Manufacturing Science and Technology Group
Room 611 - Session MS-WeA

Metrology II

Moderator: A.C. Diebold, Sematech

2:00pm **MS-WeA1 Micrometrology with Scanning Probes**, ***H.K. Wickramasinghe,*** IBM T.J. Watson Research Center **INVITED**

Scanning Probe Microscopes (SPM) have become valuable instruments for development and quality control in the semiconductor industry. They provide new capabilities for inspection and metrology of surfaces on a sub-micron scale. The key to their operation is the positioning and scanning of a small tip, or probe, at a minute distance over the surface. The ability to track the position of a surface with an accuracy of the order of a nanometer is advancing the frontiers of micro- or nano-metrology in the semiconductor industry. The standard Atomic Force Microscopes (AFM), using a conical shaped tip, is the most widely used type of SPM for inspection and metrology. This technology essentially measures depth of structures with high precision. Recent technical developments have added the capability to accurately measure width of lines and trenches, using a flared tip and an improved scanning and tracking method for the tip. Other techniques have evolved that measure magnetic properties on the nanometer scale. The talk will review the recent developments of scanning probes with special focus on applications to manufacturing.

2:40pm **MS-WeA3 Chemical Process Sensing using Mass Spectrometry in Multicomponent Reaction Systems**, ***Y. Xu,*** *T. Gougousi, N. Gupta, J.N. Kidder, Jr., G.W. Rubloff,* University of Maryland

A significant number of CVD applications in VLSI manufacturing, such as CVD of W, TiN, SiO@sub 2@, Cu, and more complex high K materials, involve multicomponent reactant mixtures. We have investigated experimentally W CVD processes from H@sub 2@/WF@sub 6@ mixtures in attempts to develop mass spectrometry based thickness and rate metrology approaches. This direction poses a variety of challenges, both fundamental and practical. First, the choice of reactant stoichiometry is dictated by the mechanistics of the application, and typically an overabundance of one reactant is required, so that transport of the other is at least in part rate-limiting. This determines which species is suitable for measurement of reactant depletion for deposition metrology. For example, in SiO@sub 2@ CVD from SiH@sub 4@/N@sub 2@O, the N@sub 2@O must be in large excess to assure fully oxidized, high quality material, while in W CVD, low H@sub 2@ to WF@sub 6@ ratio (usually 4) is required to achieve good conformality in via filling. Second, gaseous species may undergo substantial wall reactions, which in turn depend on previous process history (e.g., in W CVD, HF and WF@sub 6@ condense on walls and subsequently desorb slowly). Third, the mass spectrometer ionizer provides a second reaction region (in addition to the wafer), contributing to the depletion of the reactants and occasionally generating the same products as the CVD reaction itself (HF in W CVD from H@sub 2@/WF@sub 6@ precursors). Fourth, the combination of wall adsorption/desorption and sensor reactions can lead to another extraneous source of products. In the H@sub 2@/WF@sub 6@ system sustained H@sub 2@ flow after the WF@sub 6@ flow is terminated leads to sensor generated HF utilizing the WF@sub 6@ desorbing from the walls. We illustrate these phenomena through both experimental and modeling results, and we assess key aspects of the general approach for multicomponent CVD systems.

3:40pm **MS-WeA6 Mechanisms for the Production of Atomically Flat Surfaces Studied by Scanning Probe Microscopy@footnote 1@**, ***S.C. Langford,*** *R.F. Hariadi, J.T. Dickinson,* Washington State University

Chemical-mechanical polishing (CMP) is a critical step in the fabrication of integrated circuits, yet the complex interactions between chemical and mechanical effects in CMP are still not well understood. We examine layer by layer material removal and deposition under conditions of combined mechanical stress and chemistry using scanning probe microscopy (SPM), where the SPM tip serves as a model single asperity/abrasion particle. We focus on monolayer-deep etch pits on a model, biomaterial substrate, single crystal brushite (CaHPO@sub 4@@super .@2H@sub 2@O). Scanning across monolayer steps in undersaturated solutions at high contact forces produces distinct wear tracks due to localized double kink nucleation. Low contact force scanning in supersaturated solution produces localized deposition along steps in the scanned region. The latter suggests a novel method of producing atomically flat surfaces by mechanically controlled re-crystallization. These results allow quantitative models of wear and deposition to be developed and tested. @FootnoteText@ @footnote 1@This work supported in part by the NSF Surface Engineering and Tribology Program under Grant CMS-98-00230.

4:00pm **MS-WeA7 Investigation of Thermal Curing of an Organic Low-k Spin-on Dielectric by Variable-Angle Spectroscopic Ellipsometry**, ***F. Yang,*** *W.A. McGahan,* Nanometrics, Inc.*; C.E. Mohler, L.M. Booms,* The Dow Chemical Company

As device features of ultra-large-scale-integrated (ULSI) circuits continue to shrink, a new type of dielectric material with a low dielectric constant k is needed for replacement of SiO2 as the insulating material between multi-level interconnects. Dow Chemical's SiLK* semiconductor dielectric@footnote 1@ has been investigated as a potential candidate for the low-k dielectric. SiLK dielectric thin films are formed by spin-on process, followed by a thermal curing process, which determines the mechanical, electrical, and chemical properties. A properly cured SiLK dielectric thin film offers a dielectric constant of 2.65, along with merits of high thermal stability, excellent gap-fill properties, high solvent resistance, and low moisture absorption. In this paper, optical properties of SiLK dielectric thin films cured at different conditions are characterized using variable-angle-spectroscopic ellipsometry. Correlation is found between the optical constants in the ultra-violet wavelength region, and the extent of the cure (cure time and cure temperature). Based on the relationship between optical constants of SiLK dielectric and its curing condition, a single-parameter empirical interpolation model is developed to describe the dispersion of SiLK dielectric's optical constants. Despite a single adjustable parameter, this interpolation model closely tracks the variation of SiLK dielectric's optical constants at different curing conditions. With this model, in-line monitoring the cure of SiLK dielectric thin films can be realized. @FootnoteText@ @footnote 1@ SiLK* is a trademark of The Dow Chemical Company.

4:20pm **MS-WeA8 Assessment of Quadrupole Mass Spectrometry as an In Situ HDP-CVD Process Diagnostic Technique**, ***J.A.B. Van Hoeymissen,*** IMEC, Belgium*; C. Hughes,* BOC Edwards industrial resident at IMEC, Belgium*; M. Heyns,* IMEC, Belgium

Process control using in situ techniques is an attractive aid to semiconductor manufacturing. The potential of quadrupole mass spectrometry (QMS) has been assessed as an in situ sensing technique for a silicon oxide high density plasma chemical vapour deposition (HDP-CVD) process. In this paper it is shown that in situ measurements using mass spectrometry can play an important role in the areas of process monitoring, process control and process recipe optimization. The species present in the chamber were analysed via a gas sampling system, with pressure reducing orifice, installed just below the deposition chamber to ensure a representative, real-time, sensitive measurement. Correlation between in situ observations and oxide layer thickness was investigated. H@sub 2@O is an important reaction by-product of the deposition reaction. A direct and highly sensitive correlation between the H@sub 2@O+ signal and oxide layer thickness could be observed. In fact, this QMS signal could be used to monitor oxide layer thickness during deposition. In addition, the [H@sub 2@O+] was found to increase exponentially during deposition. During consecutive depositions of P-doped oxide (PSG) layers, the thickness of the first layer was systematically about 2% higher than the subsequent layers. In situ analysis was carried out monitoring the time resolved intensity of mass 34 (PH@sub 3@+) during the first three PSG depositions. This intensity was clearly higher during the initial phase of the first PSG deposition. The composition of the layers were analysed with SIMS. The results indicate the phosphorus content of the first layer is significantly higher during the initial phase of the deposition. These combined results indicate a higher initial [PH@sub 3@] in the deposition chamber during the deposition of the first PSG layer. These observations prompted an adaptation of the chamber conditioning and clean recipe preceding the first PSG deposition, resulting in the disappearance of the first wafer effect.

4:40pm **MS-WeA9 Determining Ion Flux and Ion Energy from Radio-Frequency Current and Voltage Measurements**, ***M.A. Sobolewski,*** National Institute of Standards and Technology

To obtain optimal results from plasma processing, the flux and energy of ions incident on the substrate must be carefully monitored and controlled. Several diagnostic techniques are used to measure ion flux and ion energy, but these techniques are typically not very compatible with the processes and reactors used by industry. Methods have been proposed for determining ion properties from the applied current and voltage waveforms, which can be measured in commercial reactors. However, such

methods are usually not very accurate because they rely on false or untested assumptions. Here, a new, more accurate method is presented which makes use of a complete model of the time-dependent ion dynamics in the plasma sheath. The model was validated by comparison to independent measurements of ion flux, ion energy, and time-resolved optical emission, for high-density discharges in an inductive GEC Reference Cell. Measurements were performed for discharges in Ar, Ar/SF@sub 6@, and Ar/Cl@sub 2@, for inductive source powers up to 370 W, rf bias powers up to 100 W, rf bias frequencies of 0.1-13.56 MHz, and pressures of 0.67-4.0 Pa. An analysis of the sensitivity of ion flux and ion energy results to model parameters will be presented, along with comparisons showing the improvement in accuracy obtained by the new technique.

5:00pm **MS-WeA10 Linking Process and Structure using Automated Analysis of AFM Images**, ***D.A. Chernoff***, *D.L. Burkhead, C.S. Cook,* Advanced Surface Microscopy, Inc.

By volume of product, the optical disc industry is the largest nanotechnology activity today. On DVDs (Digital Versatile Discs), the smallest features are about 400 nm long, 320 nm wide, 120 nm high, with a track pitch of 740 nm. Consumers need optical discs whose electrical performance during playback is consistently within specifications. Existing disc analyzers report electrical test results and engineers respond to deviations by adjusting process variables. This method provides only indirect control because the process variables determine the microstructure of the master, stamper and replica and it is that microstructure which determines ultimate electrical performance. A method is needed to examine microstructure so that one can see how each process variable affects various aspects of microstructure and to see how each aspect of microstructure affects performance. Automated, high accuracy analysis of Atomic Force Microscope (AFM) images provides the missing link. We measured the following parameters: track pitch, bump height, bump width and length (at various threshold levels), bump length, and four sidewall slope angles, in each case reporting mean, standard deviation and other statistics. From each 10 um image of a DVD stamper, containing about 100 bumps, we tabulated about 1000 values. Bump width increased with bump length, correlating with a corresponding increase in amplitude with pulse duration when a finished disc is played. Where sidewall angle deviated from the norm, we reviewed the image data to identify the specific nature of the defect. The results were statistically robust not only for mean values, but also for standard deviations, so that we could compare process variation from different pieces of equipment. Thus, feature geometry will no longer be a hidden variable in the path between controlling production equipment and observing the good or bad electrical performance of a finished disc.

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