Thursday Afternoon, October 5, 2000

Material Characterization
Room 207 - Session MC-ThA

Evolving Technologies in Surface Analysis

Moderator: E. Garfunkel, Rutgers University

2:00pm **MC-ThA1 In-situ Control of Wet Chemical Etching of Patterned Bulk-GaAs using Real Time Spectroscopic Ellipsometry***, **S.-J. Cho**, P.G. Snyder,* University of Nebraska, Lincoln*; C.M. Herzinger, B. Johs,* J. A. Woollam Co.

High controllability and reproducibility of the etching process are regarded as key factors for the success of device fabrication. In-situ optical techniques such as real time spectroscopic ellipsometry (RTSE) may be used to control etch depth as well as determine etch rates. We have previously demonstrated control of etch depth through the cap and AlGaAs layers of an unpatterned GaAs/AlGaAs/GaAs heterostructure. In this work, in-situ monitoring and control of etch depth in patterned bulk GaAs was studied using RTSE. Patterning is necessary for monitoring etching in the bulk substrate (unlike etching in thin layers), since without it optical interference would not occur. Patterned (lines or squares) pieces of bulk GaAs wafer were etched in a citric acid-hydrogen peroxide-deionized water etch solution. The grating periods were 10, 20, or 40 µm. During the etching RTSE data were taken and simultaneously analyzed, and etching was stopped when the fitted etch depth reached a predetermined value (for example, 1.6 µm). The final etch depth was confirmed by ex-situ spectroscopic ellipsometry and SEM analyses. The etch rate of bulk GaAs was also determined based on the real time fit results. The model currently used for RTSE data analysis assumes spatial coherence of the light beam over an area large compared with the grating period, but does not explicitly include diffraction effects. This model appears to work well for grating periods in the range 20~40 µm. These and the previous results show that RTSE can be used to control etch depth in both thin layers and the substrate. Research supported by AFOSR Grant #49620-96-1-0480.

2:20pm **MC-ThA2 In- and Ex-situ Characterization by Second-harmonic Generation of the RPECVD Oxidation and Nitridation of Silicon and Ex-situ Comparisons with Other Optical Techniques***, **G.D. Powell**, R.S. Johnson, G. Lucovsky, D.E. Aspnes,* North Carolina State University

We report azimuthal dependences of second-harmonic generation (SHG) spectra obtained in-situ and ex-situ from 800 to 900 nm of oxide, nitride, and oxynitride interfaces formed by Remote Plasma Enhanced Chemical Vapor Deposition (RPECVD) on singular and vicinal Si(111) substrates. Spectral dependences are linear over this limited spectral range that make it possible to compare slopes and intercepts among the different processes. SHG measurements were performed for both p-p and p-s polarization configurations. We found only slight variations for the terminations except for one parameter describing the electric field phase differences for the step for the nearest direction. We also observe significant differences, of the order of 10 meV, in ellipsometrically determined apparent critical point energies and broadening parameters under various processing conditions. Finally, we advance beyond a purely phenomenological model of a signal associated with the bond symmetries to a more physical model that directly connects the macroscopically measured far-field intensity to the dipole radiation from the anharmonic bonds.

2:40pm **MC-ThA3 What Is Needed and What Is Practically Available for Small Area, Small Depth, Chemistry Sensitive Analysis in the Semiconductor Wafer Processing Industry***, **C.R. Brundle** [1] ,* Applied Materials **INVITED**

The continuing health of the semiconductor industry depends on the insatiable demand to reduce CD (critical dimensions), eg gate length and thickness, interconnect line dimensions, barrier layer thickness. For needed analytical methods, this translates into improved spatial resolution (area and depth). In addition, because of (a) the drive towards where everything is interface, (b) the introduction of many "non-traditional" materials (eg Cu, low k, high k), and (c) a general push towards even higher yields, the need for chemical state information has increased and will continue to increase. In this paper I review, from a wafer processing perspective, what analytical instrumentation is needed, compared to what is actually available. I also try to define the differences in instrumentation needs for "analytical laboratory" usage and "metrology usage." Though I will concentrate on techniques which are thought of as surface sensitive (eg electron spectroscopies), there is no longer any practical reason to distinguish these from techniques which are not mainly thought of as surface sensitive (eg EDS, Raman). The reason is twofold. First, we need information over a range of depth. Second, such techniques can be, and have been, made more surface/interface/small area applicable than has been traditional. Finally, since our particular interests have to do with small particle defects during wafer processing, I will give examples to illustrate our current capabilities and our unmet needs.

3:20pm **MC-ThA5 Dopant-Induced Contrast of Si Devices in PEEM***, **V.W. Ballarotto**, K. Siegrist, R.J. Phaneuf, E.D. Williams,* University of Maryland

Spatial variation of doping, such as in a pn junction, produces contrast in photoelectron emission microscopy (PEEM) that may be useful for dopant profiling, failure analysis, or as a processing diagnostic of Si. The contrast mechanism arises from reduction of the effective photothreshold when Si is heavily to degenerately doped. We report on a quantitative investigation of doping-induced contrast in PEEM images of Si devices. The calibration samples consist of p-type (B) stripes of different dopant concentrations and line separations, written on n-type (N@sub d@=10@super 14@ cm@super -3@) Si(001) substrates. Using a near-threshold light source, we find that the signal intensity increases monotonically with B concentration over a range of doping from 1x10@super 18@ to 6x10@super 20@ cm@super -3@. The measured intensity ratios are in good agreement with a calculation based on photoemission from the valence band. In addition, PEEM images of memory chip devices acquired through a 2000 Å SiO@sub 2@ layer and showing clear doping contrast will be shown. This depth sensitivity and the potential for improved doping contrast will be discussed in terms of the incident photon energy above threshold. This work is supported by Advance Research Development Activity (ARDA) grant number: 97-C-3048.

3:40pm **MC-ThA6 Elemental Mapping of Sub-µm Particles and Structures by LASER-SNMS and TOF-SIMS***, **F. Kollmer**, R. Kamischke, R. Ostendorf,* Physikalisches Institut der Universitaet Muenster, Germany*; H. Bender,* Materials and Components Analysis Group IMEC*; A. Benninghoven,* Physikalisches Institut der Universitaet Muenster, Germany

Sputtering-based surface mass spectrometry as SIMS or Laser-SNMS combines high sensitivity with high lateral resolution, provided a high fraction of sputtered particles is ionized and a finely focused primary ion beam is applied. For sub-Âµm characterization the transformation probability of a surface atom into an ion should be optimized. The large fraction of sputtered neutrals and their efficient laser-postionization result in high Laser-SNMS sensitivities. The use of a time-of-flight (TOF) mass spectrometer guarantees parallel mass registration at high transmission. We report on recent results of TOF-SIMS and Laser-SNMS characterization of sub-µm particles. The analyzed particles include Al@sub 2@O@sub 3@, Fe@sub 2@O@sub 3@ and CeO@sub 2@ with diameters down to 15 nm. On the base of these results we compared both techniques one with the other and in addition with Scanning Auger Microscopy (SAM). The know how gained from these investigations was applied to supported metal catalysts and other sub-Âµm structures such as magnetic heads. By applying a combined TOF-SIMS/Laser-SNMS instrument, a direct comparison of both techniques was possible. The use of a 30 keV finely focused Ga@super +@ source and a time-of-flight instrument guarantees high lateral (@>=@80 nm) and mass resolution at high transmission. For nonresonant postionization an excimer-laser (193 and 248 nm) was applied. SAM characterization was carried out in a VG 350 F instrument. We determined useful yields, as a measure of sensitivity, for a variety of mono-elemental metal samples. The obtained useful yields in the order of 10@super -2@ (SNMS) and 10@super -3@ (SIMS) allow to analyze structures well below the spot size of the primary ion beam. We found higher elemental sensitivities for TOF-SIMS and especially Laser-SNMS compared to SAM. Quantification was possible by the use of SAM and Laser-SNMS, the latter additionally allows to estimate the total amount of material in a particle.

4:00pm **MC-ThA7 Dose Quantity Effects on Nano-scaled Dot Size and Depth Profile of Gallium Implantation on Silicon by Finely Focused Ion Beam***, **L. Shen**, L.C. Feldman, R.F. Haglund Jr., R.A. Weller,* Vanderbilt University

In a FFIB(Finely Focused Ion Beam) System, the beam current is controlled by choosing the aperture, which determines the spot size on nano-materials fabrication. The total beam dose is determined by beam current intensity, beam duration time and scan repeat rate. The total dose determines the ultimate feature size. We have investigated the total dose

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[1] **Featured Speaker - Science and Technology in the 21st Century**

effects on apparent spot size and depth profiles by controlling beam current intensity, beam dwell time and/or scan times. In particular we report the AFM apparent spot size for a single scan as a function of dwell time and compare to theoretical estimates. We also report apparent spot size as a function of number of scans. Achieving minimum spot size is critical for creating nano-scale feature size and depth profile.@footnote 1@ @FootnoteText@ @footnote 1@Supported in part by the National Science Foundation under grant DMR-9871234 and by the U.S. Army Research Office under contract DAAD-19-99-1-0283.

4:20pm **MC-ThA8 Ion Channeling Effects on the FIB Milling of Crystalline Materials***, **B.W. Kempshall**, S.M. Schwarz,* University of Central Florida*; B.I. Prenitzer,* Lucent Technologies*; L.A. Giannuzzi,* University of Central Florida*; R.B. Irwin, F.A. Stevie,* Lucent Technologies

The focused ion beam (FIB) instrument is being used as a fundamental tool for the investigation of ion beam/material interactions. One particular topic of interest is the effects of ion channeling on the sputtering behavior of Si and Cu. Although applications involving the FIB milling of Si are straightforward, the FIB milling of Cu may be complicated by effects such as the development of milling induced topographical features. The difference in milling behavior between Si and Cu may be attributed to the inherent physical properties of materials that influence the sputtering yield. As ions channel deep into the target along preferred crystallographic directions, a corresponding decrease in sputtering yield is observed. In this presentation, the effects and the mechanism of Ga+ channeling in Cu is used to illustrate the relationship between the sputtering yield, the quality of FIB mill cuts, and the surface characteristics of FIB milled Cu for various crystallographic orientations. The correlation between the theoretical Lindhard-Onderdelinden axial channeling model and the observed ion channeling contrast in the FIB milling of Cu is investigated.

4:40pm **MC-ThA9 XPS Analysis of Si Samples Prepared Using the FIB Lift-Out Technique***, **A.C. Ferryman**, J.E. Fulghum,* Kent State University*; L.A. Giannuzzi,* University of Central Florida*; F.A. Stevie,* Cirent Semiconductor

Si samples prepared using the FIB lift-out technique were analyzed using high spatial resolution XPS imaging and spectroscopy. Sample mounting and associated problems with sample location will be discussed. The analyzed samples were 5 x 20 micron cross-sections mounted on carbon-coated TEM grids. Results indicate that XPS data can be acquired from FIB lift-out samples. XPS provides new information about the effect of Ga surface residue on the Si surface.

5:00pm **MC-ThA10 Determining Area Selectivity in Small-Area XPS Analysis@footnote 1@***, **D.R. Baer**, M.H. Engelhard,* Pacific Northwest National Laboratory

For many real world applications, the ability of new XPS instrumentation to examine small features can be a tremendous advantage. Most users and vendors define the spatial resolution of these instruments in a similar relatively simple and useful fashion involving resolution of a sharply defined edge or grid. However, it can be useful and is sometimes important to know the area of a sample that actually contributes to the XPS spectra measured. We have found that measurements on well defined "dots" of a material on a substrate provide a useful experimental approach for determining the area of a specimen that contributes to a small area XPS measurement. The method provides a way to verify instrument operation and set up conditions and the adequacy of specimen alignment procedures for small area analysis. Data collected from a Phi Quantum 2000 and a Kratos Axis 165 demonstrate that the method can provide information about signal generated from regions outside of an intended analysis area. The method also demonstrates that commonly used methods of defining resolution produce values that can be 2 or 3 times smaller than the diameter of the area truly analyzed by a spectrometer. @FootnoteText@ @footnote 1@ This work was supported by the US Department of Energy, Office of Basic Energy Sciences, Geosciences Program. The measurements were made in the Environmental Molecular Sciences Laboratory, a DOE User facility operated for the USDOE by Battelle Memorial Institute.

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