Thursday Morning, November 3, 2005

Applied Surface Science
Room 206 - Session AS+TF-ThM

Thin Film Characterization

Moderator: P.M.A. Sherwood, Oklahoma State University

8:20am **AS+TF-ThM1 Characterization of Nanoscale Ceramic Gradient Coatings for Gas Analytical Microdevices**, ***M. Bruns***, *V. Trouillet, H. Mueller, E. Nold,* Forschungszentrum Karlsruhe GmbH, Germany*; R.G. White,* Thermo Electron Corporation, England

The key element of the Karlsruhe Micro Nose is a thumbnail sized gas-sensitive microarray which at present consists of 38 sensor elements on an area of 4x8mm@super 2@. It is based on a noble metal-doped SnO@sub 2@ layer, the electrical conductivity of which is highly sensitive to the composition of the ambient atmosphere and is measured between adjacent parallel platinum strip electrodes. In order to enable pattern recognition techniques these initially identical sensors have to be gradually differentiated with respect to their gas response. For this purpose gas-permeable membranes with thickness variation of approximately 2 to 10 nm were deposited across microarray using ion beam assisted deposition. In this work we focus on mixed membranes combining the gas permeability of silica and the chromatographic discriminating capability of alumina. Different geometries were achieved by shaping the ion beam profile to gradually alter the ion current density across the microarray leading to laterally different deposition rates. Various Al/Si ratios within the membranes were obtained using different substrate temperatures during deposition. In the present paper a comprehensive characterization of differently shaped nanoscale membranes is reported. Auger electron spectroscopy is used for evaluation of the geometrical integrity of the uncoated electrode pattern and for the determination of thickness profiles, respectively. Parallel angle resolved X-ray photoelectron spectroscopy provides thickness information for the membranes together with information on chemical binding states in a non-destructive manner. Ellipsometry is presented as a powerful quantification method for the determination of the desired ultra thin membrane thickness profiles. Moreover, after calibration with surface analytical data, ellipsometry allows for rapid evaluation of Al/Si concentrations ratios within the membranes.

8:40am **AS+TF-ThM2 Characterization of Low k Dielectrics Using Auger Microprobe Analysis**, ***C. Dziobkowski***, IBM Corporation, E. Fishkill*; E.D. Adams,* IBM Corporation, Essex Jct.*; J.A. Coffin, R.E. Davis, P.L. Flaitz,* IBM Corporation, Hopewell Jct.*; E.G. Liniger,* IBM Research, Yorktown Heights*; S.E. Molis, D.D. Restaino,* IBM Corporation, Hopewell Jct.

As the dimensions of integrated circuits are reduced, the capacitance between metal lines has an ever increasing impact on device performance. It increases circuit delay, results in parasitic capacitance creating crosstalk, degrades the signal to noise ratio and increases power consumption. Reduction of capacitance by employing low k dielectric materials is thought to be a solution. These low k materials have to be characterized as to their composition, uniformity, void formation and oxygen permeability requirements. This paper gives a description of the methodology developed using Auger depth profile analysis to characterize these new materials. Also important is how these Auger microprobe results can be compared to data obtained from TEM, TOF-SIMS and Rutherford backscattering analyses. The synergism of these analytical techniques is necessary to obtain the understanding needed for the integration of these low k dielectric materials with copper metallurgy in successful device fabrication.

9:00am **AS+TF-ThM3 Semiconductor-Dielectric Interfaces: Composition and Structure**, ***L.C. Feldman***, *S. Dhar,* Vanderbilt University*; J.R. Williams,* Auburn University*; L. Porter,* Carnegie- Mellon University*; J. Bentley,* Oak Ridge National Laboratory*; K.-C. Chang, Y. Cao,* Carnegie-Mellon University **INVITED**

The semiconductor-dielectric interface is the key to a successful MOSFET technology and has played the essential role in the silicon revolution. Wide-band gap materials have presented a challenge to achieve the same degree of interface perfection as silicon, although considerable progress is underway. The SiC/SiO2 interface is of particular scientific interest in this development because of its close relationship to silicon, both in processing and structure. The oxidation process in SiC yields a heavily defected SiC/SiO2 interface giving rise to poor device characteristics. Systematic use of chemical modification and processing, combined with a careful analysis of interfacial structure, results in significant progress in reducing defects and increasing inversion layer carrier density and mobility. For example nitridation of this interface results in a remarkable improvement and is a driving force for understanding the nitrogen profile and concentration. The quantitative nitrogen profile is critical to this understanding and provides a significant depth profiling challenge. Using a variety of probes including medium energy ion scattering, secondary ion mass spectroscopy, nuclear profiling and electron energy loss spectroscopy we show that the nitrogen is confined to within ~1.5 nm of the buried interface, with concentrations that are crystal face dependent and vary from 0.5 to ~1.5 x 10@super 15@/cm@super 2@. From an analysis point of view the significant new finding is the comparison of techniques and the degree of quantitative agreement between the different probes. @FootnoteText@ Supported by DARPA, N00014-02-1-0628 and ONR,N00014-01-1-0616. Research at the O. R. N. L. was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:40am **AS+TF-ThM5 Comparison of Silicon Oxynitride Produced by PIII/D and Reactive Sputtering**, ***N.D. Theodore***, *M. Bagge-Hansen, B.C. Holloway, D.M. Manos,* College of William and Mary*; C. Hernandez, T. Siggins, H.F. Dylla,* Jefferson Lab

High-purity, hydrogen-free silicon oxynitride films were successfully created using two techniques, plasma immersion ion implantation/deposition (PIII/D) and reactive sputtering. Our previous work has shown that coating 6" polished 304 stainless steel electrodes with silicon oxynitride, created by PIII/D, dramatically reduces field emission from 27 µA of at 15 MV/m to 160 pA at 30 MV/m. We have recently developed a new procedure to deposit silicon oxynitride without ion implantation using a low temperature (<200°C) Rf reactive sputtering process. Both procedures use a 750 W inductively-coupled nitrogen plasma that sputters silicon dioxide from a quartz dielectric window. The purpose of this study was to determine and compare the composition and electrical properties of the silicon oxynitride coatings created using the reactive sputtering and PIII/D procedures. The homogeneity, stoichiometry, and density of deposited/implanted layers were determined using Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared spectroscopy (FTIR), and Rutherford backscattering spectrometry (RBS). AES depth profiles determined that both procedures created homogeneous films, and FTIR and XPS spectra confirmed the creation of silicon oxynitrides with approximately 10% nitrogen. Ultraviolet photoelectron spectra and capacitance-voltage measurements will also be presented and discussed.

10:00am **AS+TF-ThM6 Microbridge Testing of Plasma Enhanced Chemical Vapor Deposited Silicon Oxide Films on Silicon Wafers**, ***Z. Cao***, Boston University*; T.-Y. Zhang,* Hong Kong University of Science and Technology*; X. Zhang,* Boston University

Plasma-enhanced chemical vapor deposited (PECVD) silane-based oxides (SiOx) have been widely used in both microelectronics and MEMS (MicroElectroMechanical Systems) to form electrical and/or mechanical components. During fabrication of such microelectronic and MEMS devices, PECVD SiOx undergo many thermal cycles, which often causes unwanted changes in thermal-mechanical properties of the material, and consequent degradation of device performance and reliability. In this paper, a novel nanoindentation-based microbridge testing method for thin films is proposed to measure both the residual stresses and Young's modulus of PECVD SiOx thin films. In this method, freestanding microbridges are fabricated from the thin films using the micromachining techniques. The tests are performed at the center of the microbridges with an instrumented nanoindentation system and the load-deflection curves are recorded. Our theoretical model used a closed formula of deflection vs. load, considering both substrate deformation and residual stress in the thin film. To simulate real thermal processing in device fabrication, some microbridges underwent various rapid thermal annealing (RTA) at temperatures up to 800ÂºC. An interferometric microscope was also used to measure the curvature profiles of the bridges. Together with nanoindentation test results on the microbridges, we were able to decide the changes in residual stresses and Youngâ?Ts modulus of the PECVD SiOx thin films under different thermal annealing. Two factors, density change and plastic deformation, were identified as controlling mechanisms of stress changes in the films. A microstructure based mechanism elucidates "seams" as source of density change and "voids" as source of plastic deformation, accompanied by viscous flow. This mechanism was applied to explain our experimental results of thermal annealing of PECVD SiOx films.

10:20am **AS+TF-ThM7 Characterization of Ultra Shallow Arsenic Implants by ARXPS, LEXES, MEIS, and Dynamic SIMS**, ***G. Conti***, *Y. Uritsky, H. Graoui, M. Foad,* Applied Materials*; C.R. Brundle,* Brundle & Associates*; D. Kouzminov,* Materials Analytical Services*; C. Hitzman,* Full Wafer Analysis*; P. Mack, J. Wolstenholme,* Thermo Electron Inc., UK

Ultra-shallow As implants are a leading-edge technology. Low voltage results in implant layers of tens of A thickness after anneal. Reliable metrology for shallow implants is needed. We characterize the implant layer as a function of nominal dose (1E14 to 2E15 ions/cm2) at 2KV, using a variety of techniques. Angle Resolved-XPS gives precise measurement of SiO2 oxide thickness, monitors the chemical state of As, and gives a non-destructive rough depth profile. Low Energy X-Ray Emission Spectroscopy, LEXES, gives a non-destructive As dose measurement, which depends on calibration against a bulk standard, for accuracy. MEIS gives a non-destructive depth distribution of atoms not in Si substitutional sites, and a dose calibrated by reference to amorphised Si. Dynamic SIMS provides very precise dose and depth distribution measurement to very low As concentrations, but is destructive and has a problem with the initial part of the depth scale and any As dose within it. Taken together a complete picture of the implant layer is obtained. Prior to annealing the As has a broad distribution, centered at about 50A depth. The outer oxide layer increases from 13A at 1E14 ions/cm2 dose to 18A for 2E15 ions/cm2 dose. ARXPS showed that samples from one particular implanter had a component of As2O3 very near the surface, well removed from the elemental As implant. The annealing conditions (N2 with 10% O2) double the oxide thickness (22A at 1E14 ions/cm2 dose; 38A at 2E15 ions/cm2), and cause the As to pile up just on the Si side of the SiO2/Si interface, but with a strong diffusion tail to 150A depth (SIMS). XPS shows that any oxide component is eliminated by anneal. A comparison of the MEIS to the SIMS depth distributions shows that the diffusion tail is in substitutional sites, and therefore not observable in the MEIS.

10:40am **AS+TF-ThM8 Hot Electron Transport Across Manganese Silicide Layers on the Si(001) Surface**, ***A. Stollenwerk***, *M.R. Krause, V.P. LaBella,* University at Albany SUNY

The need for high efficiency spin injection for spintronic applications has led to the study of different ferromagnetic interfaces. Recent theoretical studies have shown that the MnSi interface orders ferromagnetically.@footnote 1@ We performed ballistic electron emission microscopy (BEEM) on the MnSi/Si(001) Schottky barrier to study the hot electron transport properties. BEEM allows the interface to be probed on the nanometer scale and also gives the option to perform spin dependent measurements. Samples for this study were fabricated by electron beam deposition of Mn onto n-type Si(001) with thicknesses ranging from 50 to 200 Å. These layers were annealed at various temperatures in ultra high vacuum (UHV). The front side contact was fixed ex situ before the sample was reinserted into UHV to perform BEEM. Film composition has been determined by secondary ion mass spectroscopy (SIMS). The Schottky heights have been determined by fitting the BEEM spectra to the Bell-Kaiser model. The effects of temperature, film thickness and composition on the BEEM current will be discussed. @FootnoteText@ @footnote 1@S.A. Wolf et al., Science 294, 1488 (2001).

11:00am **AS+TF-ThM9 Optimization and Deposition of CdS Thin Films As Applicable to TiO@sub2@/CdS Composite Catalysis**, ***K. Prabakar***, *T. Takahashi,* Toyama University, Japan*; T. Nakashima,* Kashiwa Chuo High School, Japan*; Y. Kubota,* Yokohama City University, Japan*; A. Fujishima,* Kanagawa Academy of Science and Technology, Japan

Recently, the study of interparticle electron transfer between dissimilar semiconductors has received interesting investigations. Combining two semiconductor particles offers an opportunity to sensitize a semiconductor material having a large bandgap and energetically low-lying conduction band by another one having a small band gap and energetically high-lying conduction band. In our investigations, photosensitization of TiO@sub2@ by narrow band gap semiconductor such as CdS have been investigated and found to be effective for separation and transfer of photoexcited charge carriers. The TiO@sub2@ thin films were deposited by direct current reactive magnetron sputtering and CdS by chemical bath deposition. Thin films of CdS were deposited from a solution of analytical grade CdSO@sub4@ (1 M) and thiourea (1 M) in an alkaline solution of ammonia with a total volume of 80 ml. The temperature and time of the deposition were varied between 65 to 80 ° C and 30 to 60 minutes respectively. To vary the composition of the films, different concentrations of CdSO@sub4@ and thiourea were used. The optical band gap energy varied from 2.41 to 2.59 eV as the CdSO@sub4@ solution concentration increased from 0.4 to 2.8 ml while keeping the thiorea as 1.6 ml. The as deposited films were annealed at different temperatures to study the effect of structural (XRD) and surface properties (SEM and AFM) on the efficiency of the TiO@sub2@/CdS catalysis. The TiO@sub2@ films were found to be polycrystalline anatase structure with optical band gap energy of 3.1 eV. The degradation efficiency under visible light of methanol and methylene blue by TiO@sub2@/CdS films were investigated by FTIR and spectrophotometer respectively and the results are discussed in details. The visible light photocatalytic degradation efficiency TiO@sub2@/CdS is far higher than that of TiO@sub2@ film.

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