

Monday Evening Poster Sessions, November 2, 1998

Thin Films Division

Room Hall A - Session TF-MoP

Thin Films Poster Session

TF-MoP1 Studies on Anti-Glare, Anti-Static and Transparent Conductive Film on Display Tubes, G.K. Xi, G.H. Zhang, S.L. Li, S.M. Shao, W. Guo, X.J. He, Nankai University, China

Anti-glare, anti-static thin film and transparent conductive film are widely used in many fields of production, science and technology, such as solar cell, TV image display panel, instrument panel and transparent electrode. Anti-glare and anti-static double-layered coating on ordinary glass prepared by a sol-gel technique is introduced. The outer layer of low refractive index is coated on high refractive index inner layer. The main composition of outer layer is SiO_2 . Some SnCl_4 added into this film increases the conductivity of this film. The latter can be replaced by other hygroscopic metal salts, such as $\text{Al(NO}_3)_3$, AlCl_3 , ZnCl_2 , etc.. The inner layer is composed of TiO_2 and SiO_2 . The glass panel with the double-layered coating shows the resistivity of $10^9 \text{ } \Omega/\text{square}$ and reflectance of 1.6% which is reduced to one-fourth of that without coating. A transparent film with high conductivity on ordinary glass panel prepared by a sol-gel technique is also introduced. This Sb-doped SnO_2 film is made from alkoxide which was previously prepared in our lab. This ATO film has a polycrystalline structure. The resistivity of the ATO film has minimum value of $10^3 \text{ } \Omega/\text{square}$ while the doping level of the solution is about 7%. Finally, factors such as environmental temperature and humidity, coating technology, effecting the characteristics of these films are discussed.

TF-MoP2 Humidity Sensing Properties of Plasma Polymerized Organic Thin Films, G.B. Park, Yuhan College, Korea; J.T. Kim, D.C. Lee, Inha University, Korea; C. Kim, Korea Electronic Technology Institute

In order to fabricate humidity sensitive films, the mono-layer polymer thin films from various monomers were deposited on a comb-shaped electrode using a capacitive-coupled gas flow type plasma polymerization apparatus with rf power. The humidity sensing characteristics of these thin films were measured by means of changes in electrostatic capacitance within the frequency and humidity range of 60 Hz - 100 kHz and 20% RH - 90% RH, respectively. The capacitances of these thin films were increased with increasing relative humidity. And the increments of the capacitance grew larger with a lower discharging power and a shorter polymerization time. The linearity and increment of the capacitance change became apparent with the lower frequency of input power to thin films during the capacitance measurements. The copolymerized thin films, vinylacetate and methylmethacrylate, were fabricated using the conditions of low discharging power and short discharging time. The capacitances of these films were remarkably changed showing the linear behaviors, and these changes were continued from 20% RH to 90% RH.

TF-MoP3 Rotating-Compensator Spectroscopic Ellipsometry: Applications of Four-Parameter Stokes Vector Spectroscopy to Real Time Characterization of Non-Ideal Thin Films, R.W. Collins, P.I. Rovira, J.C. Lee, Pennsylvania State University

We have developed a multichannel ellipsometer in the rotating-compensator optical configuration, i.e., (fixed polarizer)-(sample)-(rotating compensator)-(fixed analyzer). This instrument has the advantage of providing the spectrum in the degree of polarization p of the light beam reflected from the sample, in addition to the spectra in the ellipsometric angles (ψ , Δ). In a recent advance, we have extended the instrument capabilities to a simultaneous measurement of the spectral reflectance R of the sample. As a result, the new instrument can collect four-parameter spectra [(ψ, Δ, p, R)] that characterize the unnormalized Stokes vector of the reflected light beam. The minimum measurement time is 32 ms for all four spectra from 1.5 to 4.0 eV. In this paper, instrumentation and calibration issues specific to the simultaneous reflectance measurement will be described. Applications of the rotating-compensator instrument to date include (i) optical anisotropy in nanoscale sculptured thin films of MgF_2 , (ii) nucleation and growth of nanocrystalline and polycrystalline diamond films, and (iii) optical properties, structure and stability of specular and textured transparent conducting oxide thin films. We review these applications and highlight the unique capabilities developed so far. These include the use of p along with (ψ, Δ) to characterize the evolution of thickness non-

uniformity during the growth of diamond films, and the use of R along with (ψ, Δ) to characterize the effect of annealing and H_2 -plasma exposure on the optical properties and surface roughness on micro/macroscale for textured SnO_2 used in photovoltaics applications.

TF-MoP4 Characterization of Thin Metal Films Processed at Different Temperatures, L. He, J.E. Siewenie, Northern Illinois University

Thin metal films are of considerable interest for electronic device fabrication. Not only do these films provide electrical interconnection between circuit elements, they can also be an integral part of a circuit element, as in the case of Schottky diodes and metal semiconductor field-effect transistors (MESFETs) and metal-semiconductor-metal photodetectors (MSM PDs). It is well known that the electrical conduction in metals is due to electrons, while electrical resistivity, defined as the reciprocal of the conductivity, is the result of electron collisions. The high resistivity of thin metal films result in drawback of their applications in afore mentioned devices. Recent studies have been conducted in thin metal films obtained by low temperature ($T = 77\text{K}$) deposition. Comparing to the same film formed at room temperature, the LT film resistivity could be 4 to 5 orders lower in magnitude. In another hand, metal/semiconductor Schottky barrier heights are significantly increased in materials including InP, GaAs, and InGaAs. This work extensively studied the electrical and micro-structural properties of several often used metal films including Au, Ag, Al, Pt, Pd, and Ni formed at LT and RT. Atomic force microscopy (AFM), transmission electron microscopy (TEM), and in-situ resistivity measurements were conducted. In AFM surface scanning, surface morphology consistently showed the LT and RT films difference in the grain sizes. The larger grain size in LT thin film explains the lower resistivity. TEM electron diffraction pattern showed very different degree of crystallization of the LT and RT thin films. The LT film diffraction pattern suggests that LT films develop a less regular structure which may be responsible for the Schottky barrier enhancement. LT deposition prevents both adatom diffusion and re-evaporation, leading to the earlier conducting than the same RT films.

TF-MoP5 Optical Properties of the Ge:Sb:Te System, E. Garcia-Garcia, Univ. Autonoma de Queretaro, Mexico; A. Mendoza, G. Martinez-Montes, Univ. Autonoma de Puebla, Mexico; Y.V. Vorobiev, J. Gonzalez-Hernandez, CINVESTAV-IPN, Mexico; B.S. Chao, Energy Conversion Devices

Stoichiometric compositions of the Ge:Sb:Te system are commercially used for optical data storage. In this work, we have measured using ellipsometry, the optical constants (n, k) in the range of 1.4 to 6 eV in all the stoichiometric composition in the Ge:Sb:Te system. It is known that the amorphous phase of these compositions undergoes an amorphous-to-crystalline transition at temperatures in the range from 140-160 °C depending on Sb concentration. The crystalline structure of this phase is the fcc. Heat treatments above the mentioned range produce a new crystalline-to-crystalline transition from the fcc to the hexagonal phase. The n values for the amorphous and crystalline fcc phase are not so different and both decrease from a value of about 5 to 1.4 eV to a value of 1 at 6 eV. In general, this phase transition produces an increase in the values of k in the whole energy range. The observed changes in n and k in the hexagonal phase are more complex due to the increase in the free charge density and will be discussed in the extended presentation.

TF-MoP6 Influence of Annealing Temperature on the Formation and Characteristics of Sol-gel Prepared ZnO Films, R. Castanedo-Pérez, O. Jiménez-Sandoval, S.J. Jiménez-Sandoval, A. Maldonado-Alvarez, J. Márquez-Marín, G. Torres-Delgado, Cinvestav-IPN, Mexico

ZnO films have been obtained by the sol-gel method, from a $\text{Zn(OOCCH}_3)_2$ precursor, on silica glass and silicon wafer substrates. The films, obtained by a single dipping procedure, were characterized by FT-IR and UV-VIS spectroscopy, atomic force microscopy, X-ray diffraction and ellipsometry measurements. Untreated and single-step, annealed (100-450°C) films were studied, in order to analyze the influence of temperature on the formation and properties of the ZnO coatings. Remarkably, these results indicate that ZnO forms at considerably lower temperatures than 450°C, which is usually considered in literature as a reference temperature for the formation of ZnO. Thus, a sharp absorption edge of ZnO at ca. 380 nm, can be neatly observed in the UV-VIS spectra of films annealed at 200 and 300°C, and accordingly, IR data indicate the absence of organic groups at these temperatures. Somewhat surprisingly, at 400 and 450 °C, the ZnO optical absorption edge is not as sharp as is at lower temperatures. Atomic Force Microscopy results show larger grain sizes as the annealing temperature is increased. The X-ray diffraction

Monday Evening Poster Sessions, November 2, 1998

patterns show that the films are polycrystalline and also evidence the formation of ZnO at temperatures as low as 200°C.

TF-MoP7 Structural Characterization of SrBi@sub 2@Ta@sub 2@O@sub 9@ Ferroelectric Thin Films Grown by PLD on Pt and RuO@sub 2@ Bottom Electrodes, J.M. Siqueiros, UNAM, Mexico; M.P. Cruz, CICESE, Mexico; J. Portelles, Universidad de la Habana, Cuba; R. Machorro, G. Hirata, S. Wang, UNAM, Mexico

To study the effect of the bottom electrode on the properties of the ferroelectric layer, thin SBT (SrBi@sub 2@Ta@sub 2@O@sub 9@) films were deposited by PLD on Pt/Ti/Si, Pt/TiO@sub 2@/Si and RuO@sub 2@/Si grown by DC sputtering. Due to the previously reported experience that the Ti in the adherence promoter layer diffuses to the Pt surface, a study of the SBT/electrode interface using AES and TEM is performed. XRD, SEM, STM and ellipsometry measurements were performed on the SBT films and compared with the corresponding measurements on the ceramic used as target for the deposit, which showed a layered perovskite structure.

TF-MoP8 Characteristics of CuInSe@sub 2@ Thin Films Prepared in Different Selenization Pressures, S.D. Kim, C.H. Chung, Seoul National University, Korea; K.H. Yoon, J.S. Song, Korea Institute of Energy Research, Korea; H.J. Kim, Seoul National University, Korea

CuInSe@sub 2@ based solar cell has great interest because CuInSe@sub 2@ has high absorption coefficient, suitable bandgap energy, good thermal stability, and good lattice match with window layers such as CdS, Cd(Zn)S. Its conversion efficiency has been reached above 15%. The selenization method has been known as an excellent technique to acquire low cost and high efficiency CuInSe@sub 2@ thin films. The effects of chamber pressure during selenization of Cu-In alloy layers on the optical, electrical and structural properties of CuInSe@sub 2@ films were investigated. The uniform Cu-In alloy layers could be reproducibly deposited on the glass substrate by dc co-sputtering method. The two atmospheres, Ar atmosphere at 1 atm and in vacuum of 10 mTorr, were chosen for the selenization of Cu-In layers. The properties of all films were analyzed by XRD, SEM, EDX, four point probe, Raman spectroscopy and photoluminescence. Cu-In precursors consisted of two phases, CuIn@sub 2@ and Cu@sub 11@In@sub 9@, and the amount of Cu@sub 11@In@sub 9@ phase increased with varying the composition from In-rich to Cu-rich. Less compounds of Cu-Se and In-Se were observed during the early stage of selenization and also CuInSe@sub 2@ single phase was more easily formed in vacuum than at atmospheric pressure. Therefore, CuInSe@sub 2@ films selenized in vacuum showed large grain size, smooth surface, dense microstructure, high Raman peak intensity and no secondary phases with near-stoichiometric composition. Since CuInSe@sub 2@ films selenized in vacuum could hardly release the intrinsic stress due to dense structure, Raman peak of 173 cm@super -1@, A1 mode of CuInSe@sub 2@ charcopyrite phase, shifted to higher frequency and had a broad full width of half maximum.

TF-MoP9 Growth and Characterization of Epitaxial Films of Tungsten-Doped Vanadium Oxides on Sapphire (110) by Reactive Magnetron Sputtering, P. Jin, M. Tazawa, M. Ikeyama, S. Tanemura, National Industrial Research Institute of Nagoya, Japan; K. Macak, X. Wang, U. Helmersson, Linköping University, Sweden

Some vanadium oxides undergo a semiconductor-to-metal phase transition with significant changes in optical, electrical and magnetic properties. Replacement of V by metals such as W, Mo affects greatly the properties. Since thin films of such materials are candidates for switching or memory devices, it is necessary to investigate the growth, structure and properties of the metal-doped vanadium oxides particularly in the form of epitaxial film. In this study, films of W-doped vanadium oxides were epitaxially grown on sapphire (110) by reactive sputtering a V-W alloy target. With computer control of deposition parameters, especially the oxygen flow into the Ar+O@sub 2@ discharge, a series of epitaxial films having structures of not only the best known MO@sub 2@ (M=V+W) but also others from M@sub 2@O@sub 3@ to M@sub 2@O@sub 5@ were obtained. The films were studied with X-ray diffraction (XRD), atomic force microscopy (AFM), Rutherford backscattering spectrometry (RBS), etc. The XRD theta-2theta scan shows only pairs of peaks corresponding to those from the film and the substrate. Furthermore, a series of films formed by varying slightly the oxygen flow exhibit a continuous shift of the XRD peaks in response to their metal-to-oxygen ratio. In other words, a continuous structural change was obtained by precise process controlling. The XRD pole figure study confirmed the epitaxial relationships. The phase transition properties were studied from the change in electrical resistivity against temperature. The

results demonstrate strong influences both from the metal-to-oxygen ratio and the tungsten doping amount.

TF-MoP10 Stability of Transparent Conducting Oxide Films for Use at High Temperatures, T. Minami, T. Miyata, T. Yamamoto, Kanazawa Institute of Technology, Japan

The stability of transparent conducting oxide (TCO) films in various atmospheres at high temperatures is important for applications such as transparent heaters and also in optoelectronic device fabrication processes. However, the electrical, optical and chemical properties of TCO films at high temperatures above 500 ° C have not been investigated because of the melting point of glass substrates. In this paper, we describe the stability of various TCO films at high temperatures in various atmospheres. Various TCO films consisting of binary compounds such as ZnO, In@sub 2@O@sub 3@ and SnO@sub 2@, ternary compounds such as Zn@sub 2@In@sub 2@O@sub 5@, In@sub 4@Sn@sub 3@O@sub 12@, GaInO@sub 3@, ZnSnO@sub 3@ and MgIn@sub 2@O@sub 4@ or multicomponent oxides composed of two binary compounds or two ternary compounds were tested in this work. The TCO films were deposited by magnetron sputtering on quartz substrates at room temperature or 350 ° C. The tests were carried out in various atmospheres such as air, argon gas and vacuum at temperatures up to 1000 ° C. The SnO@sub 2@, In@sub 2@O@sub 3@ and In@sub 4@Sn@sub 3@O@sub 12@ thin films were found to be more stable than other materials. It can be concluded that stability at high temperatures was mainly determined by the metal elements contained in the TCO film; high stability was obtained in TCO films rich in Sn and In.

TF-MoP11 Texture and Grain Size Modification through the Different Sintering Conditions for Ceramics of the Sr@sub 0.5@Ba@sub 0.5@TiO@sub 3@ Type, J.M. Siqueiros, UNAM, Mexico; J. Portelles, S. Garcia, Universidad de la Habana, Cuba; S. Aguilera, Universidad de Catolica de Norte, Chile; M. Xiao, UNAM, Mexico; A. Fundora, Universidad de la Habana, Cuba

The variation of the sintering conditions: temperature and sintering time, strongly modify the texture and grain size of the SBT ferroelectric compound (Sr@sub 0.5@Ba@sub 0.5@TiO@sub 3@). In this report, the sintering time is varied from 1 to 5 hours. The grain size increases as the sintering temperature is varied from 1200 to 1450 @super o@C. SEM studies allow us to determine the corresponding microstructure for each temperature and sintering time. A linear dependence of logarithm of the grain size with the sintering temperature is found. A change in the dielectric permittivity and the polarization with the sintering conditions is also reported. The analysis of the results is supported with XRD, thermoelectric and dielectric hysteresis measurements

TF-MoP12 Microstructure Study of PMN-PT Films Grown on Metal Electrodes by PLD, J.M. Siqueiros, UNAM, Mexico; J. Portelles, A. Fundora, Universidad de la Habana, Cuba; S. Aguilera, Universidad de Catolica de Norte, Chile

Thin ferroelectric films obtained by PLD from non-stoichiometric Pb(Mg@sub 1/3@Nb@sub 2/3@)O@sub 3@-PbTiO@sub 3@ (PMN-PT) ceramic targets are studied. The morphology of the resulting films for the 2.7PMN-0.1PT composition obtained in N@sub 2@ and O@sub 2@ atmospheres at room temperature obtained by SEM is reported. It is observed that, for similar deposit conditions, thicker films with columnar structure are produced in the O@sub 2@ atmosphere as compared with those grown on N@sub 2@ where very low crystallinity was detected. The film composition was determined by Auger electron spectroscopy and XPS. TEM measurements of the N@sub 2@ grown films showed scattered nanostructures embedded in a dominion structure in the paraelectric state, since the measurements were performed at room temperature, above the Curie temperature of the ceramic (15 @degree@C). This result seems to imply a diffuse phase transition associated to the film. Regions of high concentration of Niobium were detected by XPS evidencing the presence of pyrochlores, a situation confirmed by XRD. The PMN-PT/electrode interface is analyzed by TEM for samples annealed at different temperatures and the results are correlated with those obtained by XRD and SEM.

TF-MoP13 Columnar Growth of Tin from Liquid Metal Ion Source Studied by In-situ Transmission Electron Microscopy, H. Kimata, Y. Kondo, ERATO, Japan Science and Tech. Corp., Japan; K. Takayanagi, Tokyo Institute of Technology, Japan

A miniaturized liquid metal ion source (LMIS) built in a conventional transmission electron microscope (TEM) was developed to observe field desorption of tin ions and droplets, and growth of deposit from them on

Monday Evening Poster Sessions, November 2, 1998

substrates in-situ. The LMIS has a reservoir, a needle and a filament which heats the liquid tin in the reservoir. The needle is faced to an extraction electrode with a small hole. A shield plate, with a small hole, is placed behind the extraction electrode. The substrate is placed behind the shield plate. Potentials of the LMIS and the extraction electrode are ground and negatively high, respectively. The shield plate and the substrate are usually ground. Thus emitted ions are decelerated to have near zero energy at the substrate, and they land the substrate very softly. Positive bias is occasionally applied to the shield plate and the substrate, to repel positive ions for selecting neutrals from mixture of ions and neutrals. Tin ions and neutrals were emitted from the LMIS at extraction voltage ranged from 4 to 6kV, with an emission current of about 40×10^{-6} A. We observed growth of tin on a substrate in-situ. The substrate was an amorphous carbon film on a thin tungsten wire. We found that columnar tin grew; typically 500nm in length and 50nm in width, when the substrate and the shield plate were ground. Columnar growth was not found when +100V bias was applied to the substrate and the shield plate, to repel the ions. The experiments showed that ions are essential for the columnar growth. The strong magnetic field (~ 2 T) of the objective lens in the TEM, might play a role for the growth.

TF-MoP14 Characterization of TiOx Film deposited on Ti-6Al-4V Alloy by Reactive Sputtering in Oxygen Atmosphere, T. Sonoda, M. Kato, National Industrial Research Institute of Nagoya, Japan

Coating of Ti-6Al-4V alloy substrates with TiOx films by reactive sputtering in oxygen atmosphere was examined, not only to improve the biocompatibility of the alloy but also to enhance the bonding of the alloy implants to living bone. The reactive sputter deposition was carried out in oxygen gas using a magnetron d.c. sputtering apparatus with a pure titanium target. Thus the TiOx films were deposited on the alloy substrates at the rate of $300 \text{ \AA}/\text{min}$ by the magnetron sputtering, even under the surface condition at the target to be in the reactive mode. Due to reactive products covering the surface. The characteristics of the deposited TiOx films were investigated. Under visual observation, the deposited films looked light gray and appeared to be uniform and adhesive. Under SEM, the surface of the films was found to have a net-like microstructure which looked like a net consisting of micron-ordered fine meshes. Under AES, the Ti/O ratio of each film was constant in depth direction of the film. Based on XRD, it was concluded that not only the oxides (such as rutile and brookite) but also the suboxides such as Ti_4O_7 and Ti_{11}O_6 were formed in the film. Therefore, it was assumed that the obtained TiOx films improved the biocompatibility of the alloy and enhanced the bonding of the alloy to living bone. Furthermore, the hardness of the TiOx films reached over $H_v=800$ under the film thickness of $0.8 \mu\text{m}$, concluding that the coating with the films improved the hardness of the alloy. Wisby et al., *Biomaterials* 12(1991)470. Kitsugi et al., *J. Biomed. Mater. Res.* 32(1996)149. Schiller et al., *Thin Solid Films* 111(1984)259.

TF-MoP15 Photoconductivity of Free-standing Diamond Film, S.-H. Kim, Silla University, South Korea; I.T. Han, Samsung Advanced Institute of Technology, South Korea; T.-G. Kim, Miryang National University, South Korea

Thick diamond film having $700 \mu\text{m}$ thickness was deposited on polycrystalline molybdenum (Mo) substrate using high power (4 kW) microwave plasma-enhanced chemical vapor deposition (MPECVD) system. We could achieve free-standing diamond film by detaching as-deposited diamond film from the substrate. Parallel-type diamond photoconductors were fabricated on either the growth side or the substrate side of free-standing diamond film via ohmic contact metallization. We investigated the variation of photoconductivity after exposing the film surface to either oxygen or hydrogen plasma. At as-grown state, the growth side showed noticeable photoconductivity, while the substrate side gave little photoconductivity. The oxygen plasma treatment of these sides led to the insulators. After exposing the film surfaces to hydrogen plasma, on the other hand, we could observe not only distinct photoconductivity at the substrate side but also the reappearing of photoconductivity at the growth side. Finally, we suggest that the dangling hydrogen bond on the film surface may play an important role to create the photoconductivity of this film surface.

TF-MoP16 Target Compound Layer Formation during Reactive Sputtering, L.B. Jonsson, T. Nyberg, S. Berg, Uppsala University, Sweden

It is well known that a compound layer may form at the target surface during reactive sputtering. However, the significance of this layer for the

response to a change in target conditions has so far not been carefully investigated. The standard model for the reactive sputtering process does not allow for calculations of the compound thickness at the target surface. For simplicity it has been assumed that a single monolayer is responsible for the poisoning of the target. However, experiments clearly indicate that the compound layer thickness may be significantly thicker than one monolayer. For several reasons it is important to be able to quantify the thickness of this layer. The formation of the compound layer introduces memory effects into the system when the processing conditions are changed. The delay time for sputter erosion of the compound layer depends strongly on the thickness of the layer. We will present an extension of the basic reactive sputtering model that explains the formation of an arbitrary thickness of the compound layer at the target surface. From this model it is possible to examine the layer thickness dependence on the major processing parameters (reactive gas supply, sputtering power etc.). Optical emission spectroscopy studies of sputtered target material and transient target voltage response measurements confirm the validity of the new model. Blom, T. Larsson, and C. Nender, *J. Vac. Sci. Technol. A* 5, 202-207 (1987).

TF-MoP17 Characterization of YSZ(Yttria-Stabilized Zirconia) Thin Films Prepared by RF-Magnetron Sputtering for Oxygen Gas Sensor, J.W. Bae, J.Y. Park, G.Y. Yeom, Sungkyunkwan University, Korea; K.D. Kim, Korea Gas Corporation, KOREA; Y.A. Cho, J.S. Jeon, D.S. Choi, Korea Gas Corporation, Korea

Yttria-stabilized zirconia (YSZ), a well known oxygen ion conductor, is one of the many solid state ionic materials utilized in the variety of electrochemical devices including fuel cells, oxygen pumps, and chemical gas sensors. Commercial YSZ oxygen gas sensors rely on the traditional bulk ceramic fabrication and require temperatures above 600°C to achieve sufficient ionic conductivity. However, if the YSZ could be applied as a thin film, it would offer many advantages including compact dimensions with smaller power consumption, reduced ohmic losses, and lower operating temperatures. In this experiment, yttria-stabilized zirconia (YSZ) films ($0.5\text{--}1.5 \mu\text{m}$) were deposited on Pt/NiO-Ni mixed reference layer/SiO₂ substrates to characterize film properties using an RF-magnetron sputter deposition system and zirconia stabilized with 8mol% yttria (Y₂O₃) was used as a sputter target. The plasma atmospheres were pure Ar or mixtures of Ar and O₂. We employed X-ray diffraction to study the structure of YSZ films and scanning electron microscopy (SEM) to examine the film surface morphologies. Analyses by Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) were performed to examine Zr, Y, and oxygen compositions in YSZ thin films and the uniformity in compositions. Transmission electron microscopy (TEM) was employed to examine the microstructural details and crystallography of the films. Gas-sensing test was carried out for Pt/YSZ/Pt/NiO-Ni/SiO₂ film structures exposed to an atmosphere of oxygen controlled composition. The deposition rates of YSZ thin films increased with increasing rf power and total pressure, and decreased with increasing O₂ concentration. The preferred orientation of deposited YSZ films changed from (111) to (220) with increasing thickness. O/Zr ratio analyzed by XPS was increased from 1.94 to 2.4 with increasing O₂/(Ar+O₂) ratio. But yttria atomic percentage in the YSZ films were not changed. More detailed physical and chemical characteristics of YSZ thin films and their relations to gas-sensing properties will be discussed in the presentation.

TF-MoP18 Growth Characteristics and Deposition Mechanism of SrTiO₃ Thin Films by Plasma Enhanced MOCVD, Y.-B. Hahn, D.O. Kim, K.S. Nahm, Chonbuk National University, Korea

Dielectric SrTiO₃ ultra thin films having 30 - 75 nm thickness were deposited on Pt/Si and Ir/Si substrates by plasma enhanced MOCVD using high purity Ti(O-i-C)₃H₇ and Sr(tmhd)₂ and O₂. Depositions were carried out under various operation conditions. The deposition rates were substantially influenced by bubbler temperature (T_b), substrate temperature (T_s) and rf power. The optimum conditions of deposition were T_b = 60°C for Ti(O-i-C)₃H₇ and 220°C for Sr(tmhd)₂, T_s = 550°C , 130 sccm O₂, and 160 W rf with carrier gas flow rates of 40 sccm for Ti(O-i-C)₃H₇ and 130 sccm for Sr(tmhd)₂. The deposition process was controlled by chemical reaction at $< 500^\circ\text{C}$, and by mass transfer above 550°C . Decomposition of Ti(O-i-C)₃H₇ was enhanced with increasing rf power, but that of Sr(tmhd)₂ was greatly affected by the substrate

Monday Evening Poster Sessions, November 2, 1998

temperature. A deposition mechanism of SrTiO₃ was proposed based on the decomposition rates of Ti(O-i-C)₃H₇@sub 4@ and Sr(tmhd)₂@.

TF-MoP19 Investigation of Ti and Cu Atom Density in Magnetron Sputtering Process Using Atomic Absorption Spectroscopy, E. Augustyniak, S.V. Filimonov, C. Lu, Intelligent Sensor Technology

A hollow cathode lamp based atomic absorption monitor is applied to measure the relative atom density in a planar DC magnetron sputtering system for various Argon pressures (from 1 to 30 mTorr) and input powers (from 0.3 to 20 W/cm²). Deposition rates for Ti and Cu are monitored with a piezoelectric microbalance in order to infer the absolute atom densities. Absorption versus deposition data obtained from thermal evaporation of the same materials at Argon pressures in the range from 10⁻⁶ to 10⁻³ Torr are used to estimate the degree of thermalization in sputtering process. Near the target, at distance shorter than one mean-free path of sputtered atoms, the film grows mostly due to deposition of energetic atoms. However, even in this region the thermalized atoms have a significant contribution to the total absorption of resonant radiation. The experimental data show that the reduction of local number density caused by the introduction of a nearby sink for sputtered atoms, e.g., a substrate or a sensor, can be as much as 50%. Ambient gas heating in the vicinity of the target at high input power results in suppressed thermalization and decreased sensitivity of atomic absorption. The combination of atomic absorption spectroscopic and gravimetric (by piezoelectric microbalance) measurements can be a powerful tool for sputtering process characterization.

TF-MoP20 A Study of the Effect of Low Dielectric Constant PAE-2 by Plasma Treatment, T.C. Chang, National Nano Device Laboratory, Republic of China; M.F. Chou, National Chiao Tung University, Republic of China; T.W. Hsiao, National Yun-Lin University, Republic of China; J.Y. Lin, National Yun-Lin University, Republic of China, Taiwan, Republic of China; C.Y. Chang, National Chiao Tung University, Republic of China, Taiwan, Republic of China; M.S.K. Chen, A. Tuan, Air Product Company, Republic of China, Taiwan, Republic of China; S. Chou, San Fu Company, Republic of China

As the device dimensions continue to shrink to 0.25 μm and below, the interconnect delay becomes a limiting device factor and increases device speed. Integrating a low dielectric constant ILD into device is a way to reduce the interconnect delay time constant. Low density material such as PAE-2 can offer lower dielectric constant than conventional silicon dioxide insulator. The PAE-2 film has the functional groups with oxygen linkage, so an oxidative post treatment could enhance the properties of PAE-2 film. In this work, we study the effect of post-plasma treatment of PAE-2 film characteristics. The dielectric constant of PAE-2 film is found to be reduced with the application of O₂ plasma treatment. Also, the optimized condition of O₂ plasma treatment is obtained. A model is proposed to explain the effect of post-plasma treatment in PAE-2 film.

TF-MoP21 Porous Polycrystalline Silicon Micro-Electronic Sensor, P.G. Han, The City University of Hong Kong, China; H. Wong, The City University of Hong Kong, China, Hong Kong, China; M.C. Poon, The Hong Kong University of Science & Technology, China, Hong Kong, China; N. Wang, The City University of Hong Kong, China, Hong Kong, China

Porous Polycrystalline Silicon (PPS)-based microelectronic sensors have been explored in our laboratory. Using n-type epitaxial silicon wafer as the substrate, undoped poly-Si films with thickness of 700 nm were deposited by thermal decomposition of silane gas (SiH₂) in a low pressure chemical vapor deposition (LPCVD) reactor. Boron was then diffused into the samples at 950°C for 20 min to form a p-type polycrystalline silicon film. The poly-Si film was selectively anodized in HF(50%vol):C₂H₅OH(96%vol)=1:1 solution with Pt electrode at a current density of 10 mA/cm² for 10 min at room temperature. The sensitivity of the sensors based on this PPS has been characterized and analyzed at different ambient pressures and temperature as well as gas species. Results show that the current increases remarkably as the pressure decreases and over two order of magnitude change has been detected when the vacuum pressure was pumped to 10⁻² ATM. We find that both ethanol and acetone vapors can make the current increase obviously. In acetone vapor, the device even behaves like a diode. The analysis of the electrical conduction mechanisms of the sensors in vacuum environment and organic vapors will be conducted in this paper. In addition, since the fabrication process is simple, and the sensor characteristics are repeatable and reproducible, it can be easily integrated with the VLSI technology.

TF-MoP22 Thickness and Index Measurement of Transparent Thin Films Using Neural Network Processed Reflectance Data, M.F. Tabet, W.A. McGahan, Nanometrics Inc.

Artificial neural networks and the Levenberg-Marquardt algorithm are combined to calculate the thickness and refractive index of transparent thin films from spectroscopic reflectometry data. A neural network is a set of simple, highly interconnected processing elements imitating the activity of the brain which are capable of learning information presented to them. Reflectometry has been used by the semiconductor industry to measure thin film thickness for decades. Modeling the optical constants of a film in the visible region with a Cauchy dispersion model allows the determination of both thickness and refractive index of most transparent thin films from reflectance data. In this work Artificial neural networks are used to obtain good initial estimates for thickness and two Cauchy parameters A_n and B_n , these estimates are then used as the starting point for the Levenberg-Marquardt which does a few iterations to find the final solution. This measurement program was implemented on a Nanometrics NanoSpec 8000XSE and will measure thickness and index of transparent films in the range of 1000 to 16000 Å in an average of four seconds.

TF-MoP23 Characterization of PECVD Hydrogenated Amorphous Silicon (a-Si:H), T.C. Ang, Nanyang Technological University, Singapore, Republic of Singapore; M.S. Tse, Nanyang Technological University, Singapore; L.H. Chan, Chartered Semiconductor Manufacturing Ltd., Singapore, Republic of Singapore; J.L. Sudijono, Chartered Semiconductor Manufacturing Ltd., Singapore

Unhydrogenated amorphous silicon has a high defect density and this prevents it from being useful for electronic devices. Hydrogen incorporation in a-Si eliminates defects and the quality of a-Si:H depends on the way the hydrogen is incorporated rather than its content in the film. The type of plasma used and the deposition conditions determine the nature of the hydrogen bonding and the film quality. In this paper, the film characteristics of PECVD a-Si:H deposited using pure silane plasma and silane plasma diluted with argon at different deposition temperatures are studied in terms of the deposition rate, hydrogen content, refractive index and film morphology. Fourier transform infrared spectroscopy (FTIR) spectra showed that in a-Si:H films deposited with argon dilution of the silane plasma, hydrogen is bonded to Si only in monohydride groups (SiH) whereas with a monosilane glow discharge plasma, the spectra revealed the presence of SiH, SiH₂ and (SiH₂ + SiH_n) bonds. Higher hydrogen content was measured in low temperature deposited films. The RI and absorption coefficient (n, k) values of low temperature deposited films were lower and are consistent with the higher hydrogen content observed in the FTIR spectra. This is mainly due to the decreasing Si densities of the films due to the formation of Si-H and SiH_n (n>1) bonds in the films. Atomic force microscopy (AFM) was used to investigate the effect of deposition temperature on the film morphology and results showed that smaller grain sizes and tighter packing densities were characteristic of higher temperature deposited films. Results obtained through FTIR and spectroscopic ellipsometry (SE) show a correlation between the hydrogen content and the RI. Films deposited at higher deposition temperatures have lower H content and higher RI values as compared to lower temperature deposited films. Results from electrical tests on a-Si:H films deposited at different temperatures revealed changes in film quality which are consistent with the FTIR and SE observations. @FootnoteText@ W. Beyer and H. Wagner, J. non-crystall. Solids 59/60, 161 (1983). W. Beyer, Tetrahedrally-Bonded Amorphous Semiconductors, Ed. D. Adler and H. Fritzsche, Plenum Press, New York, 129, 1985. W. Beyer, Physica (Utrecht), 170B, 105, 1991. R. A. Street, Hydrogenated Amorphous Silicon, Cambridge Solid State Science Series (Cambridge University Press, Cambridge, 1991). Akihisa Matsuda, Plasma Physics Control Fusion, 39, pp.A431, 1997.

TF-MoP24 The Analysis of Silicon Oxynitrides with Spectroscopic Ellipsometry and Auger Spectroscopy, Compared to Analyses by RBS, and FTIR, H.G. Tompkins, R. Gregory, P.W. Deal, S.M. Smith, Motorola, Inc.

This work addresses the issue of whether spectroscopic ellipsometry, using the effective medium approximation (SE-EMA), may be used meaningfully to analyze PECVD silicon nitride films. We use RBS and FTIR as reference methods and compare the results to the results of SE-EMA analyses and Auger analyses. The results are that Auger analysis, using properly determined sensitivity factors, gives compositions which are within the uncertainty of the reference methods. SE-EMA, on the other hand, always overestimates the oxide contribution and underestimates the nitride contribution. Probable causes are discussed.

Monday Evening Poster Sessions, November 2, 1998

TF-MoP25 Density Measurement of Thin Glass Layers for Gas Barrier Films, N. Fukugami, H. Nishino, M. Yanaka, Y. Tsukahara, Toppan Printing Co., Ltd., Japan

In the food packaging industry, there is an increasing demand for transparent gas barrier films consisting of a 10 ~ 100 nm thick glass layer deposited on a polymer substrate. An advantage of such films is that microwaves and light waves can penetrate. It was confirmed that our glass layer had no macro defects or pinholes, and the gas penetration mechanism was mainly due to a nano structure of the glass layer. It is anticipated that the density of the glass layer is an important factor to characterize such nanostructures. Therefore, an accurate density measurement of thin layers with thicknesses ranging from 10 nm to 100 nm was needed. We used a weight-volumetric method, and obtained the density with three digits accuracy. First, densities of SiO_x layers which were deposited by vacuum evaporation on Si (100) surfaces were measured. The Si surfaces were used because they were well-characterized and smooth. The volume of a layer was obtained as a product of the layer thickness and its area. The area was defined by a window which was located in front of the Si substrate during the deposition. The thickness was obtained by measuring the edge height of stripes made over the entire surface by photolithography process. Dektak (Dektak3030, Nihon Shinku Gijutsu Inc.) was used for the edge height measurement. A weight of the layer was measured by Ultramicrobalance (Sartorius supermicro, Sartorius Inc.). The densities thus obtained were 2.15±0.01g/cm³ and 2.16±0.03g/cm³ for layers with slightly different deposition conditions. Next, a novel method was invented for the density measurement of thin glass layers deposited on polymer substrates. The method made use of the fact that parallel multiple cracks were induced in the glass layer when the film was stretched. The thickness of the glass was obtained by measuring the depths of crack openings. The accuracy of the measurement was also investigated. B. Henry, et. al., presented at the 41st Society of Vacuum Coaters Technical Conference (April 1998). M. Yanaka, Y. Tsukahara, N. Nakaso and N. Takeda, accepted to the J. Mat. Sci., 1998.

TF-MoP26 Accurate and Rapid Determination of Thickness, n and k Spectra, and Resistivity of ITO Films, K. Zhang, EG&G, US; R. Forouhi, I. Bloomer, n&k Technology, US

The transparent conductor, indium tin oxide (ITO), is an important thin film component of flat panel displays. An optimum ITO film should be both highly transparent to visible wavelengths and at the same time, conductive. In practice, however, a trade-off exists between these two attributes, making it difficult to produce a film that simultaneously meets both demands. In order to achieve the optimum balance between these properties, an effective method of characterizing ITO films is necessary. In this talk we will present results of a new measurement technique that simultaneously determines, thickness, the spectra of the refractive index (n) and extinction coefficient (k) from 190 to 1100 nm, and the energy band gap, of ITO films deposited on either transparent or opaque substrates. In addition, we will demonstrate how the film's resistivity can be correlated to the film's extinction coefficient. This technique is based on wide-band spectrophotometry, combined with spectral analysis incorporating the Forouhi-Bloomer dispersion equations for n and k. The measurement technique is non-destructive and takes only a few seconds. A.R. Forouhi and I. Bloomer, Phys. Rev. B, 34, 7018 (1986). A.R. Forouhi and I. Bloomer, Phys. Rev. B, 38, 1865 (1988).

TF-MoP27 Mechanical Properties and Strengthening Mechanisms of Pure Iron Implanted with Metal Ions, D. Yang, University of Alabama; Q. Xue, Lanzhou Institute of Chemical Physics, China

Ion implantation can alternate surface composition and microstructures of metals dramatically, consequent change of physical and chemical properties of ion implanted layers appears as a result of it. The first noticeable effect of implantation is variation of surface residual stress and microhardness. To further understand the effect of metal ion implantation on mechanical properties and strengthening mechanisms, Cr, Mo, W, Ni, Al ion implantation into pure iron was performed on a Metal Vapor Vacuum Arc (MEVVA) source implanter at doses of 5×10¹⁶ ions/cm², 1×10¹⁷ ions/cm² and 3×10¹⁷ ions/cm². The atomic concentration, element distribution, phase structure, surface residual stress and microhardness were measured by Auger Electron Spectroscopy (AES), X-ray Diffractometry and microhardness tester respectively. The relationship among the atomic radius, residual stress and hardness was studied and discussed. It was

shown that the highest atomic concentrations in the implanted layers were between 6 at.% to 38 at.% depending on the elements implanted and implantation doses. The higher and bigger the implantation doses and differences in radius between pure iron and implanted element atoms are, the larger the highest atomic concentration in the implanted layers. In addition, the ion implantation resulted in residual compressive stresses of 663-957 MPa and an increase of 5%-43% in microhardness of pure iron surface. Ion implantation dose was not the only factor influencing surface stress and hardness. The decisive factor was the existing format of ion implanted element, i. e. if the implanted element existed as solid solute the residual compressive stress would be higher and the hardness would be lower; if it existed mainly as compounds then the stresses would be lower and the hardness higher.

TF-MoP28 The Dislocation Network Developed Deep in Titanium Nitride by Ion Implantation, A.J. Perry, A.I.M.S. Marketing, Japan; D.E. Geist, Analytical Reference Materials International; Y.P. Sharkeev, Russian Academy of Sciences, Russia; S.V. Fortuna, Tomsk State University of Architecture & Building, Russia

After treatment by ion implantation, the implanted ions in metallic materials reside in an implanted zone, IZ, extending to a depth of some hundreds of nanometers. Earlier work has shown that the momentum carried by the ions affects the material to far greater depths with a dislocation network extending many microns deep. Dense, thick coatings of titanium nitride can be deposited with low residual stress and low dislocation densities at high temperatures, some 950 C, without the application of substrate bias. Normally used for wear resistance, these coatings are an ideal model system for studying the nature of the IZ and the development of the dislocation network well below it. Studies have been carried out by TEM and glancing incidence XRD. The TEM study shows that implantation leads to the formation of sub-grains within the original grain structure without any grain comminution occurring. A mechanism is then proposed to explain the development of the dense dislocation network below the IZ which is studied by XRD as a function of ion species, acceleration voltage and dose in an extended series of samples. It is found that while any ion bombardment produces a dislocation network, there is a momentum threshold level before a compressive residual stress is developed. This stress is proportional to the ion momentum, reaching values as high as 3-4 GPa, i.e. as high as that found in materials made by PVD methods. Finally we find that there is a significant change in the stress in the substrate below the titanium nitride coating which, surprisingly, can be tensile or compressive and is sensitive to the ion species implanted.

Author Index

Bold page numbers indicate presenter

- A —
Aguilera, S.: TF-MoP11, 2; TF-MoP12, 2
Ang, T.C.: TF-MoP23, **4**
Augustyniak, E.: TF-MoP19, **4**
— B —
Bae, J.W.: TF-MoP17, **3**
Berg, S.: TF-MoP16, **3**
Bloomer, I.: TF-MoP26, **5**
— C —
Castaneda-Pérez, R.: TF-MoP6, **1**
Chan, L.H.: TF-MoP23, **4**
Chang, C.Y.: TF-MoP20, **4**
Chang, T.C.: TF-MoP20, **4**
Chao, B.S.: TF-MoP5, **1**
Chen, M.S.K.: TF-MoP20, **4**
Cho, Y.A.: TF-MoP17, **3**
Choi, D.S.: TF-MoP17, **3**
Chou, M.F.: TF-MoP20, **4**
Chou, S.: TF-MoP20, **4**
Chung, C.H.: TF-MoP8, **2**
Collins, R.W.: TF-MoP3, **1**
Cruz, M.P.: TF-MoP7, **2**
— D —
Deal, P.W.: TF-MoP24, **4**
— F —
Filimonov, S.V.: TF-MoP19, **4**
Forouhi, R.: TF-MoP26, **5**
Fortuna, S.V.: TF-MoP28, **5**
Fukugami, N.: TF-MoP25, **5**
Fundora, A.: TF-MoP11, 2; TF-MoP12, **2**
— G —
Garcia, S.: TF-MoP11, **2**
Garcia-Garcia, E.: TF-MoP5, **1**
Geist, D.E.: TF-MoP28, **5**
Gonzalez-Hernandez, J.: TF-MoP5, **1**
Gregory, R.: TF-MoP24, **4**
Guo, W.: TF-MoP1, **1**
— H —
Hahn, Y.-B.: TF-MoP18, **3**
Han, I.T.: TF-MoP15, **3**
Han, P.G.: TF-MoP21, **4**
He, L.: TF-MoP4, **1**
He, X.J.: TF-MoP1, **1**
Helmersson, U.: TF-MoP9, **2**
Hirata, G.: TF-MoP7, **2**
Hsiao, T.W.: TF-MoP20, **4**
— I —
Ikeyama, M.: TF-MoP9, **2**
— J —
Jeon, J.S.: TF-MoP17, **3**
Jiménez-Sandoval, O.: TF-MoP6, **1**
Jiménez-Sandoval, S.J.: TF-MoP6, **1**
Jin, P.: TF-MoP9, **2**
Jonsson, L.B.: TF-MoP16, **3**
— K —
Kato, M.: TF-MoP14, **3**
Kim, C.: TF-MoP2, **1**
Kim, D.O.: TF-MoP18, **3**
Kim, H.J.: TF-MoP8, **2**
Kim, J.T.: TF-MoP2, **1**
Kim, K.D.: TF-MoP17, **3**
Kim, S.D.: TF-MoP8, **2**
Kim, S.-H.: TF-MoP15, **3**
Kim, T.-G.: TF-MoP15, **3**
Kimata, H.: TF-MoP13, **2**
Kondo, Y.: TF-MoP13, **2**
— L —
Lee, D.C.: TF-MoP2, **1**
Lee, J.C.: TF-MoP3, **1**
Li, S.L.: TF-MoP1, **1**
Lin, J.Y.: TF-MoP20, **4**
Lu, C.: TF-MoP19, **4**
— M —
Macak, K.: TF-MoP9, **2**
Machorro, R.: TF-MoP7, **2**
Maldonado-Alvarez, A.: TF-MoP6, **1**
Márquez-Marín, J.: TF-MoP6, **1**
Martinez-Montes, G.: TF-MoP5, **1**
McGahan, W.A.: TF-MoP22, **4**
Mendoza, A.: TF-MoP5, **1**
Minami, T.: TF-MoP10, **2**
Miyata, T.: TF-MoP10, **2**
— N —
Nahm, K.S.: TF-MoP18, **3**
Nishino, H.: TF-MoP25, **5**
Nyberg, T.: TF-MoP16, **3**
— P —
Park, G.B.: TF-MoP2, **1**
Park, J.Y.: TF-MoP17, **3**
Perry, A.J.: TF-MoP28, **5**
Poon, M.C.: TF-MoP21, **4**
Portelles, J.: TF-MoP11, 2; TF-MoP12, 2; TF-MoP7, **2**
— R —
Rovira, P.I.: TF-MoP3, **1**
— S —
Shao, S.M.: TF-MoP1, **1**
Sharkeev, Y.P.: TF-MoP28, **5**
Siewenie, J.E.: TF-MoP4, **1**
Siqueiros, J.M.: TF-MoP11, **2**; TF-MoP12, **2**; TF-MoP7, **2**
Smith, S.M.: TF-MoP24, **4**
Song, J.S.: TF-MoP8, **2**
Sonoda, T.: TF-MoP14, **3**
Sudijono, J.L.: TF-MoP23, **4**
— T —
Tabet, M.F.: TF-MoP22, **4**
Takayanagi, K.: TF-MoP13, **2**
Tanemura, S.: TF-MoP9, **2**
Tazawa, M.: TF-MoP9, **2**
Tompkins, H.G.: TF-MoP24, **4**
Torres-Delgado, G.: TF-MoP6, **1**
Tse, M.S.: TF-MoP23, **4**
Tsukahara, Y.: TF-MoP25, **5**
Tuan, A.: TF-MoP20, **4**
— V —
Vorobiev, Y.V.: TF-MoP5, **1**
— W —
Wang, N.: TF-MoP21, **4**
Wang, S.: TF-MoP7, **2**
Wang, X.: TF-MoP9, **2**
Wong, H.: TF-MoP21, **4**
— X —
Xi, G.K.: TF-MoP1, **1**
Xiao, M.: TF-MoP11, **2**
Xue, Q.: TF-MoP27, **5**
— Y —
Yamamoto, T.: TF-MoP10, **2**
Yanaka, M.: TF-MoP25, **5**
Yang, D.: TF-MoP27, **5**
Yeom, G.Y.: TF-MoP17, **3**
Yoon, K.H.: TF-MoP8, **2**
— Z —
Zhang, G.H.: TF-MoP1, **1**
Zhang, K.: TF-MoP26, **5**