## **Tuesday Afternoon, November 3, 1998**

### Thin Films Division Room 310 - Session TF-TuA

#### In-situ Characterization of Thin Films

Moderator: J.J. Nainaparampil, Air Force Research Laboratory

2:00pm **TF-TuA1 Real Time Ellipsometry Study the Deposition of Barium Strontium Titanate Thin Films**, *Y. Gao, A. Mueller, E.A. Irene*, University of North Carolina, Chapel Hill; *O. Auciello*, Argonne National Laboratory, U. S. A.; *A.R. Krauss*, Argonne National Laboratory

(Br, Sr)TiO@sub 3@ (BST) has been considered to be a candidate high K dielectric material in dynamic random access memory(DRAM) capacitors.@footnote 1@ However, the interface layer formed between the BST thin film and substrate is an obstacle to obtain the desired dielectric constant and/or leakage current level for high density DRAM applications. Therefore, a clear understanding and then control of the interface reaction between a high K dielectric film and the substrate is tantamount for further progress in high K film technology. In previous work@footnote 2@ in our laboratory, the oxygen extraction and interface reactions of YBCO superconductor thin films on different substrates have been studied using real time ellipsometry. The results of this study comprise information on film growth kinetics and the oxygen in- and outdiffusion mechanism. Presently a similar study is ongoing on the interface reaction in ion sputtered BST thin films on Si substrates. The results of this study to be presented include: 1. real time film growth kinetics 2. sensitivity of bulk film composition to oxygen background pressure 3. interface reactions with Si and with various buffer layers 4. static and high frequency dielectric constants as well as leakage currents for BST films from various growth conditions 5. optical properties, composition, structure and morphology of BST films from various growth and postdeposition annealing conditions. @FootnoteText@ @footnote 1@D.E. Kotecki, Semiconductor International, Nov. 1996, pp109-116; @footnote 2@A. Michaelis and E.A. Irene et. al, Journal of Applied Physics, June 15, (1998).

2:20pm **TF-TuA2 Optical Constants of Crystalline WO@sub 3@ Deposited by Magnetron Sputterting**, *M.J. DeVries*, *C. Trimble*, *T.E. Tiwald*, *D.W. Thompson*, *J.A. Woollam*, University of Nebraska, Lincoln; *J.S. Hale*, J. A. Woollam Co., Inc.

Crystalline WO@sub 3-x@ is an infrared (IR) electrochromic material with possible applications in satellite thermal control and IR switches. Optical constants of electrochromic materials change upon ion intercalation, usually with H@super +@ or Li@super +@. Of primary concern for device design are the optical constants in both the intercalated and unintercalated states. In-situ and ex-situ ellipsometric data are used to characterize both the deposition process and the optical constants of the film. Ex-situ data from a UV-Vis-NIR ellipsometer are combined with that from a mid-infrared FTIR-based ellipsometer to provide optical constants over a spectral range of 190 nm to 30  $\mu$ m.

#### 3:00pm TF-TuA4 Ordered Binary Oxide Films: V@sub 2@O@sub 3@ (0001)/Al@sub 2@O@sub 3@/Mo(110), Q. Guo, D.Y. Kim, S.C. Street, D.W. Goodman, Texas A&M University

Ordered binary oxide films, vanadium oxide on aluminum oxide, on the (110) molybdenum surface have been prepared in ultra-high vacuum conditions and characterized by various surface analytical techniques. Results from Auger electron spectroscopy, low energy electron diffraction, high-resolution electron loss spectroscopy, X-ray photoelectron spectroscopy and ion scattering spectroscopy indicated that the vanadia films grew on the Al@sub 2@O@sub 3@/Mo(110) surface epitaxially as V@sub 2@O@sub 3@(0001). Results from electronic structure measurements show an increase in energy of the a@sub 1g@ level in the 3d band at 100 K, which is one possible contributor to the metal-insulator transition in V@sub 2@O@sub 3@.

# 3:20pm TF-TuA5 In-Situ Thin Film Characterization, J.M. Gibson, University of Illinois, Urbana INVITED

Control and understanding provide the two primary motivations for in-situ film characterization. For vacuum deposition, one powerful in-situ probe is the high-energy electron beam. Reflection High-Energy Electron Diffraction(RHEED) is a well-known method, used in Molecular Beam Epitaxy(MBE), for both purposes. Direct imaging using high energy electron beams forms the basis of transmission electron microscopy (TEM), which provides far more microstructural information than RHEED. Due to the instrumental complexity and invasiveness of the technique, however, insitu TEM is directed at understanding, rather than controlling growth. I illustrate with examples from my group's work which includes: understanding the growth of very thin oxides, both on metals@footnote 1@ and semiconductors;@footnote 2@ the nature of small particle sintering in the formation of nanophase metallic thin films;@footnote 3@ island growth and stress@footnote 4@ in epitaxial Ge on Si; and AlN epitaxy on sapphire.@footnote 5@ The emphasis is that understanding through in-situ microscopy can lead to control, without in-situ probes, through better understanding of growth processes. @FootnoteText@ @footnote 1@J. C. Yang and J. M. Gibson, Appl. Phys. Lett. 70, 3522 (1997). @footnote 2@X. Chen and J. M. Gibson, Appl. Phys. Lett. 70, 1462 (1997). @footnote 3@M. Yeadon, J. C. Yang, M. Ghaly, D. Olynick, R. Averback, and J. M. Gibson, Appl. Phys. Lett. 71, 1631 (1997). @footnote 4@R. D. Twesten and J. M. Gibson, Phys. Rev. B 50, 17628 (1994). @footnote 5@M. Yeadon, M. T. Marshall, F. Hamdani, S. Pekin, H. Morkoc, and J. M. Gibson, Journal of Applied Physics 83, 2847 (1998).

#### 4:20pm TF-TuA8 Direct Three-Dimensional Characterization of Buried Interface Morphology with Quantized Electron Waves, *I.B. Altfeder, D.M. Chen,* The Rowland Institute for Science

We present a novel in situ and nondestructive technique for characterizing buried interfaces in metal/semiconductor heteroepitaxy. The principle of the technique is based on the quantum confinement of the internal electron source in the metal film. Due to the high sensitivity of the quantized electron waves to the boundary conditions, discrete interference fringes are formed spontaneously on the surface of the film and can be directly imaged by a scanning tunneling microscope.@footnote 1@ These fringes coincide precisely with the substrate atomic terraces so that the metal appears to be "transparent". The absolute depth of the film, on the other hand, can be accurately determined form the tunnel I-V measurements of the quantized energy spectra near the Fermi level, hence making it possible to characterize the buried interface morphology in all three dimensions nondestructively. @FootnoteText@ @footnote 1@I. B. Altfeder, K. A. Matveev, and D. M. Chen, Phys. Rev. Lett. 78, 2815 (1997).

#### 4:40pm TF-TuA9 A Novel Design of a Reflecton Analyzer for Elemental and Isotopic Analysis by MSRI (Mass Spectroscopy of Recoil Ions)@footnote 1@, K.L. Waters, K. Baudin, J.A. Schultz, Ionwerks

Mass Spectroscopy of Recoil Ions (MSRI) is a recently developed technique which has been reviewed@footnote 2@ and has been used in real time monitoring of nitride@footnote 3@ and oxides growths. The technique is similar to Time of Flight SIMS (TOF/SIMS) in that a pulsed keV primary ion beam is impinging at grazing incidence onto the analyzed surface. Sputtered ions are collected and analyzed by TOF. By careful design of the extraction optics using differential pumping and by placement of the analyzer in the forward scattering angle at around 60-70 degrees, it is possible to achieve mass spectra of directly recoiled binary ions which are devoid of molecular interference at pressures in excess of 1mTorr. For the optic positioned at 1 inch from the focal point of the primary ion beam onto the sample, Mo isotopes have been resolved with a resolution at half maximum of 450. Operation at 2 inches from the focal point reduces the resolution to 250 and reduces the collection and transmission of recoiled ions by a factor of two. Examples will be given of the use of the MSRI technique for both exsitu and real time control of deposition process. @FootnoteText@ @footnote 1@Financial support of this work by US Air Force SBIR Contract F33615-970C-1035 @footnote 2@M. S Hammond, J.A Schultz, A. R. Krauss J.Vac.Sci. Technol. A 13(3) 1995 @footnote 3@E. Kim, I. Berishev, A. Bensaoula, S. Lee, S.S. Perry, K. Waters, J.A. Schultz Appl. Phys. Lett. 71 (21) 1997

#### 5:00pm TF-TuA10 The Interaction of Al Atoms with Surface-Bound Organic Functional Groups Studied by In-situ XPS, Infrared Spectroscopy and ToF-SIMS, A.E. Hooper, G.L. Fisher, Pennsylvania State University; R.L. Opila, Bell Laboratories, Lucent Technologies; N. Winograd, D.L. Allara, Pennsylvania State University

The interaction of vapor-deposited Al atoms with self-assembled monolayers of structure HS(CH2)15CH3 and HS(CH2)15COOCH3 was studied with multiple in-situ techniques over a range of Al coverages from submonolayer to multilayers. XPS and IR show the Al to be unreactive with the CH2 units but highly reactive with the oxygen atoms in the ester group to form a 1:1 Al:ester bridged type of complex with an intact CH3 group and a loss of double bond character for the C=O group. In the case of the CH3-termimnated surface, ToF-SIMS shows the Al atoms penetrate into the organic monolayer and diffuse to the Au-S interface. Both XPS and ToF-SIMS indicates that subsequent reaction with the S atoms appears to occur,

## **Tuesday Afternoon, November 3, 1998**

but in such a way as not to disturb the packing of the monolayer hydrocarbon chains. A dynamic fluctuation mechanism is proposed to explain the AI atom penetration in the CH3 case. In the ester-terminated film, penetration appears to be thwarted by the rapid reaction of the AI atoms with the ester terminal groups to form a dense monolayer of organometallic species.

### **Author Index**

### Bold page numbers indicate presenter

- A --Allara, D.L.: TF-TuA10, 1 Altfeder, I.B.: TF-TuA8, 1 Auciello, O.: TF-TuA1, 1 - B --Baudin, K.: TF-TuA9, 1 - C --Chen, D.M.: TF-TuA8, 1 - D --DeVries, M.J.: TF-TuA2, 1 - F --Fisher, G.L.: TF-TuA10, 1 - G --Gao, Y.: TF-TuA1, 1 Gibson, J.M.: TF-TuA5, 1 Goodman, D.W.: TF-TuA4, 1 Guo, Q.: TF-TuA4, 1 -H -Hale, J.S.: TF-TuA2, 1 Hooper, A.E.: TF-TuA10, 1 -I -Irene, E.A.: TF-TuA1, 1 -K -Kim, D.Y.: TF-TuA4, 1 Krauss, A.R.: TF-TuA1, 1 -M -Mueller, A.: TF-TuA1, 1

- O -Opila, R.L.: TF-TuA10, 1 - S -Schultz, J.A.: TF-TuA9, 1 Street, S.C.: TF-TuA4, 1 - T -Thompson, D.W.: TF-TuA2, 1 Trimble, C.: TF-TuA2, 1 - W -Waters, K.L.: TF-TuA9, 1 Winograd, N.: TF-TuA10, 1 Woollam, J.A.: TF-TuA2, 1