#### Thin Films Division Room 4C - Session TF-MoP

#### **Poster Session**

# TF-MoP1 Effect of HCl Catalyst in the Formation of Flat Structures of Ta@sub 2@O@sub 5@ Thin Films by Sol-Gel Technique, S. Santucci, C. Cantalini, A.R. Phani, University of L'Aquila, Italy

Stoichiometric Ta@sub 2@O@sub 5@ thin films have been successfully deposited on Si (100) substrates by sol-gel technique using tantalum ethoxide as precursor. The films were annealed at different temperatures. The compositional and structural characteristics of the films were systematically examined with the aid of X-Ray Diffraction, Atomic Force Microscopy and X-ray Photoelectron spectroscopy. We demonstrate that upon using Hydrogen chloride (HCI) as catalyst, we obtained uniform flat like structures of Ta@sub 2@O@sub 5@ as observed in tapping mode atomic force microscopy measurements have shown the small presence of SiO@sub 2@ interface layer in the deposited films. A plausible mechanism to obtain flat structures is also explained.

#### TF-MoP2 Mechanical Properties and Residual Stresses in AlN Films Prepared by Ion Beam Assisted Deposition, Y. Watanabe, N. Kitazawa, Y. Nakamura, National Defense Academy, Japan; C. Li, T. Sekino, K. Niihara, Osaka University, Japan

Aluminum nitride (AIN) thin films were prepared on silicon single crystal substrates by ion-beam assisted deposition method, and the influence of the nitrogen ion beam energy on mechanical properties and residual stresses was studied by changing the nitrogen ion beam energy from 0.1 to 1.5 keV. Mechanical properties were examined by a nano-indentation method and residual stresses were evaluated by film curvature measured by an optical cantilever system. The films show elastic behaviors during loading and unloading processes, but the residual depth after the unloading process increases with increasing the ion beam energy, resulting in decreasing in the returned energy ratio. All the films are found to be in compressive stress state and the values of the stress decrease with increasing the ion beam energy. Decreasing tendency is also observed in the relationship between the ion beam energy and film hardness. So as to study the effect of thermal treatment on relaxation of residual stresses, the films were annealed in nitrogen atmosphere at 723 K, and it is found that the films prepared with the high energy ion beam are relaxed more easily than those prepared with the low energy ion beam. These results suggest that rearrangement of AIN occurs readily in the films prepared with the high energy ion beam. It is proposed that the mechanical properties and residual stresses are closely related with each other and they can be controlled by the ion beam energy.

#### TF-MoP3 The Effects of Temperature and Morphology on the Electron Stimulated Desorption of H@super -@ from Thin Hydrocarbon Films, A.D. Bass, L. Parenteau, F. Weik, L. Sanche, University of Sherbrooke, Canada

In recent years, low energy electron impact techniques have been used to study thin molecular solids under ultra high vacuum (UHV) and have provided valuable information on the electronic interactions that underlie such phenomena as radiation damage to biological materials, dielectric and surface mediated photo-chemistry.@footnote aging 1@ Measurements of the electron stimulated desorption (ESD) of molecular and atomic anions have in part revealed the importance in radiation damage of such processes as dissociative electron attachment (DEA) and dipolar dissociation (DD). In general, the molecular films used in these studies are formed by vapor deposition under UHV conditions. Until very recently,@footnote 2@,@footnote 3@ little consideration was given as to how film preparation and morphology affected ESD measurements and we address questions of this type in this work. We present measurements of the ESD yield of H@super -@ from pure films of n-hexane, ethyl-benzene and benzene as a function of incident electron energy (0 - 20 eV) at various film temperatures between 20 K and their respective sublimation points. The three molecules were chosen as their films display contrasting degrees of porosity, crystallinity etc., Our measurements reveal the variation in the ESD yield from DEA and DD processes as a function of film temperature (and hence morphology). These changes are correlated to others seen in the low energy electron transmission spectra for films of the same molecules deposited at various temperatures. @FootnoteText@ @footnote 1@See for example L. Sanche, IEEE Trans. Dielec. Elec. Insulat. 4, 507 (1997) @footnote 2@ W.C. Simpson M.T. Sieger, T.M. Orlando, L.

Parenteau, K. Nagesha and I. Sanche, J. Chem. Phys. 107 8668 (1997) @footnote 3@K.P. Stevenson, G.A. Kimmel, Z. Dohnalek, R.S. Smith and B.D. Kay, Science 283 1505 (1999).

#### TF-MoP4 Oxidation Studies and Chemical State Analysis of Polycrystalline Magnetron Sputtered (Ti,Al)N Films, A. Kale, S. Seal, S. Sundaram, University of Central Florida

In order to improve the functional properties of hard coatings, recent investigations have been directed to Ti-N based multicomponent materials. In particular the nitride (Ti,Al)N with a Ti:Al ratio of 1:1 seems to be a promising alternative to the widely used TiN. A disadvantage of TiN in hightemperature applications is that it oxidizes rapidly at temperatures above 500 C. In contrast, (Ti,Al)N coatings are characterized not only by high microhardness and dense microstructure, but also by much higher thermal stability. They exhibit better oxidation resistance and hence improved performance over that of TiN. Because of their outstanding properties with respect to hardness, wear resistance, oxidation resistance and corrosion resistance, it seems to be desirable to study the crystal structure and mechanical and chemical properties in detail. The (Ti,Al)N coatings were dc sputter deposited onto 316SS substrates under ambient as well as liquid nitrogen temperatures. The as formed films were oxidized in a vertical fused-silica tube furnace in a pure O2 flowing atmosphere at varying temperatures (700-900C). Both types of films were compared to each other with respect to their mechanical as well as chemical properties. The characterization work involved x-ray diffraction (XRD) to study the amorphous or crystalline nature of the films thus predicting their crystal structure. Scanning electron microscope (SEM) and transmission electron microscope (TEM) images will provide information about the particle size and film thickness. Auger electron spectroscopy (AES) and x-ray photoelectron spectroscopy (XPS) will provide rapid information of elements in the top few atomic layers in addition to the compositional analysis and detailed chemical bonding information. The difference in film stoichiometry will be compared at the two different deposition conditions and will thus reflect their behavior under oxidizing conditions.

#### TF-MoP5 Characterization of VO@sub 2@ Epitaxial Films with Different Orientations Grown on Sapphire (110) by Sputtering, *P. Jin, S. Tanemura,* National Industrial Research Institute of Nagoya, Japan; *K. Macak, U. Helmersson,* Linkoping University, Sweden

Vanadium dioxide is one of the most important compounds in the V-O system. It exhibits a semiconductor-to-metal phase transition at 68°C, accompanied by large changes in electrical resistivity (up to 10@super 5@) and optical properties (from transmitting to reflecting in the infrared). Thin films of vanadium dioxide have been studied for electrical and optical switching devices. It is known that an epitaxial film, which can be grown on sapphire substrate, exhibits properties comparable to single crystal. However, there is little information on the deliberate control of orientation of an epitaxial film and the effect on film property. In this study, thin films vanadium dioxide were grown using DC reactive magnetron sputtering on single crystal 110-oriented sapphire substrates held at 400-600°C. The magnetron source was of the unbalanced type giving rise to more intense bombardment as compared to conventional sources. Different film orientations was achieved with different sputtering geometry, most likely due to varying energetic particle bombardment and growth rate, i.e., an on-axis deposition (substrate facing target) and an off-axis deposition resulted in well-defined (100)- and (001)- oriented epitaxial films, respectively. The epitaxial films were examined by x-ray diffraction using conventional @theta@-2@theta@ scans, rocking curves, and pole figure plots. The compositions were determined from Rutherford backscattering spectroscopy (RBS) and the surface morphologies were studied with atomic force microscopy (AFM). The different film orientations resulted in significant difference in film properties such as the temperature and sharpness of the phase transition, as confirmed from the measurement of electrical resistance vs temperature.

#### **TF-MoP6 Towards a Fully Monitored Fourier Transform Infrared Spectroscopic Ellipsometer**, *J.C. Cigal*, *G.M.W. Kroesen*, Eindhoven University of Technology, The Netherlands

Infrared ellipsometry emerged few years ago as a powerful sensitive and non-intrusive optical technique for characterizing of surfaces, interfaces and thin films. The recent improvements in Fourier transform analysis and of polarizers helped to overcome problems formerly restricting the applications of such a technique. Among the different ellipsometer configurations in use, we opted for a rotating compensator ellipsometer running in the middle infrared. This technique offers several advantages compared to other ellipsometric methods, such as the non-ambiguous

determination of ellipsometric parameters and the insensivity to source and detector polarization. However, the principal fact preventing a widespread application of rotating compensator for spectroscopic purposes was the absence of a good spectroscopic retarder. We are currently developing one available in the 1000-4000 cm@super -1@ spectral range. The principle is based on internal reflection inside a Zinc Selenide crystal. Moreover, measurement speed can significantly be improved by an accurate synchronization between the scanner of the spectrometer and the stepping motors used to rotate the polarizers and the compensator. This will allow us, as a next stage, to perform in-situ and real time measurements.

TF-MoP7 Improved Light Stability of Colored SiO@sub 2@ Coatings Containing Organic and Metalorganic Dye Molecules, *L.L. Diaz-Flores*, Inst. Tecnologico de Saltillo, Mexico; *J.J. Perez-Bueno*, Univ. Autonoma de Queretaro, Mexico; *F.J. Espinoza-Beltran*, *R. Ramirez-Bon*, *Y.V. Vorobiev*, *J. Gonzalez-Hernandez*, CINVESTAV-IPN, Mexico

The sol-gel method has been used to prepare SiO@sub 2@coatings containing various amounts of organic and metalorganic dyes, introduced in the starting solutions. The starting solutions were mixed of tetraethylorthosilicate, water and ethanol. In order to have a better dispersion of the molecular dye into the glass matrix, the starting solutions were subjected to a milling process at various times using an appropriated ball mill. It is observed that in the coatings prepared from solutions without the milling process, the dye is agglomerated into small particles and those prepared from solutions subjected to the milling process show much better dye dispersion. A good dispersion is achieved after about 4 hr of milling, this is reflected in an increase in the optical absorption and makes the samples stable to light exposure. Both results are explained using a model which predicts the degree of dye dispersion. Using this model, an estimation of the size of the dye aggregates is made.

**TF-MoP8 Diamond Formation Using a Low-Pressure Inductively Coupled Plasma**, *H. Ito*, Nagoya Municipal Industrial Research Institute, Japan; *K. Teii*, Nagoya University, Japan; *M. Ito*, Wakayama University, Japan; *M. Hori*, Nagoya University, Japan; *T. Takeo*, Nagoya Municipal Industrial Research Institute, Japan; *T. Goto*, Nagoya University, Japan

Diamond was successfully synthesized by using a H@sub 2@-rich CH@sub 4@/CO/H@sub 2@ and H@sub 2@-rich CH@sub 4@/H@sub 2@ inductively coupled plasma at a low pressure of 11 Pa. The ratio of particle size to deposition time, which is a criterion of the diamond growth rate, in H@sub 2@-rich CH@sub 4@/CO/H@sub 2@ mixture gas plasmas was larger than that in H@sub 2@-rich CH@sub 4@/H@sub 2@ mixture gas plasmas. The nondiamond phases in the deposits increased as CO gas was added to H@sub 2@-rich CH@sub 4@/H@sub 2@ mixture gas as observed by the Raman spectra. In order to investigate the mechanism for the diamond formation, C-atom densities in the plasmas were measured by using a vacuum ultraviolet absorption spectroscopy. In addition CH, OH, Hatom emission intensities were measured by optical emission spectroscopy. As a result, the C-atom densities and OH emission intensity increased with increasing the mixture ratio of CO to CH@sub 4@, while CH and H-atom emission intensities were almost constant. The generation of C-atoms from CO gases was much larger than that from CH@sub 4@. On the basis of the correlation between the quality of deposits and the C-atom densities in the plasma, C-atoms were found to contribute to form amorphous carbon phases rather than diamond phase. Moreover, using the low-pressure inductively coupled diamond formation plasmas, the importance of the abstraction of diamond surface-bonded H-atoms by OH radicals was suggested.

#### **TF-MoP9 Microstructure of Ti:D Films Prepared by Reactively rf Sputtering, S. Nakao**, P. Jin, K Saitoh, Y. Miyagawa, S. Miyagawa, National Industrial Research Institute of Nagoya, Japan

Metal deuteride films have attracted much attention because of potential application for neutron source in ion beam technology. Especially, titanium deuteride (Ti:D) films are much stable at relatively high temperature up to about 400 @super o@C. In this study, Ti:D films were prepared by reactively rf sputtering and the microstructure was examined. Ti:D films were deposited mainly on Si substrates under the various conditions of the rf power and the ratio of Ar and D@sub 2@ gases. Thin film x-ray diffraction (XRD) measurements were carried out to examine the crystal structure of the films. Rutherford backscattering spectrometry (RBS) and elastic recoil detection analysis (ERDA) were performed with a 1.7 MV tandem-type ion accelerator to analyze the composition of the films. From the results of the XRD measurements, it was found that the Ti:D films have a delta-phase (fluorite) crystal structure at low rf power of 100 W.

However, the results of RBS and ERDA measurements revealed that the Ti:D films contained the impurity elements such as hydrogen and oxygen. It was inferred from the quantitative analysis that the films were composed of delta-phase titanium deuterides (or hydrides) and amorphous titanium oxides.

#### TF-MoP10 Growth of Si Thin Films on CeO@sub 2@/Si(111) Substrate Prepared by Electron Beam Evaporation, C.G. Kim, J.H. Yang, B.S. Moon, C.Y. Park, Sung Kyun Kwan University, Korea

The Si/CeO@sub 2@/Si structure is one of the silicon-on-insulator (SOI) and was prepared by a hetero-epitaxially growing method. Cerium dioxide(CeO@sub 2@) is an insulating material with a lattice mismatch of 0.35% to silicon. Si film was grown on CeO@sub 2@(111)/Si(111) substrate by using high-vacuum evaporation. We have studied on the growth mechanism of Si for the various deposition conditions and analyzed by X-ray diffraction, double crystal XRD, TEM, AFM and the mobility measurement. For homo-epitaxial growth of Si, a better epitaxial Si film had been formed 800@super o@C. But, for the Si epitaxial growth on the CeO@sub 2@(111)/Si(111) the substrate temperature was limited to about 620@super o@, because a dissociated oxygen from CeO@sub 2@ and an out-diffused carbon. The Si film was epitaxially growth along (111) direction of the CeO@sub 2@ at 620@super o@C and consisted of domains oriented along (111) direction. The mobility was 56.4cm@super 2@/(Vs) at carrier density of 5.87x10@super 19@/cm@super 3@.

### TF-MoP11 Origin of Electrical Property Distribution on Surface of ZnO:Al Films Prepared by Magnetron Sputtering, *T. Minami*, *T. Miyata*, *T. Yamamoto*, *T. Nishitani*, Kanazawa Institute of Technology, Japan

This paper investigates the origin of electrical property distribution on the substrate surface of ZnO:Al films prepared by magnetron sputtering on substrates placed parallel to the target surface. The films were prepared using a magnetron sputtering apparatus with a sintered or powder target (diameter of 140mm) and either a dc or rf plasma power source. When the films were prepared on substrates at the same temperature with the same deposition rate under optimized sputter deposition and target preparation conditions, the lowest obtained resistivity found in ZnO:Al films prepared by either dc or rf magnetron sputtering was roughly the same. However, the ZnO:Al films prepared by dc sputtering exhibited a larger increase of resistivity at locations on the substrate corresponding to the target erosion area than found in films prepared by rf sputtering. In contrast, the resistivity distribution of ZnO:Al films prepared by rf magnetron sputtering with an applied external magnetic field which focused the rf plasma was similar to that of films prepared by dc magnetron sputtering. Thus, the difference in electrical property distribution obtained between rf and dc magnetron sputtering is mainly related to the activity and amount of oxygen reaching the substrate surface as well as its spatial distribution.

#### TF-MoP12 Effect of C@sub 2@ Radicals on Diamond Growth Using Low-Pressure, Radio Frequency, CH@sub 3@OH/H@sub 2@/H@sub 2@O Inductively Coupled Plasma, T. Shiomi, H. Nagai, M. Hiramatsu, M. Nawata, Meijo University, Japan

Previously we demonstrated the successful formation of diamond crystals using a low-pressure, radio frequency (rf, 13.56 MHz), inductively coupled plasma (ICP) in the total pressure range of 9.3-18.6 Pa.@footnote 1@ In contrast with conventional methods of diamond chemical vapor deposition (CVD) employing high-pressure plasma (@>=@100 Pa), in the case of lowpressure, high-density, and highly dissociated plasmas, carbon dimer (C@sub 2@) radicals or carbon atoms instead of methyl radicals might be major species for film formation. C@sub 2@ radical is considered to be one of important radicals for the nanocrystalline diamond deposition using plasma-enhanced CVD.@footnote 2@ In this work, C@sub 2@ radical density in a low-pressure (@<=@13 Pa), rf-ICP employing CH@sub 3@OH/H@sub 2@/H@sub 2@O source for diamond CVD was measured using absorption spectroscopy with Xe lamp emitting a continuous spectrum as a light source. The correlation between the absolute C@sub 2@ radical density and the quality of diamond films was investigated. In the Raman spectra of diamond formed using a low-pressure rf-ICP, a broad peak around 1140 cm@super -1@ arising from nanocrystalline diamond was observed together with the strong 1332 cm@super -1@ diamond Raman peak. C@sub 2@ radical density increased almost linearly with increasing rf input power or CH@sub 3@OH partial pressure. On the other hand, C@sub 2@ radical density decreased with increasing H@sub 2@O partial pressure, while the intensity of 1332 cm@super -1@ diamond Raman peak increased with increase of the H@sub 2@O partial pressure up to 5.3 Pa. @FootnoteText@@footnote 1@H. Noda, H. Nagai, M. Shimakura, M. Hiramatsu, and M. Nawata, J. Vac. Sci. Technol. A 16, 3170

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TF-MoP13 Properties of Indium Oxide Thin Films Prepared by Reactive Electron Beam Evaporation Technique for EMI Control, J. Asbalter, A. Subrahmanyam, Indian Institute of Technology, India

It is well known that transparent conductors like Indium oxide (IO) are good Electro Magnetic Interference (EMI) shields. In the present investigation, the EMI shielding property of indium oxide thin films has been studied as a function of growth temperature. All the IO films are grown on glass substrates by reactive electron beam evaporation technique at a chamber pressure (with the reactive gas, oxygen) of 2.5 x 10@super -4@ milli bar. The substrate temperature is varied between 160@super o@C -200@super o@C. All the films show metallic properties (carrier concentration, N = 10@super 19@/cc, Hall Mobility =20-30 cm@super 2@ V@super -1@ sec@super -1@) with an optical transmission above 85% (at 500 nm wavelength). It is found that the EMI shielding efficiency (SE) of IO films (of 100 nm thickness) is very much comparable to that of the silver coated metal sheet in the measured frequency range 1 MHz till 100 MHz . As is well known that high frequecny shielding is related to the plasma frequency and the low frequency shielding is dependant on the magnetic properties. The AC (magnetic) susceptibility of the films measured in the temperature range 300 K till 6 K show very interesting magnetic properties. All the films show diamagnetic behavior at room temperature (300 K). For the films prepared at 200@super o@C, there is a clear paramagnetic behavior below 250 K. Present work analyses the reasons for the paramagnetic nature and its consequence on the low frequency (< 10 MHz) shielding efficiency.

#### TF-MoP14 Diffusion of Cu from PVD Al-Cu Alloy and CVD Cu Thin Films into CVD Al Thin Films Inside Submicron Via Holes, *B. Rogers*, Vanderbilt University

Solid state diffusion of Cu from copper containing films into a chemical vapor deposited (CVD) Al film was evaluated as a method to dope the CVD Al with Cu atoms to enhance its electromigration resistance. CVD Cu and PVD Al-1.5 wt% Cu thin films were used as the copper sources. Thin film stacks consisting of CVD Al/CVD Cu and PVD Al-Cu/CVD Al were deposited onto unpatterned silicon dioxide films as well as silicon dioxide films patterned with 0.6-micron diameter by 1.2-micron deep via holes. Samples were annealed for 0, 5, 15, or 60 minutes at 360, 390, or 420 degrees Celsius. Backscatter electron microscopy was used to image theta phase (Al@sub 2@Cu) precipitates inside vias. The number of precipitates and fraction of via cross-sectional area covered by the precipitates were used to estimate the amount of Cu present in the vias. These results are compared to Rutherford backscatter spectrometry and Auger electron depth profiling analyses of samples with films deposited onto unpatterned substrates.

#### TF-MoP15 Process Control and Properties of Aluminum Doped Zinc Oxide Films Deposited by High Rate Mid-frequency Reactive Magnetron Sputtering, N. Malkomes, M. Vergöhl, B. Szyszka, T. Matthée, Fraunhofer Institute for Surface Engineering and Thin Films, Germany

Aluminum doped Zinc oxide films are promising candidates for economic TCO applications. To reach high deposition rates (about 7 nm/s at 4.5 W/cm2) in combination with optimum TCO properties by reactive midfrequency (MF) sputter technique, the process window has to be precisely controlled. In order to overcome the typical hysteresis problem the process stabilization was done by plasma impedance control for ease of use, enabling to stabilize the deposition process in any working point on the scurve of the corresponding hysteresis loop. In addition the setpoints were characterized by partial pressure measurements, optical emission spectroscopy (OES), and with in-situ spectroscopic ellipsometry. The influence of deposition parameters (working point, pressure, temperature) on the electrical and optical properties as well as film growth and morphology were studied by photometry, Hall-measurements and in-situ and ex-situ spectroellipsometry, respectively. Due to the bandgap widening the optimum films show neutral color. The ellipsometric spectra could be well modelled without using interface layers indicating the dense structure of the films. Electrical measurement yield that the optimum resistivity of ZnO:Al films deposited on unheated substrates is about 2.5 times higher than at 200°C substrate temperature. In the latter case, a value of 290µOhm cm could be reached. In addition the process window of stoichiometric films is widened due to oxygen partial pressure limited forming of the film on the heated substrate. .

TF-MoP16 Low-temperature Growth of Ti(C,N) Thin Films on D2 Steel and Si(100) Substrates by PEMOCVD, B.-C. Kang, J.-H. Boo, Y.K. Cho, J.-G. Han, C.H. Heo, SungKyunKwan University, Korea

We have deposited Ti(C,N) thin films on Si(100) and D2 steel substrates in the temperature range of 150 - 300 @super o@C using tetrakis diethylamido titanium (TDEAT) and titanium isopropoxide (TIP) by pulsed DC plasma enhanced metal-organic chemical vapor deposition (PEMOCVD) method. Polycrystalline Ti(C,N) thin films were successfully grown on either D2 steel or Si(100) surfaces at temperature as low as 150 @super o@C. Compositions of the as-grown films were determined with XPS and RBS. From XPS analysis, thin films of Ti(C,N) with low oxygen concentration were obtained. RBS data were also confirmed the changes of stoichiometry and microhardness of our films. Radical formation and ionization behaviors in plasma are analyzed by optical emission spectroscopy (OES) at various pulsed bias and gases conditions. H@sub 2@ and He+H@sub 2@ gases are used as carrier gases to compare plasma parameter and the effect of N@sub 2@ and NH@sub 3@ gases as reactive gas is also evaluated in reduction of C content of the films. In this study, we found that He and H@sub 2@ mixture gas is very effective in enhancing ionization of radicals, especially N@sub 2@ resulting is high hardness. The higher hardness of film is obtained to be ca. 1700 HK 0.01 but it depends on gas species and bias voltage. The proper process is evident for H@sub 2@ and N@sub 2@ gas atmosphere and bias voltage of 600 V. However, NH@sub 3@ gas highly reduces formation of CN radical, thereby decreasing C content of Ti(C,N) thin films in a great deal. Compared to PVD TiN films, the Ti(C,N) film grown by PEMOCVD has very good conformability; the step coverage exceeds 85% with an aspect ratio of more than 3.

#### TF-MoP17 A Study on the Characteristics of TiN Thin Film Deposited by Atomic Layer Chemical Vapor Deposition Method, *H. Jeon, J.W. Lee, J.H. Koo, Y.S. Kim, Y.D. Kim, D.S. Kim,* Hanyang University, Korea

A TiN film which exhibits a NaCl structure is now used as a diffusion barrier in ULSI device because it shows a very low resistivity, good adhesion characteristics and thermal stability.@footnote 1@ In this study, we deposited TiN film on Si substrate by using atomic layer chemical vapor deposition system.@footnote 2@ The TiN film deposited by this method is expected to have excellent physical and electrical properties.@footnote 3@ In this system, the TiCl@sub 4@ and NH@sub 3@ gases as Ti source and an reactant were supplied, separately and Ar purge gas was added between each source and reactant supply to suppress the direct reaction between source and reactant. The process parameters to grow TiN were process temperature, number of cycle to supply the reactant and source gases, source supplying time, and purging time. After growing this TiN film, the physical and electrical properties were measured by XRD, AFM, SEM, AES, TEM, RBS and a four point probe. The crystallinity and the surface and interface were analyzed by XRD, SEM and TEM. The root mean square toughness of TiN surface was measured by AFM and its value was about 15Å. The chemical analysis was done by AES and the Cl content in TiN film was below the detection limit of Auger Electron Spectroscopy which was below 1%. We will compare these TiN thin film data with other deposition method, such as PECVD and MOCVD and will discuss the TiN film growing method based on the thermodynamic consideration and atomic size computer modeling. @FootnoteText@ @footnote 1@J. E. Sundgren, Thin Solid Films, 128, 21-44 (1985) @footnote 2@S. Yokoyama, H. Goto, T. Miyamoto, N. Ikeda, K. Shibahara, Applied Surface Science, 112, 75-81 (1997) @footnote 3@T. Suntola, Thin Solid Films, 216, 84-89 (1992).

## **TF-MoP18 Non-Stoichiometric PMN-PT Films Grown by Laser Ablation**, *A. Fundora*, Universidad de la Habana, Cuba; *J.M. Siqueiros*, UNAM, Mexico; *J. Portelles*, Universidad de la Habana, Cuba

Films of Pb(Mg@sub 1/3@Nb@sub2/3@)@sub2.1@ Ti@sub0.3O3@ (PMN-PT) have been grown on Pt/SiO@sub2@/Si substrates by pulsed laser ablation. The dielectric and microstructural properties of the non stoichiometric thin films of the type: 2.1PMN-0.3PT are studied in this work. The nature of the ferroelectric layer-electrode interface is analyzed by transmission electron microscopy (TEM) as well as the effect of its characteristics in the performance of the multilayer system. Surface structure and cross section studies were performed by scanning electron microscopy (SEM). Curves of dielectric permittivity as a function of temperature and hysteresis loops are reported.

TF-MoP19 The Advancing Techniques and Sputtering Effects of Oxide Films Fabricated by Stationary Plasma Thruster with Argon and Oxygen Gases, J. Cho, KIST, Korea; Y. Ermakov, Mirea, Russia; K.H. Yoon, Yonsei University, Korea; S.K. Koh, KIST, Korea

The using of stationary plasma thruster (SPT) ion source, invented previously for space application in Russia , in experiments with surface modifications and film deposition systems is reported here. Plasma in the SPT is formed and accelerated in electric discharge taking place in the crossed axial electric and radial magnetic fields. Brief description of the construction of specific model of SPT used in the experiments is presented. With gas flow rate 39 ml/min, ion current distributions at several distances from the source are obtained. These was equal 1-3 mA/cm2 within an ionbeam ejection angle of ï,± 200 with discharge voltage 160 V, for Ar as a working gas. Such an extremely high ion current density allows us to obtain the  $\hat{a} \in \{$  films with deposition rate  $\hat{a} \in \{$  by sputtering  $\hat{a} \in \{$  target. It is shown a possibility of using of reactive gases in SPT (O2 and N2) along with high purity inert gases used for cathode to prevent the latter contamination. It is shown the SPT can be operated at the discharge and accelerating voltages up to 600 V. The results of presented experiments show high promises of the SPT in sputtering and surface modification systems for deposition of oxide thin films on Si or polymer substrates for semiconductor devices, optical coatings and metal corrosion barrier layers. Also, we have been tried to establish in application of the modeling expertise gained in electric and ionic propulsion to permit numerical simulation of additional processing systems. In this mechanism, it will be compared with conventional DC sputtering for film microstructure, chemical composition and crystallographic considerations.

#### TF-MoP20 An Alternative Procedure for the Deposition of Close-Spaced Sublimation CdTe/CdS Solar Cells, H.R. Moutinho, R.G. Dhere, M.M. Al-Jassim, C. Ballif, L.L. Kazmerski, National Renewable Energy Laboratory

In previous work (JVST 1998 and 1999), we demonstrated that a recrystallization process causes the changes in physical properties of CdTe films heat-treated with CdCl@sub 2@. Using this information, we were able to induce recrystallization in close-spaced sublimation (CSS) CdTe films, depositing these films at temperatures about 200°C lower than usually used in this process. This lower-temperature deposition process is very attractive in the fabrication of solar cells because it implies in energy economy, and also avoids or minimizes the problem with diffusion of impurities from the glass substrate to the active elements in the cell. In the present work, we deposited CSS CdTe films on solar-cell substrate structures using relatively low temperatures and varied deposition parameters, (e.g., deposition temperature and growth rate). We also subjected the films to two different treatments (dipping in CdCl@sub 2@/methanol solution and exposure to CdCl@sub 2@ vapor), and varied many parameters, such as treatment temperature and time, and saturation of the solution. The objective was to optimize the deposition and heattreatment parameters to obtain high efficiency cells. The structure of the CdTe films was studied using atomic force microscopy, to obtain information on average grain size and surface topography; X-rays diffraction, to obtain information on lattice parameter and phase formation; and X-ray Photoelectron Spectroscopy, to study film composition. We correlated the various deposition and treatment parameters with the performance parameters (quantum efficiency, opencircuit voltage, short-circuit current, fill factor, and efficiency) of completed devices. We showed that, although the solar cells fabricated at higher temperatures still provide the best efficiencies, the low temperature method can produce solar cells with intermediate efficiencies (>10%), which will be more attractive for industrial application, because of the manufacturing and economic advantages.

#### TF-MoP21 CdS/CdTe Interface Analysis by Transmission Electron Microscopy, R.G. Dhere, M.M. Al-Jassim, K.M. Jones, H.R. Moutinho, T.A. Gessert, L.L. Kazmerski, National Renewable Energy Laboratory

CdTe-based polycrystalline solar cells are leading candidates for terrestrial photovoltaic applications. High efficiency devices have been obtained despite large lattice mismatch between hexagonal CdS and cubic CdTe. Best CdTe based devices have been made with CdS/CdTe structure. Knowledge of the properties of the CdS/CdTe interface is critical to improve the understanding of the device as this interface lies close to the active junction in the device. In the present work, CdS was deposited by chemical bath deposition on Si substrates and CdTe was deposited by close spaced sublimation. Si substrates were used to facilitate the preparation of thin cross-sectional specimens for TEM analysis. The chemical nature of the CdS/CdTe interface, structural properties, and their dependence on the fabrication parameters e.g. substrate temperature (475-600°C) and post-

deposition CdCl@sub 2@ heat treatment were analyzed. In addition, the effects of the interface structural defects on the crystallinity of CdS, prior to CdTe deposition, were examined. Small spot energy dispersive spectroscopy (EDS) of the interface revealed a considerable amount of sulfur in CdTe. The concentration of sulfur, in general, was higher in the grains with higher density of structural defects and at the grain boundaries. Planar defect density in CdTe films increased with substrate temperature while the threading dislocation density decreased. Interface analysis showed that the majority of the crystalline defects in the CdTe films, deposited on CdS, were generated at the interface. The crystallinity of CdS did not have major influence on the interface defect generation.

#### **TF-MoP22 In-Plane Texturing in Evaporated Cr Films,** *J.F. Whitacre,* University of Michigan; *Z.U. Rek,* Stanford Synchrotron Radiation Laboratory; *J.C. Bilello, S.M. Yalisove,* University of Michigan

The evolution of crystallographic texture in Cr films thermally evaporated using no energetic assistance was examined. In particular, the existence of an in-plane texture in films deposited onto obliquely oriented substrates was studied. All films were grown using electron-beam evaporation in a UHV chamber on (100) test-grade Si wafers with native oxide. The substrates were positioned such that their surface normals were oriented either 0° or 60° with respect to the adatom flux vector. Texturing, grain development, and surface morphology were studied using x-ray and electron diffraction, transmission electron microscopy (TEM), and scanning electron microscopy (SEM). The films grown on substrates oriented perpendicular to the flux vector developed a strong (110) out-of-plane texture, but showed no signs of in-plane texturing. These films had welldefined crystalline columnar grain structures and faceted surface morphologies. Those films grown on obliquely oriented substrates also displayed columnar grain structures and surface facets, though they were inclined ~35° with respect to the substrate surface normal. Despite this tilt, the out-of-plane texture was still (110). A heuristic model is proposed which describes the evolution of in-plane texture in evaporated films and accounts for the morphology and grain development observed. The combination of obliquely arriving adatoms and anisotropic surface facets creates an in-plane shadowing phenomena. If surface diffusion lengths are limited to grain dimensions, the model shows how grains with particular inplane crystallographic orientations will grow at the expense of others. This process is modeled numerically and compared with experimental results. Work supported under ARO Army contracts DAAH 04-95-1-0120 and DAAG 55-98-1-0382. Some data collected at SSRL, funded by the US DoE.

## TF-MoP23 Sputter Deposition of Ni Thin Films For Nickel Silicide Metallization, *H. Zhang*, Tosoh SMD, Inc.

NiSi is considered as one of the candidates to replace TiSi@sub 2@ contact in deep sub-miron metallization due to its low resistivity and lower formation temperature. Sputter deposition of Ni thin film is one of the crucial steps in nickel salicide (self-aligned silicide) process. One problem associated with sputter deposition of Ni is that Ni is a ferromagnetic material and is difficult to sputter. A Ni sputtering target results in low magnetic flux intensity in front of the target because the target shunts a considerable percentage of magnetic flux from system magnets. High magnetic flux density can be obtained by using a high pass-through flux (PTF) Ni target that allows maximum magnetic flux permeate through a target. In this study, the effects of target PTF and sputtering process parameters such as Ar pressure, sputtering power and substrate temperature on sputter process were studied. Ni targets with the PTF% of 40% (high PTF) and 30% (low PTF) were tested. Ni thin films were deposited on 200 mm (100) Si wafers. Sputter deposition rate, I-V characteristics, film sheet resistance and film uniformity were measured under various sputter conditions. The high PTF target resulted in low sputtering impedance and better Rs film uniformity. Rapid thermal processing (RTP) was carried out to form nickel silicides at temperatures between 300C to 900C for various times. Phases and microstructure of the films were characterized. The sheet resistance decreased significantly after annealing at 400C to 600C due to formation of NiSi. The significant increase in sheet resistance after annealing above 700C was attributed to formation of NiSi@sub 2@ phase.

#### TF-MoP24 Synthesis of Highly Oriented Piezoelectric AlN Films by Reactive Sputter Deposition, F. Engelmark, G. Fuentes, I.V. Katardjiev, A. Harsta, U. Smith, S. Berg, Uppsala University, Sweden

Nucleation and growth of polycrystalline AIN films on thermal and CVD oxide have been studied during RF reactive sputter deposition. The influence of the growth conditions, namely deposition pressure, RF power, Ar/N@sub 2@ ratio, substrate temperature, on film properties has been systematically studied. The properties of interest are crystallinity, degree of

orientation, crystallite size, surface roughness, stress, piezoelectric coupling, acoustic velocity and others. The films have been analyzed with RBS, ESCA, XRD, ellipsometry, SEM, AFM, stress measurements, etc. It is found that these properties are sensitive functions of all deposition parameters and that there exist optimal deposition conditions under which films of high quality are obtained. The films at optimal conditions were analyzed with the following results: FWHM XRD 0.216 deg, FWHM rocking curve 1.62 deg, crystallite size 38 nm, optical index 2.15, surface roughness 31 Angstroms, stress 400 MPa. Further, to study the electro-acoustic properties of the films surface acoustic wave (SAW) filters were fabricated operating at 534 MHz. The thin film structure consists of AlN/SiO@sub 2@/Si. The electrodes of the interdigital transducers were made of Al. Examination of the frequency response indicated an acoustic velocity of 4900 m/s and a moderate coupling coefficient.

#### TF-MoP25 Surface Morphology Analysis in Correlation with Crystallinity of CeO@sub 2@(110) Layers on Si(100) Substrates, *T. Inoue*, *T. Nakamura*, *S. Nihei*, Iwaki Meisei University, Japan; *Y. Yamamoto*, Hosei University, Japan

In the course of the study on epitaxial growth of CeO@sub 2@ layers on Si(100) substrates, it is found that the layer has (110) orientation and requires substrate temperature above 820°C. Recently, we have succeeded in lowering growth temperature by more than 100°C by using newly developed "electron beam assisted evaporation". In general, epitaxial growth needs enough migration energy for adsorbed atoms and/or molecules. In the vicinity of the critical condition for epitaxial growth, CeO@sub 2@ layers having various crystallinity are obtained depending on growth conditions such as growth temperature, pre-treatment of the Si surface, contents of residual gas in the vacuum atmosphere and so on. It is very important to understand the growth mechanism, which rules crystallinity of the layer. We will present surface morphology analysis by atomic force microscopy (AFM) in correlation with crystallinity of the layers determined by reflection high energy electron diffraction (RHEED). It is clearly observed that surface morphology changes with crystallinity of the CeO@sub 2@ layers. Single crystal samples show a nanometer-scaleperiodically corrugated sturcture, which consists of (111)-facets. On the other hand, the surface of poly-crystalline samples with a strong tendency of orientation consists of tetrahedral hillocks with irregular-rotationalorientations within the horizontal plane. Samples with a ring RHHED pattern show a very finely grained surface. These features clearly reflect the difference in the growth mechanism, especially at the early stage of the growth. Results on quantitative analysis of AFM data will be given.

### TF-MoP26 Oxide Thin Films for Electroluminescent Phosphors, J.S. Lewis, P.H. Holloway, University of Florida

The use of oxide phosphors for thin-film electroluminescent (TFEL) displays has been investigated. Thin films of Zn@sub 2@GeO@sub 4@:Mn were deposited by RF magnetron sputtering from powder targets. The devices exhibited a brightness value of 85 cd/m@super 2@ versus 100 cd/m@super 2@ for the traditional ZnS:TbOF, another green emitting phosphor. The Zn@sub 2@GeO@sub 4@:Mn devices emit in the green with CIE color coordinates x = 0.30 and y = 0.66, which is a more saturated green than ZnS:TbOF. These data show that the performance of oxide phosphors rival that of the traditional sulfide based phosphors. Introduction of surface roughness and further optimization of processing should improve the values even further. Data from blue-emitting oxides will also be presented, as will injection layer schemes which should improve charge injection and lead to lower threshold voltages.

#### TF-MoP27 XPS and AES Investigation on the Oxidation Resistance of Plasma-treated Copper Leadframe, A. Wong, Nanyang Technological University, Singapore; R.G. Krishnan, Institute of Microelectronics, Singapore; G. Sarkar, Nanyang Technological University, Singapore

Copper is widely used as a material in the microelectronic plastic packaging and IC interconnect applications. The oxidation of copper to form oxide due to microelectronic processing can result in poor copper metal to epoxy mold compound (Cu/EMC) bonding causing package delamination which compromises package reliability. Besides, metallization material problems such as increment in signal transmission delays, decrease power dissipation and decrease reliability to electrical and thermal stress migration failures can occur. Plasma treatment of copper is believed to be able to enhance its oxidation resistance. Based on the x-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES) results, it was possible to confirm firstly, the successful incorporation of C and N into a copper film above the copper substrate using plasma treatment and secondly, the change in oxidation resistance of this film as a result of the treatment. Thickness measurements from AES depth profiles also confirmed the dominant influence the film thickness have on the enhancement of oxidation resistance in the film as compared to a small change in film stoichiometry. XPS and AES analyses show that once the film was heated at 250@super o@C, the N/Cu (atomic concentration) ratio of the film decreased from 1.074 to 0.635. Heating at 300@super o@C cause further decaying of the N/Cu ratio to 0 which is confirmed by the disappearance of the XPS N 1s signal. There is no difference in the binding energy of the XPS Cu 2p3/2 signal of the pristine copper and the treated copper. The oxidation of pristine copper at 250  $^\circ\text{C}$  can be seen from the shift of the XPS Cu 2p3/2 binding energy at 932.70 eV to 933.60 eV and an increasing Full-width-halfmaxima (FWHM) from 1.00 eV to 3.00 eV. However, these are not observed for the oxidation-resistant film at 250 °C as there is a delay in the oxidation onset temperature (shown by the shift in binding energy and increase in FWHM of the XPS Cu 2p3/2 spectra) by at least 50 °C, which depends on the film thickness. The surface analysis data and the oxidation resistance of the film are finally correlated and discussed.

#### **TF-MoP29** Optimization of the Reflectivity of Magnetron Sputter Deposited Silver Films, *M. Vergöhl, N. Malkomes, B. Szyszka, F. Neumann, T. Matthée,* Fraunhofer Institute for Surface Engineering and Thin Films, Germany; *G. Bräuer,* Leybold Systems GmbH, Germany

Silver films were deposited by means of dc and mid-frequency (MF) magnetron sputter deposition on floatglass in order to achieve a maximum reflectivity over the entire visual and infrared spectral range. The films were investigated by means of ex-situ and in-situ spectroscopic ellipsometry, reflectivity, conductivity measurements, and atomic force microscopy (AFM). The following deposition parameters were varied: midfrequency and dc-technique, power density, sputtering pressure, substrate temperature, sputtering gas (Ar, Kr, Ne), nitrogen and oxygen residual gas, and film thickness. With the aid of in-situ spectroscopic ellipsometry, it can be shown that for certain process parameters an optimum layer thickness exists for achieving a maximum reflectivity in the visual spectral range. With increasing thickness, optical losses come into play which are due to the formation of larger grains. This optimum layer thickness is smaller in the films deposited in the MF mode, indicating a smoother surface and smaller grain size compared to the DC mode. As an optimum value, a reflectivity of R=99.3% is achieved. The resistivity of this film was found to be @rho@=2.4 µ@Omega@ cm. The measured reflectivity is close to the theoretical value, which was determined from the Drude-Lorentz fit parameters with respect to the resistivity. The measured reflectivity of the sputter deposited films will be compared to thin films which are prepared by e-beam evaporation.

#### TF-MoP30 Preparation and Characterization of RF-sputtered SrTiO@sub 3@ Thin Films, K. Radhakrishnan, C.L. Tan, H.Q. Zheng, G.I. Ng, Nanyang Technological University, Singapore

Strontium titanate, SrTiO@sub 3@ (STO) material has found wide applications ranging from capacitor dielectrics in ICs to semiconductor memories and submicron ICs. This paper focuses on synthesis of STO thin films on Si and GaAs substrates under various growth conditions such as oxygen partial pressure and substrate temperature, and post annealing conditions. STO films were deposited by RF-magnetron sputtering in Ar/O@sub 2@ plasma. The substrate temperature was 200 to 300°C. Platinum was used as electrodes. The dielectric constant increased with increase in O@sub 2@ partial pressure during sputtering. However, it showed a decreasing trend when the partial pressure ratio, O@sub 2@/Arwas >1. The dielectric constant measured for these samples is low (14 to 22). X-ray diffraction measurements (XRD) showed peaks mainly due to substrate with weaker peaks corresponding to STO phase. The STO samples were annealed to study the effect of annealing on dielectric constant. The films were annealed for 1 hr under O@sub 2@ flow. It was observed that the dielectric constant was around 20 when the films were annealed below 500°C. However, when the temperature was above 500°C the dielectric constant value increased five times. A high value of 125 was measured for the film annealed at 600oC. The increase in the dielectric constant was due to the development of stable STO phase when annealed under O@sub 2@. Films annealed above 500°C showed intense XRD peaks corresponding to STO phase for (110), (200) and (211). The effect of film thickness on the dielectric constant was determined using 100 to 425nm thick samples. The dielectric constant increased from 117 to a high value of 145 when the thickness was 425nm. The average breakdown voltage measured capacitors with STO film thickness of 110nm was found to be 855kV/cm.

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