolution of sputter ripples in this system. To argue for this conclusion, a comparison with sputter rippling evolution on Si(001) will be made.

**11:00 AM KK6.7**

**Lateral Templating for Guided Self-Organization of Si Sputter Ripple Morphologies.** Alexandre Cuenat<sup>2,1</sup>, H. Bola George<sup>1</sup>, Kee-Chul Chang<sup>3</sup>, Jack M. Blakely<sup>3</sup> and Michael J. Aziz<sup>1</sup>; <sup>1</sup>Div. Engrg. & Appl. Sci., Harvard University, Cambridge, Massachusetts; <sup>2</sup>Applied Nanotechnologies, Materials Centre, National Physical Laboratory, Teddington, Middlesex, United Kingdom; <sup>3</sup>Materials Science Center, Cornell University, Ithaca, New York.

We study the influence of a patterned Si substrate on self-organized ripples spontaneously appearing during uniform Ar<sup>+</sup> ion irradiation. We show that the long-range order of the ripples can be greatly enhanced by imposing lateral boundary conditions on the sputter instability. The self-organization can be controlled by changing the size of pre-patterned regions made on the substrate. Conditions to control and guide the self-organization are discussed. The effect of nonlinearities in the model for ripple evolution in the presence of boundaries is discussed. These observations demonstrate the potential utility of lateral templating to guide the self-organization of surface nanostructures.

**11:15 AM KK6.8**

**Sputtered Morphology of Cu(001) Surface in Different Temperature-Flux Phase Space.** Wai Lun Chan and Eric Chason; Division of Engineering, Brown University, Providence, Rhode Island.

A wide range of surface morphologies can be produced on a surface during low energy ion bombardment (100-1000eV). These range from periodic structures (ripples, domes and pits) to kinetic roughening. Two major roughening mechanisms are usually identified as responsible for the ion-induced patterning, namely: 1) curvature dependence on the sputter yield and 2) presence of a Schwoebel barrier to interlayer transitions. Each of those mechanism can

dominate at different points in the temperature-flux phase space. We have measured the temperature-flux conditions for transitions between different regimes of morphological evolution on the Cu(001) surface. A simple model balancing the rates for the two roughening mechanisms and smoothing by surface transport is used to explain the experimental results. This work was supported by the U.S. Department of Energy under contract DE-FG02-01ER45913.

**11:30 AM KK6.9**

**Fabrication and Atomistic Modeling of Ion-Etch Nanostructures on Substrates.** Maria Stepanova and Steven K. Dew; Electrical and Computer Engineering, University of Alberta, Edmonton, Alberta, Canada.

We have implemented and investigated numerically a new process to fabricate self-organized metal dots, networks, and nanowires on non-metallic substrates. We have deposited a thin film of Cu on Si or SiO<sub>2</sub> substrates and etched the film by a neutralized Ar beam in a standard ion mill system. Due to the kinetic mechanism known as the sputter instability, ion etching produces a nonequilibrium surface morphology. At the stage when the etched surface approaches the substrate, we have investigated the surface by SEM and observed self-organized Cu nanopatterns embossed on the substrate. The characteristic size of the Cu features ranges between 15nm and 40nm. The pattern shape can be partially controlled by changing the angle of ion incidence. Normal incidence leads to arrays of Cu dots and networks whereas grazing incidence produces Cu nanowires directed along the projection of the ion beam on the surface. We also report our numerical investigation of this process. Our model comprises a kinetic Monte-Carlo simulation of nonequilibrium ion-etch morphologies, conversion of the surface morphologies into arrays of Cu clusters on a substrate, and coarsening of the surface patterns. Our simulations correctly reproduce the observed trends and identify major control factors for the process. The reported process of nanofabrication has a strong potential for nanoelectronics and photonics.

SESSION KK7: Novel Techniques and Applications of Nanopatterning

Chairs: Eric Chason and Hanchen Huang  
Wednesday Afternoon, December 1, 2004  
Room 308 (Hynes)

**1:30 PM KK7.1**

**Nanopatterning of GaAs(110) Vicinal Surfaces by Hydrogen-Assisted MBE.** Miguel L. Crespillo<sup>1</sup>, Jose L. Sacedon<sup>1</sup>, Bruce A. Joyce<sup>2</sup> and Paloma Tejedor<sup>1</sup>; <sup>1</sup>Instituto de Ciencia de Materiales de Madrid, CSIC, Madrid, Madrid, Spain; <sup>2</sup>Department of Physics, Imperial College of Science, Technology and Medicine, London, United Kingdom.

Homoepitaxial growth of GaAs on (110) vicinal surfaces under different kinetic regimes leads to the formation of distinct morphological instabilities associated with step propagation in the presence of Ehrlich-Schwoebel barriers at step edges. Thus, step bunching in the Ga-supply limited regime is attributed to diffusion-mediated step attraction due to preferential attachment of Ga adatoms from the upper terraces. On the other hand, growth under As-deficient conditions leads to the formation of meanders, ripples and pyramidal mounds on the surface, indicating that the adsorbed arsenic species undergo reaction and incorporate to surface steps preferentially from the lower terrace. We have studied the changes undergone by the GaAs(110) surface misoriented towards (111)A by 2 deg, in both growth kinetics and surface morphology during homoepitaxy when i) native oxide removal is carried out at low temperatures under a flux of atomic hydrogen, but growth is carried out in the absence of atomic hydrogen and ii) when both oxide removal and epitaxial growth are carried out in the presence of atomic hydrogen. Under As-rich conditions (450 C, V:III ratio=30), we observe a substantial reduction in macrostep height and terrace width when native oxide desorption is carried out at 450 C with H\* prior to growth from As<sub>4</sub> and Ga beams. In both cases growth proceeds in the step flow mode and leads to the formation of a rather straight step morphology, with step edges along <1-10> predominating over <1-12>-type steps. In contrast, growth of GaAs in the presence of H\* after oxide removal in the same ambient takes place by simultaneous step flow and bidimensional nucleation on the terraces, the macrostep height is also reduced and the step edges are preferentially aligned along the <1-12> direction. On the other hand, hydrogen-assisted MBE growth of GaAs on the vicinal (110) surface under As-deficient conditions (450 C, V:III ratio=10) proceeds by step flow in the presence of Ehrlich-Schwoebel barriers, as revealed by the self-organized pattern of pyramidal mounds aligned along the tilt direction, <001> and bounded by <1-12> edges. While this well-ordered structure is formed after deposition of 500 monolayers

(ML) of GaAs in the presence of H\*, we had previously observed the formation of a similar pattern by conventional MBE growth only after deposition of 1500 ML of GaAs. As opposed to growth limited by adatom incorporation kinetics at low temperatures, we have invariably observed a characteristic faceting of the surface with very straight steps aligned along the <1-10> direction when GaAs growth is carried out at 620 C with a V:III ratio of 10, regardless of atomic hydrogen being used during oxide desorption and/or epitaxial growth. v

#### 1:45 PM **KK7.2**

**On the Formation Kinetics of Thin Nanopatterned Layers on Silicon Wafers Created by Hydrogen Plasma Exposure.**  
Reinhart Job, Yuelong Huang, Yue Ma and Wolfgang Duengen; Electrical Engineering and Information Technology, University of Hagen, Hagen, Germany.

Plasma hydrogenation applied on standard (100)-oriented Czochralski silicon (Cz Si) wafers at about 250 C causes a nanopatterning of the treated surface regions. Patterning with structures sizes well below 100 nm can be achieved depending on the process conditions, i.e. thin nanophase silicon layers can be created. The formation of such nanostructures at the surface regions of plasma hydrogenated wafers was investigated for various process conditions. The plasma treatment was applied either at 13.56 MHz or 110 MHz frequency. The impact of post-hydrogenation annealing in vacuum on the morphology and evolution of the nanostructures was studied up to 1200 C. The properties of the plasma treated wafer surface and subsurface regions were analyzed by atomic force microscopy (AFM), high resolution scanning electron microscopy (SEM), and depth resolved micro-Raman spectroscopy. The formation kinetics of the nanopatterned silicon layers can be discussed on base of a combined etching/redeposition mechanism. During the H-plasma treatment Si atoms are released from the wafer surface, and small Si crystallites are redeposited again. During this process meta-stable Si-H molecules (most probably SiH<sub>2</sub>) might be formed in the H-plasma, which are decomposed again and induce the redeposition of the small Si crystallites onto the wafer surface. It was observed that the formation of redeposited crystallites with (111)-orientation is energetically more favourable than the one with (100)-orientation. During annealing at temperatures of 800 C or higher the crystallites in nanopatterned surface layer are dissolved again, and Si atoms formerly located in the redeposited crystallites are rebuilt onto the wafer surface. This reconstruction is not optimally executed, i.e. the initially (100)-oriented surface is not completely reconstructed again, and extended damage areas with (111)-orientation remain at the wafer surface. Therefore, after high temperature annealing, the reconstructed surface and subsurface layers of the treated wafers exhibit strong tensile stress up to rather deep wafer regions, as was observed by depth resolved micro-Raman analysis. The impact of the hydrogen related defects and the nanopatterning at the wafer surface on various semiconductor device structures will be briefly discussed.

#### 2:00 PM **\*KK7.3**

**Spontaneous Structural Pattern Formation at the Nanometer Scale in Kinetically Restricted Homoepitaxy on Vicinal Surfaces.** H.-J. Ernst, L. Douillard, N. Neel and T. Maroutian; CEA Saclay, DSM/Drecam/SpSci, Gif-Sur-Yvette, France.

The concept of spontaneous pattern formation in epitaxial growth is currently actively explored as a promising pathway for lateral nanostructuring of surfaces. Often, the origin of self-organization is traced back to the presence of an excess energy barrier for adatom diffusion associated with asymmetric features in the crystalline structure, the Ehrlich-Schwoebel barrier. Upon growth of Cu on vicinal Cu surfaces at moderate substrate temperatures a step-meandering instability develops, resulting in an in-plane patterning of the surfaces at the nanometer scale with a temperature- and flux-dependent characteristic wavelength. This meandering instability is superseded by a step-bunching instability during growth at higher temperatures. Specifically, the meandering instability acts as a precursor to the bunching instability, indicating that a one-dimensional treatment of bunching in step flow growth is not sufficient. These nanostructured surfaces might be used as templates in order to guide the growth of materials, which do not show spontaneous self-organization.

#### 2:45 PM **\*KK7.4**

**Control of Carbon Nanofiber Synthesis by Catalyst Nanoparticle Crystal Structure, Orientation, and Faceting.** Anatoli V. Melechko<sup>1,2</sup>, Igor A. Merkulov<sup>1</sup>, Kate L. Klein<sup>1,2</sup>, Jack C. Wells<sup>3</sup>, Douglas H. Lowndes<sup>4</sup> and Michael L. Simpson<sup>1,2,4</sup>;

<sup>1</sup>Molecular-Scale Engineering and Nanoscale Technologies, Oak Ridge National Lab, Oak Ridge, Tennessee; <sup>2</sup>Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee; <sup>3</sup>Computational Materials Sciences, Oak Ridge National Lab, Oak Ridge, Tennessee; <sup>4</sup>Condensed Matter Sciences, Oak Ridge National Lab, Oak Ridge, Tennessee.

Carbon nanofibers can be produced by decomposition of hydrocarbon compounds on surfaces of nanoparticles of transition metals such as Fe, Ni, and Co at moderately high temperatures. Carbon nanofibers consist of graphene layers wrapped into a conical ("herring-bone") or a cup-like ("bamboo") shape that are stacked to form cylindrical nanostructures. The shape of these graphene cones (i.e. cone angle) determines the type of material that is produced. For example, multiwall nanotubes are nanofibers with a zero cone angle. The cone angle depends on the shape of the surface of the catalyst nanoparticle at the time they are formed, orientation of the catalytically active surface, kinetics of the deposition process etc. In this work we present electron microscopy studies of catalyst nanoparticles (Ni, Fe, and Co) and vertically aligned carbon nanofibers produced by plasma enhanced chemical vapor deposition on Si(111) and Si(001) surfaces. The results show the correlation between nanoparticle orientation and the Si substrate that in its turn is reflected in the atomic structure of carbon nanofibers. The relationship between catalyst particle equilibrium shape (i.e. faceting) of Ni, Fe, and Co that are fcc, bcc, and hcp, respectively, is studied with regard to structure and synthesis dynamics of the resulting nanofibers. A geometrical model based on an assumption of growth rate anisotropy is utilized to explain the shape of the catalyst particle-carbon nanofiber interface.

#### 3:15 PM **KK7.5**

**Synthesis of Ultrasharp Vertically Aligned Silicon Nanocones by Catalytic Plasma Enhanced Chemical Vapor Deposition.** K. L. Klein<sup>1,2</sup>, A.V. Melechko<sup>1,2</sup>, P.D. Rack<sup>4</sup>, D.K. Hensley<sup>1</sup>, J.D.

Fowlkes<sup>2</sup>, H.M Meyer<sup>3</sup>, L.F. Allard<sup>3</sup>, D.H. Lowndes<sup>4</sup> and M.L Simpson<sup>1,2</sup>; <sup>1</sup>Molecular-Scale Engineering and Nanoscale Technologies Research Group, Oak Ridge National Laboratory, Oak Ridge, Tennessee; <sup>2</sup>Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee; <sup>3</sup>Microscopy Microanalysis and Microstructures Group, Oak Ridge National Laboratory, Oak Ridge, Tennessee; <sup>4</sup>Condensed Matter Sciences Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

We present an innovative method for the production of vertically aligned, ultrasharp silicon nanostructures with tip diameters as small as 10 nm. Silicon nanocones were produced using dc plasma-enhanced chemical vapor deposition (dc-PECVD) using thin film copper or gold as a catalyst and the silicon substrate as a sole source of silicon. High resolution SEM, TEM, EDX, STEM, and AES were utilized to determine the microstructure and composition of the nanocones. We have explored variations in the structure and growth mode of these nanocones with respect to growth environment. This structure will be described in detail and a growth mechanism proposed. The similarities and differences of this new growth process as compared to standard SiH<sub>4</sub>-based vapor-liquid-solid growth will be discussed. Lastly, we will describe their potential use in applications such as field emission cathodes and gene delivery arrays.

#### 3:30 PM **KK7.6**

**Self-Assembly and Dynamics of Oxide Nano-Rods on NiAl(110).** John Philip Pierce and Kevin F. McCarty; Materials Physics, Sandia National Laboratories, Livermore, California.

We observe the spontaneous formation of parallel rod structures upon exposing a clean NiAl(110) surface to oxygen at elevated temperatures (850-1250 K). By following the self-assembly of individual nano-rods in real time with low-energy electron microscopy (LEEM), we are able to investigate the kinetic processes by which the rods lengthen along their axes and thicken in the direction normal to the surface of the substrate. The oxide rods, whose lengths can be in excess of several microns while their heights and widths are quantized respectively in units of 2 Å and 16 Å, lengthen across virgin areas of the substrate at a constant rate if the oxygen pressure and growth temperature are fixed. The exponential dependence of this axial growth rate on temperature yields an activation energy of 1.1 eV. We do not observe changes in the axial growth rate as growing rods pass within very close proximity to each other, which suggests that they do not compete for growth flux in order to elongate. Apparently, the growth is determined by the rate at which growth species attach to the ends of the rods, rather than by the rate at which growth species diffuse to the ends of the rods from distant points on the substrate. Scanning tunneling microscope (STM) images and movies captured with LEEM during synthesis (desorption) show that the nano-rods can thicken (thin) perpendicular to the surface in atomic layer-by-layer fashion. We commonly observe that a rod's rate of elongation decreases sharply when it crosses a step to a lower atomic terrace and increases when it crosses a step to a higher terrace, showing that the number of atomic layers in the rod can change at step crossings. Diffraction analysis and dark-field imaging with LEEM indicate that these structures are crystalline, likely to be alumina (Al<sub>2</sub>O<sub>3</sub>) or spinel (NiAl<sub>2</sub>O<sub>4</sub>), and have an epitaxial relationship to the substrate. We use this information to discuss the origins of these highly anisotropic structures. This work was supported by the Office

**3:45 PM KK7.7**

**Quasi-Periodic Nanopatterning of Heteroepitaxial Layers by Misfit Dislocations Demonstrated for the PbTe on PbSe (001) Case.** Gunther Springholz and Wiesauer Karin; Intitut fuer Halbleiterphysik, University of Linz, Linz, Austria.

Nanopatterning of surfaces using dislocation arrays has recently evolved as a novel tool for fabrication of ordered nanostructures. Apart from the method of twisted wafer bonding, dislocation formation can also be induced by strained-layer heteroepitaxial growth. Usually, however, in this case rather irregular dislocation networks are formed due to the limited dislocation kinetics. In the present work, it is shown that it is possible to produce highly periodic dislocation arrays in strain-relaxed layers if several conditions are fulfilled. These are (i) the presence of a homogenous dislocation nucleation mechanism, (ii) a high lateral dislocation mobility within the interface, and (iii) the presence of a strong repulsive dislocation-dislocation interaction. These conditions are usually met only in heteroepitaxial systems with large lattice-mismatch and with misfit dislocations that have a Burgers vector confined to the interface plane. As an experimental example, we have studied the case of PbTe heteroepitaxial layers grown by molecular beam epitaxy on PbSe (001) substrates. From in situ scanning tunneling studies of the epilayer surface structure as a function of layer thickness it is shown that all three prerequisites are met in this system, and that therefore, nearly perfect square arrays of misfit dislocations are formed. The action of strong repulsive dislocation interactions as driving force for the underlying ordering process is also revealed by the fact that the observed course of strain relaxation can only be explained by theoretical models in which mutual dislocation interactions are taken into account. Due to the effect of the dislocation stress fields it is expected that the periodic dislocation arrays can be used as templates for deposition of ordered self-assembled nanoislands.

**4:00 PM KK7.8**

**Growth of Nanopillar Arrays using Glancing Angle Deposition.** Sai V. Kesapragada, Ken D. Girardin and Daniel Gall; Department of Materials Science and Engineering, Rensselaer Polytechnic Institute, Troy, New York.

Periodic arrays of Si, Ta, Cr, and CrN nanopillars, 0.1-2.0 $\mu$ m tall and 50-250 nm wide, were grown by ultra-high-vacuum glancing angle sputter deposition, in pure Ar or N<sub>2</sub> discharges at 1-3 mTorr. The height and width of the pillars decreases with increasing the azimuthal angle between the incident flux and the surface normal, which was varied from 66-88°. The nanopillar spacing, 100-500 nm, is controlled by initial surface patterning. Si (100) substrates are patterned by dispersing monolayers of 500-nm-diameter polystyrene or 160-nm-diameter silica colloid spheres to form a periodic close-packed array. E-beam lithography was utilized for patterning down to 50 nm feature sizes. Nanopillars grow perpendicular to the substrate surface when the substrate is continuously rotated about the polar axis during deposition. In contrast, a stationary substrate yields columns that are tilted towards the deposition flux.

**4:15 PM KK7.9**

**Ripple formations in sputtering and depositions by energetic cluster beam irradiations.** Noriaki Toyoda and Isao Yamada; Laboratory of Advanced Science and Technology for Industry, University of Hyogo, Kamigori, Hyogo, Japan.

Formation of ripples in sputtering and deposition process by energetic gas cluster ion beam irradiations are studied using large cluster ions with several thousands of atoms in one cluster ion. The gas cluster ions realize high-density energy depositions near a target surface, which originates non-linear collisions and completely different collision processes from atomic or molecular ion bombardments. Due to this dense energy deposition on surface, near-surface atoms are easily ejected. The most interesting characteristic of gas cluster ion irradiations is that gas cluster ions create a lot of laterally sputtered particles, which are called eLateral sputtering. These laterally sputtered particles showed significant influences on ripple formations at oblique incidence of gas cluster ion beams. A plenty of ripples were formed around incident angle of 60° but the surface was quite smooth at near normal or glancing angle irradiations. The ripple formations by gas cluster ion irradiations were not only observed at sputtering process but also appeared during the gas cluster ion beam assisted depositions. In this study, the ripple formations during sputtering and ion beam assisted deposition processes are studied and formation mechanism will be discussed.

**4:30 PM KK7.10**

**Large-Scale Epitaxial Growth of Hexagonal Arranged ZnO Nanowires Using Au Nanohole Membranes as Templates.**

Hong Jin Fan<sup>1</sup>, Woo Lee<sup>1</sup>, Kornelius Nielsch<sup>1</sup>, Margit Zacharias<sup>1</sup>, Armin Dadgar<sup>2</sup> and Alois Krost<sup>2</sup>; <sup>1</sup>Max Planck Institute of Microstructure Physics, Halle, Germany; <sup>2</sup>Institute of Experimental Physics, Otto-von-Guericke-University, Magdeburg, Germany.

Fabrication of single-crystalline one-dimensional semiconductor nanostructures with controlled orientation and spatial position is of great importance for their technological applications. We report here the successful large-scale growth of hexagonal arranged ZnO nanowire arrays on sapphire and GaN/Si substrates by combining substrate nanopatterning and the vapor-liquid-solid (VLS) growth process. In a first step, gold membranes of hexagonal-packed nanotubes with a low aspect ratio were obtained from an electrochemical duplication process of perfect ordered alumina membranes prepared by imprint-lithography and anodic oxidation. Then, these metal membranes were used as shadow masks to deposit ordered Au nanodot arrays on the substrates. Subsequent growth of ZnO via a VLS epitaxial process results in vertically-aligned and hexagonal-arranged ZnO nanowires with (0001) orientation. Here, the Au nanodots arrays serve as both catalyst for nanowire growth and template for specifying the position of individual nanowires. As the size (30 to 250 nm) and separation of 500 nm of the Au nanodots are tunable by choosing suitable deposition masks for the Au deposition, the resulting ZnO nanowires are adjustable in their diameter and inter-wire distance. Such nanowire arrays could have applications as photonic crystal, electron field emitters or nanosensor arrays.

**4:45 PM KK7.11**

**Leakage Currents through Thin Silicon Oxide Grown on Atomically Flat Silicon Surfaces.** Valerian Ignatescu and Jack M. Blakely; Materials Science and Engineering, Cornell University, Ithaca, New York.

We report some results that indicate that silicon oxide grown on step-free surfaces has better dielectric properties than that on normal, stepped surfaces. Advanced microprocessors now feature gate oxides that are 1.2-1.3 nm in thickness. For such thin silicon oxides the leakage current is high enough to substantially increase the power consumption of the transistors and therefore the heat generated by them. The high power dissipation is already a serious problem for current high frequency microprocessors. The most obvious path is to switch to a high-k dielectric material but other insulators, such as hafnium and zirconium oxides, even though they have good dielectric properties, are not compatible with the polysilicon material used for gate electrodes. The use of atomically smooth Si surfaces can potentially reduce leakage currents through silicon oxide layers. An increased roughness of the silicon/silicon oxide interface leads to a reduced local effective oxide thickness and, in consequence, to a higher leakage current [1]. Atomically flat surfaces were obtained by heating patterned silicon samples in ultra-high vacuum following the procedure described elsewhere [2]. After that, we grew a thin oxide layer with several different thicknesses (between 3 and 6 nm) using dry thermal oxidation. The AFM analysis of the surface after the oxidation shows that the topography of the surface is preserved during the oxidation process (even though the roughness of oxide surface is higher than the silicon surface roughness before oxidation). A 400 nm aluminum thin film was deposited on the entire sample surface and a second level of lithography was used to generate arrays of 18x18  $\mu$ m MOS capacitors on top of the stepped and step-free regions. We find that capacitors built on step-free regions are able to withstand a significantly higher voltage before breakdown compared to those on normal stepped regions. Also the absolute value of the slope of the Fowler-Nordheim plot, which is proportional to the effective thickness of the oxide, is higher for the capacitors situated on step-free surfaces. As expected, the difference between the electrical properties of the oxide grown on stepped and atomically flat surfaces is bigger for thinner oxide layers. References: [1] A. C. Oliver and J. M. Blakely, Mat. Res. Symp. Proc. 743, V 4.6.1 (2003) [2] D. Lee and J. Blakely, Surface Science 445, 32 (2000)

SESSION KK8: Thin Film Patterning  
Chairs: Robert Averbach and Michael Nastasi  
Thursday Morning, December 2, 2004  
Room 306 (Hynes)

**8:00 AM \*KK8.0**

**Synthesis of One Dimensional Materials: Interfacially Driven Chemistry Approach.** Sheng Dai, Chemical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Two central challenges in nanoscale science and technology are, first, to develop efficient methods for synthesizing high quality nanomaterials with well-controlled properties, and, second, to direct their assembly from the nanoscale upward into functional configurations. Control of nanomaterials properties generally requires

synthesis of only a narrow size distribution, while functional assembly usually implies controlling the location and/or the orientation of the nanomaterials. It must be emphasized that the directed assembly requirement cannot be met at this time by even the highest resolution electron beam lithography, for both technical and economic reasons. Consequently, successful syntheses often will require spatially confining and self-assembly growth of nanoscale structures. This presentation will be focused on chemistry approach toward the efficient synthesis and replication of one-dimensional materials. The various growth methodologies developed at our research group for synthesizing such key building block in nanotechnology will be discussed.

#### 8:30 AM **KK8.1**

##### **The effects of low-energy ion-irradiation on surface pattern formation in sputter-deposited epitaxial TiN(001) layers.**

TaeYoon Lee, Kenji Ohmori, Suneel Kodambaka, Joe Greene and Ivan Petrov; Materials Science and the Fredrick Seitz Materials Research Laboratory, University of Illinois at Urbana-Champaign, Urbana, Illinois.

We have chosen TiN(001) as a model system for investigating the effects of low-energy ion-irradiation on surface morphological evolution during sputter-deposition of epitaxial transition-metal nitride layers. As a first step, we used focused ion beam milling technique followed by high-temperature (1400 °C) annealing in air to produce  $\approx 5 \mu\text{m}$ -wide, atomically-flat, step-free terraces on MgO(001) substrates. Epitaxial TiN(001) films, 3000-Å-thick, were then grown on these patterned substrates at low-homologous growth temperatures ( $0.25 < T_s/T_m < 0.37$ ;  $T_s = 600 - 1050 \text{ }^\circ\text{C}$ ) using ultrahigh-vacuum reactive magnetron sputter deposition in pure  $\text{N}_2$ . The  $\text{N}_2^+$  to Ti metal ratio ( $J_i/J_{Ti}$ ) incident at the growing film was varied from 1 to 14 while the ion energy was maintained constant at  $\approx 20 \text{ eV}$ . Using atomic force microscopy, we find that all TiN(001) layers grown at  $T_s < 925 \text{ }^\circ\text{C}$  exhibit square-shaped growth mounds whose widths increase exponentially with  $T_s$  irrespective of  $J_i/J_{Ti}$ . However, layers grown with high flux ( $J_i/J_{Ti} = 14$ ) consist of mounds that are approximately two times larger than those obtained at  $J_i/J_{Ti} = 1$ , suggesting higher surface adatom diffusion under high-flux conditions. In both cases, we observe fractal island shapes indicative of relatively low edge-atom mobilities. TiN(001) layers grown with high flux at  $T_s > 925 \text{ }^\circ\text{C}$  are nearly atomically-flat with only the top 3-4 atomic layers exposed. This is in dramatic contrast to the films deposited at the same temperature under the low-flux conditions, where over 10-20 atomic layer height mounds are observed. Based upon temperature-dependent measurements of the widths of growth mounds and island shapes, we propose that low-energy ion-irradiation preferentially enhances surface-adatom diffusion with little effect on edge-atom mobilities over this growth temperature range.

#### 8:45 AM **KK8.2**

##### **Self-Organization of Curved Ripples on Titanium Crystals Irradiated with Focused Ion Beam.**

Wei Zhou<sup>1</sup>, Haixia Qian<sup>1</sup>, Yongqi Fu<sup>1</sup> and Bryan Kok Ann Ngoi<sup>1</sup>; <sup>1</sup>School of Mechanical & Production Engineering, Nanyang Technological University, Singapore, Singapore; <sup>2</sup>NTU, Singapore, Singapore.

We report the formation and self-organization of nano- and micro-scale structures during sputtering of titanium crystal surface at room temperature with a rastered 1 nA, 30 keV Ga<sup>+</sup> Focused Ion Beam (FIB). We use in-situ real-time secondary ion and secondary electron imaging to follow the evolution of surface features from a low dose of  $7.5 \times 10^{16}$  ions/cm<sup>2</sup> to a high dose of  $4.8 \times 10^{18}$  ions/cm<sup>2</sup> and characterize the final surface morphology using AFM. In contrast to usual expectation that surface topology produced at normal ion beam incidence consists of hillocks or depressions, we observe periodic ripples with regular spacing. We provide evidence to demonstrate that the ripples form spontaneously rather than by direct writing of the FIB. Ripples on surface of a single crystal are oriented in the same direction, but the ripple orientation changes with the crystallographic orientation of the sputtered titanium crystal surface. We explain the observation using a model in which the ripple structure is the result of the balance between the erosion rate and the anisotropic diffusion of adatoms and vacancies on surface activated by the ion beam sputtering. When the ion beam is tilted by a small angle up to 30 degree, the ripple orientation is still determined by the crystallographic direction. However, when the FIB incident angle is increased to 45 or 60 degree, curved ripples occur at high ion doses. No existing models can explain the morphological evolution from the well developed straight ripples to the curved ones. We therefore attempt to present a new model, which attributes the formation of curved ripples to competition between the formation of ripples due to anisotropic surface diffusion and the formation of incident-angle dependent ripples of the Bradley-Harper type.

#### 9:00 AM **\*KK8.3**

##### **Microstructural and Surface Morphological Evolution at the**

**Atomic Scale during Growth of Transition-Metal Nitride Coatings: Self-organized Nanostructures.** Joe Greene, Chan-Soo Shin, Suneel Kodambaka, Kenji Ohmori and Ivan Petrov; Frederick Seitz Materials Research Laboratory, Department of Materials Science, University of Illinois, Urbana, Illinois.

Polycrystalline TiN and related transition-metal (TM) nitride thin films are typically deposited by reactive magnetron sputter deposition and employed as diffusion barriers in microelectronics as well as hard, wear-, and corrosion-resistant coatings in mechanical and optical applications. Since cubic TM nitrides are highly anisotropic, control of preferred orientation is essential. We use a combination of HR-XRD, TEM, HR-XTEM, AFM, and STM analyses to characterize micro- and nanostructures. Kinetic Monte Carlo modeling and analytic theory, accounting for anisotropic adatom diffusivities and Ehrlich barriers at descending step edges together with kinetic roughening and atomic shadowing processes, are used to develop an atomic scale understanding of microstructural and surface morphological evolution. Adatom transport and surface site energies required for the models are obtained from in-situ high-temperature STM and LEEM analyses. The overall results yield design rules which allow the synthesis of controlled microstructures with greatly enhanced physical properties. Taking these concepts a step further, and using spontaneous natural patterning processes found in nature, we show that self-organized nanostructures consisting of commensurate nanolamellae, nanocolumns, nanospheres, and nanopipes can be synthesized to further extend the range of achievable properties. All of these structures are a result of kinetic limitations and require low growth temperatures combined with low-energy (less than the lattice atom displacement potential), very high flux, ion irradiation during deposition. This is easily achievable using tunable magnetically-unbalanced magnetrons. Property enhancements include increases in oxidation resistance, diffusion barrier lifetime, and hardness by factors from 50% to orders of magnitude. The range of colors available for decorative coatings is greatly expanded and new applications such as biologically active optical probes and selective sieves for biomolecules become possible. Finally, we extend these ideas to low-dimensional nanostructures such as perfectly periodic 1D wires and 2D domes, arcs, pyramids, and teardrops.

#### 10:00 AM **\*KK8.4**

##### **Physical self-assembly and nano-patterning.** Toh-Ming Lu,

Physics Department, RPI, Troy, New York.

The morphology of thin film growth front formed by a physical vapor deposition technique is controlled by many factors including: surface diffusion, sticking coefficient, and shadowing. Instabilities of growth can occur if the shadowing is more dominant compared to other surface effects and can lead to many diverse physically self-assembled 3D quasi-periodic nanostructures. In this talk I will discuss the fundamental nucleation and growth mechanisms of isolated islands due to the shadowing effect in an oblique angle deposition configuration, and the time evolution of diverse 3D nanostructures including rods, beams, and spiral springs, and their microstructures that can be grown on these isolated islands. In addition, I will present novel deposition methods that can control the width and geometry of these 3D nanostructures on a templated substrate with periodic seeds through a variety of substrate rotation and tilt schemes. \*Work supported by NSF

#### 10:30 AM **KK8.5**

##### **Chemistry-Mediated 2D-3D Transition of In Thin Films.** H. L. Wei<sup>1</sup>, Hanchen Huang<sup>2</sup>, Chung Ho Woo<sup>1</sup>, X. X. Zhang<sup>3</sup> and L. G.

Zhou<sup>2</sup>; <sup>1</sup>Electronic and Information Engineering, Hong Kong Polytechnic University, Hong Kong, China; <sup>2</sup>Mechanical Engineering, Rensselaer Polytechnic Institute, Troy, New York; <sup>3</sup>Physics, Hong Kong University of Science and Technology, Hong Kong, China.

This talk reports a mechanism of chemistry-mediated 2D-3D transition during In thin film deposition, and the corresponding evolution of nanoscale islands. Using magnetron sputtering technique, we deposit In on Au substrate. Despite the fact that In wets on Au, In islands prevail over the uniform film soon after the deposition starts. The 2D-3D transition is found to be a result of the formation of Au<sub>3</sub>In on the Au substrate. The alloy formation leads to non-wetting of In, thereby the high mobility of In atoms and In clusters, and eventually well-separated In islands. The structures of In and Au are characterized by scanning electron microscopy, transmission electron microscopy, and electron diffraction.

#### 10:45 AM **KK8.6**

##### **Large Scale Engineered Nanostructured Surfaces by Reactive Ion Etching with Kinetically Self-Assembled Non-continuous Metal Film as Etching Mask.** Wei Wei, Mark Bachman and Guann-Pyng Li; UC,Irvine, Irvine, California.

The fabrication of nanostructured surfaces in different materials is of

broad interest in recent years not only because of fundamental materials science issues, but also because of a variety of potential applications that may be enabled. In electronic materials, nanostructured surfaces can lead to quantum electronics, field emission sources, efficient photonic transducers, and photoluminescence devices. In biomedical applications, nanostructured surfaces can change fluid flow, modify hydrophobicity, affect cellular adhesion and response, perform protein recognition, and initiate chemical reactions. In materials engineering, nanostructures can affect adhesion quality, change optical properties, and modify mechanical performance of thin films. Different techniques have been developed to obtain nanostructured surfaces in the past few years for different applications. These include soft lithography, ion irradiation, spray deposition of nanoparticles, e-beam lithography, anodization of thin aluminum or silicon films, and plasma deposition of metal nanoparticles. However, no single method has been shown to be ideal, particularly for patterning large surface areas. It is well understood that very thin metal films, prepared by conventional deposition techniques, such as electron gun deposition, thermal evaporation, and sputtering deposition, are always polycrystalline and non-continuous because of the crystal structure mismatch and wetting properties between the deposited materials and the substrate materials. In this study, we explored the possibility of using annealed non-continuous metal film enabled etching technique to produce large nanostructured surfaces. A monolayer non-continuous Ag film is deposited on silicon wafer with a thin layer of silicon dioxide using E-beam deposition, and then vacuum thermal annealing was applied on the deposited films, causing nano-scaled Ag particles to migrate and agglomerate into self-assembled islands of larger nanometer dimensions. We controlled the density and average size of the metal islands through thickness of the initial film and subsequent annealing rates. Reactive ion etching through the metal islands mask into the underneath silicon dioxide layer was performed following the annealing process. Nanostructured silicon dioxide surfaces with different features, such as nanopoles, nanocones, and nanopillars, were explored by controlling the etching process, i.e. isotropic etching or unisotropic etching, through varying etching conditions, such as RF power, chamber pressure, and gas ratio. Detailed study on the characterization of non-continuous Ag films was also carried out to verify the effects of the initial film thickness and the annealing rates on the subsequent Ag islands morphology, size, and interstitial voids size. Preliminary results regarding the effect of the nanostructures towards an increased contact angle of hydrophobic surfaces is also shown.

#### 11:00 AM KK8.7

##### **Photocatalysis of Hypercolumnar TiO<sub>2</sub> Thin Films.**

Motofumi Suzuki, Kazuaki Kishimoto, Wataru Maekita, Kaoru Nakajima and Kenji Kimura; Department of Engineering Physics and Mechanics, Kyoto University, Kyoto, Kyoto, Japan.

Recently, thin films with highly controlled columns such as helix and zigzag have been developed. Furthermore, integration of these morphologies has been achieved. These 'hypercolumnar' structures are engineered by dynamic oblique deposition (DOD), in which the deposition angle and/or the azimuthal deposition direction are varied dynamically during the deposition, in contrast to ordinary oblique columnar structures which are generated on the stationary substrate. Not only electric, magnetic and optical but also chemical, medical and biological applications of thin films with hypercolumnar structures are expected. In particular, high porosity and the large surface area of hypercolumnar structures seem suitable for the chemical applications. In this work, we report on the relationship between the hypercolumnar morphology and photocatalytic properties of obliquely deposited TiO<sub>2</sub> thin films. The hypercolumnar titanium oxide films were deposited on a glass substrate at room temperature in an electron beam (EB) evaporation system. In order to tailor the columnar shape such as zigzag, cylinder, and helix, the deposition angle  $\alpha$  measured from substrate normal and the azimuthal motion of the substrate were controlled independently. After the deposition, samples were annealed in air at 500 °C for 1 h. X-ray diffraction revealed that the annealed samples were composed of single phase of TiO<sub>2</sub> anatase, which is suitable for photocatalyst. Photocatalysis of the samples was evaluated by photobleaching of methylene blue (MB). Experimental results such as morphology and photocatalysis are compared with those obtained by our original thin film growth simulator VFiGS. The columnar formation by oblique deposition is quite efficient to enhance the photocatalytic activity. An optimum morphology is available at the deposition angle of about 70° and the shape of the columns such as zigzag, cylinder, and helix is less important for the reaction in the water solution. Thickness dependence of photocatalysis of the samples deposited at  $\alpha=72^\circ$ , indicates that the active thickness of the hypercolumnar thin films is about 200 nm and is limited by the absorption of UV light. In other words, the interior surfaces of hypercolumns works well as photocatalyst. These facts can be understood in terms of the effective surface area of obliquely deposited thin films. This work was supported by The 21st Century COE Program "Center of Excellence

for Research and Education on Complex Functional Mechanical Systems" and by The Mazda Foundation's Research Grant.

#### 11:15 AM KK8.8

##### **Suppression of Reaction between Si Substrate and Obliquely Deposited Fe Atoms.**

Shinji Jomori, Motofumi Suzuki, Kohei Kinoshita, Kaoru Nakajima and Kenji Kimura; Department of Engineering Physics and Mechanics, Kyoto University, Kyoto, Kyoto, Japan.

The growth of iron silicides on a Si substrate has long been studied in the last two decades both from scientific and technological points of view, since the iron silicides have diverse physical properties depending on their crystal structures and compositions. Unfortunately, crystallographic and morphological structures have not been successfully controlled. For example, it is known that Fe and Si are highly reactive with each other and form islands when Fe is deposited from the normal direction at high temperature. Some of those islands have facets and specific height. By using oblique deposition technique, where the self-shadowing effect is dominant, the higher species among the islands will be grown selectively. In addition, it will be interesting for the case of Fe and Si system that not only the self-shadowing but also the surface reaction and diffusion play important roles in the formation of nanopatterns. In this work, we have investigated the effect of the deposition angle on the reactive growth of Fe-Si on the Si substrate. Iron was deposited intermittently up to about 10 ML on a Si(001) substrate kept at 470 °C under the pressure less than  $1.0 \times 10^{-8}$  Torr. The deposition angle was set at 85° measured from surface normal. Between each deposition, the samples were analyzed by the high resolution Rutherford backscattering spectroscopy (HRBS). In the HRBS measurements, we measured random and  $\langle 111 \rangle$  channeling spectra in order to estimate the depth profile of the composition and the number of (unshadowed) Si atoms displaced from their own lattice positions. Remarkably, the number of displaced Si atoms was almost constant and independent of the amount of Fe deposition, although it increases with twice of the number of deposited Fe atoms when Fe is deposited from surface normal. In addition, we confirmed the existence of islands ( $\approx 10$  nm height), which were small in number, by atomic force microscope (AFM). This means that the reaction between Si and Fe is significantly suppressed and that the Fe rich nano-islands grew selectively. Therefore, by controlling the deposition geometry for the reactive systems such as Fe-Si, novel self-organization technique will be realized. This work was supported by The 21st Century COE Program "Center of Excellence for Research and Education on Complex Functional Mechanical Systems" and by The Mazda Foundation's Research Grant.

#### 11:30 AM KK8.9

##### **In situ TEM Study of Three-Dimensional Patterning of Voids in Electron Irradiated CaF<sub>2</sub>.**

Tianhua Ding<sup>1</sup>, Sha Zhu<sup>1</sup> and Lumin Wang<sup>1,2</sup>, <sup>1</sup>Department of Nuclear Engineering and Radiological Sciences, Univ. of Michigan, Ann Arbor, Michigan; <sup>2</sup>Department of Materials Science and Engineering, Univ. of Michigan, Ann Arbor, Michigan.

It has been known that defect superlattices may form in electron irradiated CaF<sub>2</sub>. However, the nature of the defects on the superlattice point (i.e., Ca colloids or voids) has not been certain and the patterning mechanism has not been fully understood. In this study, both natural and synthetic CaF<sub>2</sub> were irradiated under 200 keV electron beam with in situ TEM observation of the dynamic process of defect patterning. Through-focus bright field images from three zone axes (100), (110) and (111) demonstrated that the superlattice structure is simple cubic. Energy-filtered transmission electron microscopy (EFTEM), high-angle annular dark-field (HAADF) STEM imaging, as well as thickness mapping, were used to characterize the void nature of the superlattice formed under electron irradiation. The superlattice reached steady state after an electron dose of  $6 \times 10^{20} \text{ e}^-/\text{cm}^2$  with void radius about 5-6 nm. Videos recorded during the in-situ observation reveal the patterning process of the void superlattice. Coalescence was prevalent at the initial stages. Migration and preferential growth were dominant at the final stages of the superlattice formation. At a dose higher than  $1 \times 10^{21} \text{ e}^-/\text{cm}^2$  the superlattice structure was destroyed. These critical doses of void superlattice formation and deformation seem to be independent of dose rate. The kinetic rate equations can describe the initial growth of the voids, and the anisotropic diffusivity of the vacancies explains the superlattice formation.

#### 11:45 AM KK8.10

##### **Surface Nanopatterning via Dendritic Oxide Growth.**

Guangwen Zhou<sup>1</sup>, Liang Wang<sup>2</sup>, Judith C. Yang<sup>2</sup>, John E. Pearson<sup>1</sup>, Pete M. Baldo<sup>1</sup> and Jeffery A. Eastman<sup>1</sup>; <sup>1</sup>Materials Science Division, Argonne National Laboratory, Argonne, Illinois; <sup>2</sup>Materials Science and Engineering Department, University of Pittsburgh, Pittsburgh,



Pennsylvania.

Reaction-diffusion systems have attracted intensive attention to create a diversity of patterns due to the existence of the long-range effect of diffusion and purely local reaction interactions in these systems. In this work we present an oxidation reaction-diffusion strategy for forming oxide nanopatterns involving early-stage oxidation of metal alloys containing noble elements. Elements such as Au, Pt, Ag etc., do not form stable oxides under normal conditions, while reactive elements, such as Cu, Ni, Fe, etc., form stable oxides. Specifically, the oxidation of  $\text{Cu}_x\text{Au}_{1-x}$  (100) alloys was visualized by an in situ environmental ultra high vacuum (UHV) transmission electron microscope (TEM). The oxidation of the  $\text{Cu}_x\text{Au}_{1-x}$  alloy proceeds by an island growth mode, i.e., oxidation occurs at the island perimeters, followed by a transition to a dendritic growth mode, where the sharp corners of the island are the preferential directions for the dendrite development. The critical island size for the transition from a square shape to the dendritic growth and the branch size of the dendritic islands are determined experimentally. The growth of Cu oxide islands is accompanied by the rejection of Au atoms from the oxide, leading to a Au-rich zone in the alloy at the metal-oxide interface. Our in situ TEM observations indicate this gold-rich zone has a non-uniform distribution and the zone near the flat edges of the island is more Au-rich than the sharp corners, which causes preferential dendritic growth at the island corners. These experimental observations are consistent with a kinetics analysis based on the 2D Fick's diffusion equations.

SESSION KK9: Laser Patterning of Surfaces  
Chair: Kalman Varga  
Thursday Afternoon, December 2, 2004  
Room 306 (Hynes)

**1:30 PM KK9.1**  
**Directed Assembly of Thin Film Nanostructures Under Laser Induced Rapid Spatio-Temporal Surface Modulations.** Chi Zhang<sup>1</sup>, Wei Zhang<sup>1</sup>, Adam Bauer<sup>1</sup> and Ramki Kalyanaraman<sup>1,2</sup>;

<sup>1</sup>Physics, Washington University in St. Louis, St. Louis, Missouri;  
<sup>2</sup>Center for Materials Innovation, Washington University, St. Louis, Missouri.

Thin film nucleation and growth is a self-assembly process where the initial stages naturally consist of nanometer sized clusters. Typically, on defect-free isotropic surfaces, these nanoclusters are a result of random walk surface diffusion and binding. The coupled time and length scale in nucleation, as determined by nucleation rate and saturated cluster density, are determined primarily by growth rate and substrate temperatures. So, appropriate surface temperature modulations should induce morphology changes. In recently published work, we presented novel studies showing that the application of a rapid spatio-temporal surface modulation in-situ with film deposition directs the assembly of periodic structures. Evidence shows that the assembly is a result of anisotropic diffusion flux, reevaporation and locally modified nucleation. The experiment consists of physical vapor deposition simultaneous with pulsed laser interference irradiation of a Si(100) surface. Deposition of Co, Ag or  $\text{TiO}_x$  results in periodic arrays of dot or line-like features whose periodicity and spacing resemble the interference patterns. The size scale of the resulting dot or line-like features can be varied from the nanometer to micron scale by deposition parameters. The rapid spatio-temporal modulation comes from the fringe spacing (200 to 2000 nm) and short laser pulse width (9 ns). In this work, we present fundamental experimental studies of the influence of these rapid surface modulations on film nucleation that leads to directed assembly of nanostructures. This experimental approach offers a simple way to assembly large-area arrays in a parallel process and is likely to lead to a cost effective manufacturing process.

**1:45 PM KK9.2**  
**Comparison of Morphology Evolution of Ge(001) Homoepitaxial Film Grown by Pulsed Laser Deposition and Molecular Beam Epitaxy.** Byungha Shin<sup>1</sup>, John P. Leonard<sup>2</sup>,

James W. McCamy<sup>1</sup> and Michael J. Aziz<sup>1</sup>; <sup>1</sup>Division of Engineering and Applied Science, Harvard University, Cambridge, Massachusetts; <sup>2</sup>Materials Science and Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania.

Using a dual MBE-PLD UHV chamber, we have conducted the first experiments under identical thermal, background, and surface preparation conditions to compare homoepitaxial growth morphology in Pulsed Laser Deposition (PLD) and Molecular Beam Epitaxy (MBE). We have studied germanium (001) homoepitaxy as a model system to compare these two processes. Surface morphology is characterized by in-situ Reflection High Energy Electron Diffraction and post-growth Atomic Force Microscopy. Our results reveal that in both cases film morphology evolves in a similar fashion: initially

round-based mounds form, followed by pyramid-like mounds with edges of the square base along  $\langle 100 \rangle$  directions, with a film roughness increasing with film thickness. The areal mound density is higher for PLD films than for MBE films grown at the same average growth rate and temperature. Furthermore, the dependence upon film thickness of roughness and mound separation differs for PLD and MBE. We discuss these results in terms of the similarities and differences between kinetic mechanisms expected to be operating in PLD and MBE.

**2:00 PM KK9.3**  
**Metastable Nanostructures Synthesis Upon Pulsed-Laser Induced Liquid-Solid Interface Reaction.** Qiu-Xiang Liu, Cheng-Xin Wang, Yu-Hua Yang and Guo-Wei Yang; State key laboratory of optoelectronic materials and technologies, School of Physics Science & Engineering, Zhongshan University, Guangzhou 510275, P. R. China, Guangzhou, China.

Laser ablation in high-temperature environment has been demonstrated to be a general route to synthesize elemental semiconductor and binary oxides nanowires such as Si, Ge,  $\text{SiO}_2$ , GeO, ZnO, etc. However, laser ablation to fabricate nanostructures is usually conducted in vacuum or in dilute gases. Very interestingly, laser ablation of solid target immersed in liquid shows the behavior different from that in vacuum or in gases. The ablation species are highly confined in a small region in the vicinity of the solid-liquid interface due to the inertia of liquid. The irradiation configuration gives instantaneous high density of energetic species, high pressure and high temperature. The formation of clusters in metastable phase is expected in this region, if this region is in a proper thermodynamic state. Very recently, we developed a novel laser ablation to synthesize metastable nanostructures, which was named to be pulsed-laser induced liquid-solid interfacial reaction (PLIIR). By used PLIIR, a series of nanocrystals including diamond with cubic and hexagonal structure, cubic  $\text{C}_3\text{N}_4$ , and boron nitride with cubic and explosion phase, and one-dimensional nanostructures such as Ag-Ni nanorods were prepared. Two points can be abstracted from PLIIR investigations, one point is that PLIIR is likely to synthesize metastable phase, and another one is that new phase formation involves in both liquid and solid. In this study, we report that an abnormal nanodendrites of silver and silver oxides are grown upon pulsed-laser induced reaction at the interface between the solid target and silver nitrate solution. To our best knowledge, few studies involved in nanodendrite formation of silver and its oxides have been reported in the published literature yet. By employed high-resolution transmission electronic microscope (HRTEM) equipped with energy depressive x-ray spectrometer (EDS) and selected area diffraction (SAD), the fabricated nanopatterns are identified to be the composite structure that consists of silver nanoparticles and silver oxides nano-plumes with polycrystalline structure. In detail, these silver nanocrystals are trunks of the nanodendrite, and their size is in the range of 30 to 50 nm. The silver oxides nano-plumes are branches of the nanodendrite, and their wide and length are in the range of 20 to 50 nm and 30 to 100 nm, respectively. Eventually, a vapor-liquid mechanism of the formation of the nanopatterns upon pulsed-laser induced liquid-solid interface reaction is proposed, and both silver clusters in vapor and silver ions in solution are simultaneously involved.

**2:15 PM KK9.4**  
**Self-Organized Formation of Patterns on Tin Irradiated with Femtosecond Laser.** Wei Zhou<sup>1</sup>, Hongyu Zheng<sup>2</sup> and Haixia Qian<sup>1</sup>;  
<sup>1</sup>School of Mechanical & Production Engineering, Nanyang Technological University, Singapore, Singapore; <sup>2</sup>Singapore Institute of Manufacturing Technology, Singapore, Singapore.

We used a Ti:Sapphire femtosecond laser (775 nm wavelength, 150 fs pulse duration, 1000 kHz repetition rate, intensity of above  $10^{12}$  W/cm<sup>2</sup>) to irradiate a polished surface of polycrystalline tin in an ambient environment and observed a well-developed surface modulation with a period of 1.4-2.8 micron. We further observed that the ripple orientation was influenced by the crystallographic orientation of the tin surface, in contrast to usual observation that the ripples are perpendicular to the beam polarization. The classical model on laser-induced rippling predicts the dependence of the ripple period on the laser wavelength. In contrast, our experiments demonstrate that the ripple period increases logarithmically with fluence for the fixed laser wavelength used. The evolution of ripples indicates that the surface periodic patterns are not formed due to direct writing in the ablation process and they are likely to be due to some spontaneous relaxation process on the highly non-equilibrium surface after the ions generated in the multiphoton ionization process left.

**2:30 PM KK9.5**  
**Laser Ablation of Carbon Films Used for Transfer of Electronic Devices.** Toshiyuki Sameshima and Nobuyuki Andoh;

Electrical engineering, Tokyo A&T University, Koganei, Tokyo, Japan.

Pulsed laser-induced ablation of diamond-like carbon (DLC) films was applied to transfer thin film structures to foreign substrates. Because DLC films has a hard and flat surface, thin film layered structure such as thin film transistor circuits can be formed on the DLC films. DLC films also have high optical absorption coefficients  $10^5 \sim 10^6 \text{ cm}^{-1}$  from visible to ultraviolet. Moreover there is a high stress between DLC films and substrates. These properties mean that DLC is a suitable material for laser ablation and transfer of thin film structures to foreign substrates. In the experiment, DLC films with a 200-nm thickness were formed on quartz glass substrates at room temperature by a sputtering method. 100-nm-thick aluminum layers were subsequently formed on the DLC films by a thermal evaporation method. The aluminum surface was coated with a paste. The samples were then pasted to plastic substrates. 30-ns-pulsed XeCl excimer laser was irradiated to DLC layers through the quartz substrates at room temperature in air atmosphere. When the laser energy density was  $400 \text{ mJ/cm}^2$ , the DLC layers were completely ablated and removed from the quartz substrates. No carbon remained on the substrates. Aluminum layers were also removed from the quartz substrates with no damage. The aluminum layers were successfully transferred to the plastic substrates. Dynamics of laser ablation of DLC films is discussed using transient conductance and transient reflectivity measurements. We will also demonstrate transfer of electronic device such as thin film circuits to foreign substrates using the present method.