

# Tuesday Evening Poster Sessions, November 4, 2003

## Manufacturing Science and Technology

### Room Hall A-C - Session MS-TuP

#### Poster Session

##### **MS-TuP1 Perfluoroelastomer Sealing Performance in Plasma Environments, S. Wang, J.M. Legare, DuPont Dow Elastomers, L.L.C.**

Perfluoroelastomers (FFKM, e.g. Kalrez, etc.) are widely used as seals on semiconductor wafer processing equipment where plasma technology is applied. These processes include etching, ashing and plasma enhanced chemical vapor deposition. The seals need to exhibit good plasma resistance in order to withstand chemical attack and maintain sealing functionality. The seals must contribute minimal contamination to the process, hence particle generation and metallic contamination are of major concerns when selecting sealing materials. This paper discusses the interactions of plasma with sealing materials, and the test methods used in the evaluation, and then compares the performance of various FFKM's with varying compositions. The results indicate that conventional FFKM products, which contain carbon black or mineral fillers, are not suitable for plasma applications where both etch rate/weight loss and particle generation are critical. Unfilled FFKM's, or FFKM's with fillers that react with plasma to form volatiles, are better suited for sealing applications on semiconductor wafer processing equipment where reactive plasmas are used.

##### **MS-TuP2 A Novel Power Supply with Arc Handling for High Peak Power Magnetron Sputtering, D.J. Christie, F. Tomasel, W.D. Sproul, D.C. Carter, Advanced Energy Industries, Inc.**

The potential of high peak power magnetron sputtering has created growing interest, because it can generate a dense plasma with high target material ion content. At the required power densities, process arcs are not avoidable. Unless properly handled, arcs generate macro-particles and target damage, limiting the usefulness of the technique. However, coatings quite suitable for industrial applications may be applied if the pulsed supply incorporates arc handling. We have created such a power supply, capable of peak powers up to 3 mega-Watts and peak currents to 3000 A, at discharge voltages reaching 2 kV. It has voltage ring-up capability for pulse by pulse plasma ignition. This new power supply technology enables the practical application of a whole new range of sputtering processes, based on pulsing magnetrons to high peak powers.

##### **MS-TuP3 GaAs Recycling using Supercritical Fluid SCFCO@sub2@ with O@sub2@ and H@sub2@O, T. Momose, O. Otomo, Miyagi National College of Technology, Japan**

Contamination with valuable and poisonous metals such as GaAs dust produced in semiconductor technology must be controlled not to contaminate our life cycles. Most of the metals are embedded underground sealed by concrete, but not complete. Therefore, supercritical fluid (SCF) carbon dioxide (CO@sub2@) with high solubility for organic compounds was employed for simpler recycling for metals. @footnote 1@ Samples were GaAs(1:99) grains obtained from the wall of the sputtering apparatus. They(0.5 g) were kept in SCFCO@sub2@ with H@sub2@O of 4 c.c. for 30 min at a constant temperature and pressure. The treated water was analyzed using ICP (Vop-MarkII, Kyoto-Koken or Z-5700, Hitachi). However, the solubility of GaAs using SCFCO@sub2@ with H@sub2@O was low about 30 mg/L for As @footnote 2@. Therefore, to increase solubility, oxygen gas (O@sub2@) was introduced into the treatment chamber and SCFCO@sub2@ was filled and treated. The solubility at 50 °C for 30 minute was 3 g/L at 10-30 mega-Pascal (MPa) in SCFCO@sub2@ with O@sub2@ of 0.05 MPa and 6 g/L at 25 MPa in SCFCO@sub2@ with O@sub2@ of 0.15 MPa. The addition of O@sub2@ into SCFCO@sub2@ is effective to increase the solubility of Ga about two orders.@footnote 1@ T.Momose et.al, JVST, A17(4), Jul/Aug, 1999, 1391.@footnote 2@ T.Momose et.al, Proc. 55th Appl. Phys. Tohoku Local Conf., 8pB7, Tohoku Univ., Dec. 2000, 198.

##### **MS-TuP4 Progress toward Spatially Programmable CVD, J. Choo, L. Henn-Lecordier, Y. Liu, R.A. Adomaitis, G.W. Rubloff, University of Maryland**

A preliminary sequence of experimental tests performed to obtain engineering performance data for the Programmable CVD reactor system, together with simulation-based analysis of these data, will be described in this paper. The Programmable Reactor system was developed to improve across wafer sensing and control of reactant gas composition; to demonstrate this concept, a 3-segment prototype reactor was constructed

by modifying a commercial tungsten CVD cluster tool. The key design features of the prototype system include a segmented showerhead assembly which allows control of precursor gas composition to each segment, a reversed flow of residual gas up through the showerhead to reduce inter-segment gas transport, and in-situ residual gas sampling tubes within each segment. These modifications constitute a major evolution in actuator and sampling capabilities relative to conventional CVD designs. To assess the ability of the prototype system to control gas composition across the wafer surface, a sequence of experiments was performed in which the spacing between the showerhead assembly and wafer surface was varied; pure Ar, WF6, and H2 was fed to each of the 3 segments in each test. Visual observations of the deposition patterns demonstrate sharp hexagonal deposition patterns corresponding to the shape of the showerhead segments for the close-spaced experiments; the pattern becomes more diffused as spacing is increased due to increased across-wafer diffusion. Surface resistance measurements reveal the thickest deposition directly under the WF6-fed segment and measurable deposition under Ar and H2 fed segments; simulation analysis of transport within the 1D segments will show that there is significant back-diffusion down each segment from a common exhaust volume, accounting for the observed deposition patterns.

##### **MS-TuP6 Effects of Oxidant on Polishing Selectivity in the Chemical Mechanical Planarization of W/Ti/TiN Layer, K.J. Lee, Y.-J. Seo, DAEBUL University, Korea; S.Y. Kim, ANAM Semiconductor Co., Inc., Korea; W.S. Lee, Chosun University, Korea**

Tungsten is widely used as a plug for the multi-level interconnection structures. However, due to the poor adhesive properties of tungsten on SiO2 layer, the Ti/TiN barrier layer is usually deposited onto SiO2@sub 2@ for increasing adhesion ability with tungsten film. Generally, for the tungsten-chemical mechanical polishing (W-CMP) process, the passivation layer on the tungsten surface during CMP plays an important role. In this paper, the effects of oxidants controlling the polishing selectivity of W/Ti/TiN layer were investigated. The alumina(Al@sub 2@O@sub3@) abrasive containing slurry with 5 % H@SUB2@O@SUB2@ as the oxidizer was studied. As our preliminary experimental results, very low removal rates were observed for the case of no-oxidant slurry. This low removal rate is only due to the mechanical abrasive force. However, for Ti and TiN with 5 % H@SUB2@O@SUB2@ oxidizer, different removal rate was observed. The removal mechanism of Ti during CMP is mainly due to mechanical abrasive, whereas for TiN, it is due to the formation of metastable soluble peroxide complex. This work was supported by Korea Research Foundation Grant(KRF-2002-005-D00011).

##### **MS-TuP7 Slurry Characteristics by Surfactant Condition at Copper CMP Process, I.P. Kim, N.H. Kim, Chung-Ang University, Korea; J.H. Lim, Growell Telecom; S.Y. Kim, Dongbu-Anam Semiconductor, Korea; E.G. Chang, Chung-Ang University, Korea**

Copper is rapidly replacing aluminum interconnections due to its higher conductivity and lower electrical resistance in the semiconductor industry. Therefore, CMP (chemical mechanical polishing) is essential technology in microelectronic fabrication for the planarization of globally complex device topography. In this study, we evaluated the characteristics by the addition of 3 different kinds of nonionic surfactant to improve the dispersion stability of slurries. Slurry stability is an issue in any industry in which settling of particles can result in poor performance. So we observed the variation of particle size and settling rate when the concentration and addition time of surfactant are changed. When the surfactant is added after milling process, the particle size and pH became low. It is supposed that the particle agglomeration was disturbed by adsorption of surfactant on alumina abrasive. The settling rate was relatively stable when nonionic surfactant is added about 0.1-1.0 wt%. When molecular weight (MW) is too small like Brij 35, it was appeared low effect on dispersion stability. It is assumed that it can't prevent coagulation and subsequent settling with too small MW. The proper quality of MW for slurry stability was presented about 500,000. Consequently, the addition of nonionic surfactant to alumina slurry has been shown to have very good effect on slurry stabilization. If we apply these results to copper CMP process, it is thought that we will be able to obtain better yield.

##### **MS-TuP8 A Study on Recycle of Abrasive Particles in One-used Chemical Mechanical Polishing (CMP) Slurry, S.W. Park, Y.-J. Seo, DAEBUL University, Korea; S.Y. Kim, ANAM Semiconductor Co., Inc., Korea**

Recently, the recycle of CMP (chemical mechanical polishing) slurries have been positively considered in order to reduce the high COO (cost of ownership) and COC (cost of consumables) in CMP process. Among the composition of slurries (buffer solution, bulk solution, abrasive particle,

# Tuesday Evening Poster Sessions, November 4, 2003

oxidizer, inhibitor, suspension, antifoaming agent, dispersion agent), the abrasive particles are one of the most important components. Especially, the abrasive particles of slurry are needed in order to achieve a good removal rate. However, the cost of abrasives is still very high. In this paper, we have collected the silica abrasive powders by filtering after subsequent CMP process for the purpose of abrasive particle recycling. And then, we have studied the possibility of recycle of reused silica abrasive through the analysis of particle size and hardness. Also, we annealed the collected abrasive powders to promote the mechanical strength of reduced abrasion force. Finally, we compared the CMP characteristics between self-developed KOH-based silica abrasive slurry and original slurry. As our experimental results, we obtained the comparable removal rate and good planarity with commercial products. Consequently, we can expect the saving of high cost slurry. This work was supported by Korea Research Foundation Grant(KRF-2002-041-D00235).

**MS-TuP9 Corrosion Characteristics of Diffusion Barrier Ta in Copper CMP, D.W. Lee, N.H. Kim, Chung-Ang University, Korea; J.H. Lim, Growell Telecom; S.Y. Kim, Dongbu-Anam Semiconductor; E.G. Chang, Chung-Ang University, Korea**

The diffusion barriers are required for Cu metallization because of the higher diffusivity of Cu in Si and dielectrics. Several barrier materials such as TiN, W, and Ta(TaN) have been studied for diffusion barrier. Among them, Ta-based diffusion barrier has been put emphasis on presently. Ta-base diffusion barrier has no reaction with Cu, better adhesion to both Cu and dielectric, desirable microstructure against Cu diffusion, and heteroepitaxial relationship with Cu. On this study, the corrosion characteristics of the diffusion barrier Ta in Copper Chemical Mechanical Polishing has been investigated. Key experimental variables that have been investigated are the corrosion rate by different agents containing slurry of Cu CMP. Whenever Cu and Ta films were corroded adding each oxidizer, the corrosion rate of Ta was much lower than that of Cu. That is, the difference in the corrosion rates of Ta by oxidizer was not larger as compared with Cu. As corroded by complexing agents, the corrosion rate of Ta was close to 0. The corrosion rate of Ta increased as added HNO<sub>3</sub> and CH<sub>3</sub>COOH compared with the reference slurry; on the other hand, it decreased with addition of HF. In addition, resulting corrosion rate went up with lower pH of agent. However, the corrosion rates by agents were significant small. Hence, it doesn't affect on the removal rate of Cu CMP practically. Consequently, this can be explained by assuming that the mechanical effect dominates than the chemical effect on the polishing rate of Ta(TaN).

**MS-TuP10 Measurement of Energy Flux at the Substrate in a Magnetron Sputter System Using an Integrated Sensor, S.D. Ekpe, S.K. Dew, University of Alberta, Canada**

Knowledge of the energy flux in a sputter deposition system is essential in predicting the properties of the growing film. The use of discrete sensors such as thermocouples for heat measurement has a potential contact problem due to the temperature jump between the surface of the wall and the surrounding gas especially at very low pressures. Embedded sensors such as a microfabricated polysilicon thin film thermistor eliminates the problem associated with the discrete sensors. In this study, the fabricated sensor is calibrated using ohmic self-heating before the deposition plasma is switched on, and also after the plasma is switched off (passive mode). At low pressures (up to 20 mTorr), pressure has an insignificant effect on the thermal resistance of the sensor. For substrate temperatures of up to 250°C, the sensor response is linear with input power. Values of steady state energy flux measured with the sensor range from 5 to 46 mW/cm<sup>2</sup> for aluminum and 14 to 114 mW/cm<sup>2</sup> for copper depending on the process conditions, and compare well with those determined theoretically. Magnetron power was varied between 75 and 300 W, gas pressure 5 - 10 mTorr and substrate-target distance 10.8 - 21 cm.

**MS-TuP11 Temperature Sensor for Multi-layered Substrate using Optical Fiber type Low-coherence Interferometry, K. Takeda, T. Shiina, M. Ito, Y. Okamura, Wakayama University, Japan; N. Ishii, Tokyo Electron Ltd., Japan**

Multi-layered substrates, such as silicon on insulator (SOI) are very useful for MEMS and so on. In the fabrication processes of multi-layered substrates, plasma etching is frequently employed. In such process, the temperature control of each layer, especially top layer, will be required to realize much finer pattern because the interlayer of SOI etc. is dielectric with low thermal conductivity. We have developed novel temperature sensor for measuring each layer of multi-layered substrates using low-coherence interferometry. The sensor is based on Michelson

interferometer, which consists of a super luminescent diode (SLD: wavelength = 1550 nm), a laser diode (LD: wavelength = 850 nm), a scanning reference mirror, optical fibers and so on. In this sensor, the optical pass length of each layer is derived from the length between peaks of SLD interference signals. The shift of each optical pass length is precisely measured by Michelson interferometer using LD, which uses the same optical path as that using SLD. The sensor is, therefore, robust to mechanical and temperature disturbances. We have evaluated the shift of each layer of the three-layered substrate. The top and bottom layers of the substrate are silicon, 360 μm in thickness. The interlayer is made of quartz, 1 mm in thickness. As a result, it has been verified that the shift of optical length of each layer is proportional to each temperature measured by thermo-couple sensors, which corresponds to theoretical values. From these results, we have confirmed that the developed sensor can measure the temperature of each layer without continuous monitoring the shift of optical pass length if the initial temperature is known.

**MS-TuP13 Calibration Standards for X-ray Metrology Systems using a Traceable High-resolution Diffractometer, D. Windover, J. Cline, National Institute of Standards and Technology**

The wealth of information that can be obtained from the characterization of thin film structures by means of X-rays techniques has led to a dramatic increase in their use in recent years. However, currently there are no traceable standards available to calibrate the behavior of these new specialized characterization systems. High resolution X-ray instruments require an entirely new type of artifact to characterize both the parallel beam optics and the performance of the goniometers. Likewise, X-Ray reflectivity requires accuracy in the incident and reflection angles orders of magnitude higher than needed for previous conventional powder diffraction calibration. This work details current efforts for providing NIST Standard Reference Materials for high resolution X-ray diffraction and reflectivity that are traceable to the SI. We focus primarily on the construction and calibration of an advanced X-ray diffractometer designed from the onset to offer flexibility in its configuration and traceable measurements. Features include: high intensity, well characterized, parallel beam X-rays produced using a rotating anode and finely controlled goniometer rotations with both accuracy and precision monitored by optical encoders. We discuss the calibration of the accuracy for the 2θ and θ axes through the method of circle closure. We demonstrate both vibration and temperature effects on goniometer positioning and discuss current improvements to the system designed to account for these effects. Proposed structures for high resolution and reflectivity standards will be presented.

## Author Index

### Bold page numbers indicate presenter

— A —

Adomaitis, R.A.: MS-TuP4, **1**

— C —

Carter, D.C.: MS-TuP2, **1**

Chang, E.G.: MS-TuP7, **1**; MS-TuP9, **2**

Choo, J.: MS-TuP4, **1**

Christie, D.J.: MS-TuP2, **1**

Cline, J.: MS-TuP13, **2**

— D —

Dew, S.K.: MS-TuP10, **2**

— E —

Ekpe, S.D.: MS-TuP10, **2**

— H —

Henn-Lecordier, L.: MS-TuP4, **1**

— I —

Ishii, N.: MS-TuP11, **2**

Ito, M.: MS-TuP11, **2**

— K —

Kim, I.P.: MS-TuP7, **1**

Kim, N.H.: MS-TuP7, **1**; MS-TuP9, **2**

Kim, S.Y.: MS-TuP6, **1**; MS-TuP7, **1**; MS-TuP8, **1**; MS-TuP9, **2**

— L —

Lee, D.W.: MS-TuP9, **2**

Lee, K.J.: MS-TuP6, **1**

Lee, W.S.: MS-TuP6, **1**

Legare, J.M.: MS-TuP1, **1**

Lim, J.H.: MS-TuP7, **1**; MS-TuP9, **2**

Liu, Y.: MS-TuP4, **1**

— M —

Momose, T.: MS-TuP3, **1**

— O —

Okamura, Y.: MS-TuP11, **2**

Otomo, O.: MS-TuP3, **1**

— P —

Park, S.W.: MS-TuP8, **1**

— R —

Rubloff, G.W.: MS-TuP4, **1**

— S —

Seo, Y.-J.: MS-TuP6, **1**; MS-TuP8, **1**

Shiina, T.: MS-TuP11, **2**

Sproul, W.D.: MS-TuP2, **1**

— T —

Takeda, K.: MS-TuP11, **2**

Tomasel, F.: MS-TuP2, **1**

— W —

Wang, S.: MS-TuP1, **1**

Windover, D.: MS-TuP13, **2**