SYMPOSIUM J

Texture and Microstructure in Electronic and Magnetic Films

April 1 – 3, 2002

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TUTORIAL

ST J: TEXTURE AND MICROSTRUCTURE IN THIN FILMS
Monday, April 1, 2002
8:30 a.m. - 4:30 p.m.
Salon 3/4 (Marriott)

Texture plays an increasingly important role in the application of materials in electronic and magnetic devices. The texture of thin films and multilayers must be controlled to optimize properties and performance and to increase device lifetimes. The current definition of texture incorporates not only preferred orientation, as described by the orientation distribution function, but also the grain boundary character distribution, which describes the statistics of lattice misorientation and grain boundary plane orientation. Symposium J aims to bring together those engaged in electronic and magnetic device research, development, production and failure analysis, where texture has a documented or potential effect on the relationships between processing, properties and performance. Material classes of interest include interconnect metals, electronic bulk ceramics, magnetic films, ferroelectric and piezoelectric films, dielectrics, and buffer, barrier and seed layers.

The tutorial will cover the following areas:

- Groundwork for understanding basic texture concepts and applying them in thin film and bulk situations
- Electron backscatter diffraction techniques applied to local and global texture phenomena and issues in which microstructure and grain boundary character are of importance
- Texture in thin films, including the formation, development, measurement and analysis of thin film texture

The final tutorial session will be devoted to a round-table discussion of texture and microstructure issues.

Instructors:
Jerzy A. Szpunar, McGill University
Stuart I. Wright, TSL/EDAX
Hualong Li, McGill University

SESSION J1: TECHNIQUES AND APPLICATIONS
Chair: David P. Field
Tuesday Morning, April 2, 2002
Nob Hill A/B (Marriott)

8:00 AM *J1.1
CRYSTALLOGRAPHIC TEXTURE AND PHASE MEASUREMENTS DURING DAMASCENE COPPER PROCESSING.
Kris Kozaczek, Dave Kurtz, Roger Martin, Paul Moran, HyperNex, Inc., State College, PA; Ken Rodbell, IBM Research, Yorktown Heights, NY; Patrick Dehaven, Sandra Malhotra, IBM Microelectronics Division, East Fishkill, NY.

The rapid adoption of damascene copper processing has brought about an increased need to understand and control microstructure in the barrier and metal layers during manufacture. We have developed and implemented a methodology for rapidly characterizing thin film polycrystalline microstructures on 200 mm Si substrates using an x-ray based metrology tool to measure crystallographic texture and phase in a time frame suitable for in-line applications. The acquired data can be used as a direct measure of the deposition process in terms of film quality, reproducibility and stability over time. The spatial distribution of crystallographic texture and phase can be measured on a single wafer in order to monitor in-wafer uniformity. More importantly, the same measurements can be carried out at predetermined intervals on wafers from a single deposition tool, and the results used to create a database that can be applied to trend charting and to the establishment of acceptance criteria. The methodology developed makes use of several novel data analysis and collection techniques, such as the use of a direct matrix transformation method to determine the orientation distribution function (ODF) from a group of truncated pole figures. Useful quantitative outputs of the ODF, such as volumetric fractions, can be used in quantifying texture evolution within and between wafers. On the fly texture compensation can also be used to generate useful quantitative phase measurements. We present the principles of operation of this metrology tool and examples collected at IBMs Advanced Semiconductor Test Center (ASTC) in East Fishkill, NY.

8:30 AM J1.2
A COMPARISON OF TEXTURE MEASUREMENTS USING X-RAY, NEUTRON AND ELECTRON DIFFRACTION TECHNIQUES. Mark Vaudin and Stephen Banovic, Materials Science and Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, MD.

Texture in biaxially stressed aluminum sheet was measured using a number of different techniques, as a means of assessing the efficacy of a number of the commonly available texture measurement techniques. Two x-ray diffraction (XRD) techniques were employed, one using an area detector and one a 2-circle diffractometer. Four-circle neutron diffraction (ND) methods were also applied, and the final technique was electron backscatter diffraction (EBSD). The diffraction methods all indicated similar trends in texture. The 2-circle method gave detailed data over limited regions of orientation space, but compared well with the area detector findings. Differences in degree of texture were apparent when comparing the neutron and x-ray results, with the ND results typically indicating a lesser degree of texture than XRD. Possible reasons for this are being investigated, in particular with respect to the change in diffraction peak profiles as the orientation of the specimen is altered. Early examination of the EBSD results suggests that they are more in line with the ND data. Implications for the various texture measurement techniques will be discussed.

8:45 AM J1.3
COMPARISON OF SURFACE ROUGHNESS OF SPUTTER-DEPOSITED AND EVAPORATED MAGNETIC THIN FILMS MEASURED BY SOFT-X-RAY SCATTERING AND ATOMIC FORCE MICROSCOPY USING CARBON NANOTUBE TIPS.
B.M. Barnes, J.J. Kelly IV, M. Friesen, D.P. Lagally[a], D.E. Savage, and M.G. Lagally[a], University of Wisconsin-Madison, Madison, WI; [a]Piezomax Technologies, Middleton, WI.

In order to provide a more quantitative assessment of interfacial roughness in magnetic multilayer thin films, the surface roughness of a variety of metallic thin films and multilayers deposited by evaporation and sputter deposition was investigated by soft-x-ray-scattering and atomic force microscopy (AFM) in the intermittent-contact mode using carbon nanotube (CNT) tips. The lateral length scales over which AFM provides information are limited on the lower end by tip diameter and on the upper end by the lateral dimension of the scan ($\sim$10 μm)[1]. The use of CNT tips allows us to reduce the lower limit and also to provide a truer view of texture. Soft x-ray scattering allows probing of length scales from the wavelength of the x-ray ($\sim$1 nm) to an upper limit set by instrumental broadening ($\sim$0.5 μm). Soft-x-ray scattering is indirect, requiring modeling for interpretation[2], but it does allow the measurement of buried interfaces. Conventional modeling, assuming a form of correlation function, suffices to parameterize the surface roughness of deposited films that exhibit fractal roughness on a short length scale and are smooth on a long length scale (i.e., self-affine with a long range cutoff). The modeling breaks down for surfaces that become non-fractal due to preferred grain sizes, ordered underlayers, and other types of texture. Parameters obtained from AFM measurements were integrated with soft-x-ray scattering modeling to provide a clearer understanding of the rms roughness, lateral correlation length, and "jaggedness" of such films than either technique alone could provide. Correlation lengths were obtained directly from the AFM data and used in the fitting of the x-ray data. Samples with different types of texture, including periodic nanotextures, were investigated. Implications of these results for studies of magnetic interface roughness and its effect on interfacial magnetism will be discussed. Supported by Seagate and ONR. The Synchrotron Radiation Center is funded by the NSF (Award No. DMR-0084402).
[1] C. Teichert, et al. Appl. Phys. Lett. 66, 2346 (1995). [2] S. Sinha, et al. Phys. Rev. B 38, 2297 (1988).

9:00 AM J1.4
IN-SITU OBSERVATION OF ELECTROCHEMICAL DEPOSITION OF COPPER AND COBALT ON Au(111) USING AN OBLIQUE INCIDENCE OPTICAL REFLECTIVITY DIFFERENCE TECHNIQUE. Jeremy Gray, University of California, at Davis, Davis, CA; Walther Schwarzacher, University of Bristol, Bristol, UNITED KINGDOM; Xiangdong Zhu, University of California-Davis, Davis, CA.

We demonstrate an oblique incidence optical reflectivity difference technique as a viable surface probe capable of exploring sub-monolayer and multilayer adsorption and dissolution in electrochemical deposition. The optical method was implemented in conjunction with cyclic voltammetry and underpotential deposition (UPD) to characterize nucleation and growth of Cu and Co on Au(111). Reflectivity changes were observed for both Cu and Co from a fraction of a monolayer to multilayers. In the case of Co, we also studied adsorption and dissolution for a dilute (0.02M) and a concentrated (0.2M) Co solution. The optical signals clearly indicate

that there exists a significant difference between Cu and Co electro-deposition on Au(111). Presumably, this difference can be a result of an inherent difference in electrochemistry between these two systems, or is a ramification of the magnetic character of Co. We compare our findings with similar studies of Cu and Co on Au which utilized in-situ atomic force and scanning tunneling microscopies. Our results show that the optical technique can be made into an integral part of many electrochemical systems and implemented as a useful in-situ deposition probe.

9:15 AM J1.5
LOCAL STRAINS IN Al INTERCONNECTS DURING THERMAL CYCLING AND ELECTROMIGRATION MEASURED USING CONVERGENT BEAM ELECTRON DIFFRACTION.
Stephan Krämer, Julie Nucci, Cynthia A. Volkert, Eduard Arzt, and Manfred Rühle, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

Energy filtered convergent beam electron diffraction (CBED) was used to measure *in situ* the development of local lattice strain and texture during thermal cycling and electromigration testing of 300 nm wide unpassivated Al interconnects. Due to several recent improvements in the method, it is now possible to evaluate the triaxial strain state of the as-fabricated interconnect with high spatial resolution (10-100 nm) and with an accuracy of 10^{-4}. In addition, the orientations of regions as small as 20 nm could be determined using Kikuchi patterns. Initial measurements performed as a function of position along the interconnect show that the strain state is predominately uniaxial. However, the strain state varies locally within single grains as well as from grain to grain by as much as 50%, which cannot be explained by elastic or plastic anisotropy due to the different crystallographic orientations of the grains. It will therefore be discussed whether these inhomogeneities are generated by dislocation strain fields or by local variations in grain boundary or interface structure. Measurements during thermal cycling provide evidence for local inelasticity, even for temperatures where no macroscopic plasticity is observed. This will also be discussed in terms of changes in local dislocation or interface structure. Finally, measurements during electromigration testing reveal changes in grain orientation and show that strains are generated at the ends of the tested segments which do not follow the behavior predicted by continuum electromigration models.

SESSION J2: METAL FILMS
Chair: John A. Sutliff
Tuesday Morning, April 2, 2002
Nob Hill A/B (Marriott)

10:00 AM *J2.1
DEVELOPMENT AND EVOLUTION OF TEXTURE IN POLYCRYSTALLINE THIN FILMS. H.J. Frost, Dartmouth College, Thayer School of Engineering, Hanover, NH; and C.V. Thompson, MIT, Dept. of Materials Science and Engineering, Cambridge, MA.

Texture and microstructure in thin polycrystalline films develop during film formation, and evolve during subsequent processing, via a variety of different mechanisms, and in response to a variety of different driving forces. Different driving forces may give preference to different particular crystallographic orientations. Similarly, different kinetic limitations on crystal growth or grain growth may also favor different particular crystallographic orientations. A notable example is the competition during grain growth in FCC metal films between surface energy minimization, favoring (111) textures, and strain-energy minimization, which favors (100) textures. Similar competitions also occur during the growth to coalescence of islands during initial film formation, and the growth of columnar grains during film thickening. In this paper we will review the state of experimental observations and related computer simulations of these competitive texture evolution processes.

10:30 AM J2.2
TEXTURE DEVELOPMENT AND TWINNING IN POLY-CRYSTALLINE GOLD THIN FILMS. Rachel E. Cefalu and Alexander H. King, Purdue University, School of Materials Engineering, West Lafayette, IN.

Gold thin films usually develop a $< 111 >$ texture upon heating. We will show that this texture can be influenced by the formation of twins, which may be influenced by the substrate material. Gold thin films were evaporated on rock salt and glass, and incrementally annealed without removal from the substrate. The texture was mapped using an area detector x-ray diffractometer. The films showed a $< 111 >$ fiber texture on both substrates with the addition of a [511] fiber texture component, which is much more pronounced on rock salt. The [511] direction is a twin rotation direction showing that there is more twinning on rock salt than on glass although there is no lattice matching between the rock salt and gold. The reasons for difference in texture will be discussed and its implications for other f.c.c. metal films, and choice of appropriate substrates, in various applications, will be considered. This work is supported by the National Science Foundation, Grant #DMR0096147.

10:45 AM J2.3
MOLECULAR DYNAMICS SIMULATIONS OF Cu AND Ta THIN FILM DEPOSITION. Peter Klaver, Barend Thijsse, Delft University of Technology, Laboratory for Materials Science, Delft, THE NETHERLANDS.

We report simulation results of film deposition of Cu on Ta and Ta on Cu in order to study the evolving film microstructures and interface effects. Cu was deposited on different beta-Ta (001) surfaces and on bcc surfaces with (100) and (110) orientations. Ta was deposited on Cu (111) surfaces. As in experiments, epitaxial growth and amorphous films were both observed. In almost all cases films grew amorphous, epitaxial growth was observed during deposition on bcc (110) surfaces and on flat beta-Ta (001) surfaces at low temperature. The roughness and temperature of the Ta substrate play a crucial role. Weaker bound Ta adatoms can mix into the Cu, inhibiting the formation of crystalline Cu. Higher temperatures increase mixing kinetics, so, perhaps counterintuitively, higher deposition temperatures were found to result in amorphous films. In the cases of epitaxial growth of Cu on Ta, this growth is usually preceded by a few partially disordered monolayers, not because Ta atoms intermix but because of the crystallographic mismatch. However, these disordered layers undergo significant transformation during further deposition, sharpening the transition between the substrate and the deposited film, down to one monolayer in one case. Still, the epitaxial relations of Cu on Ta are far more complicated then simple NW or KS relations. Ta, although thermodynamically immiscible with Cu, rapidly penetrates into Cu after deposition, because Ta in Cu has a considerably higher cohesion energy than Cu in Cu. This makes it energetically favourable for separate Ta atoms to diffuse into Cu lattice positions. The tendency of Ta atoms to immerse themselves in Cu is so strong that Ta penetration actively amorphisises part of the Cu substrate lattice during deposition. Also, it causes a Cu enrichtment at the surface. These effects emphasize that bulk thermodynamics cannot be simply applied when free surfaces are available.

11:00 AM J2.4
EVOLUTION OF MICROSTRUCTURE IN Ti-Ta BILAYER THIN FILMS ON POLY-Si AND Si(001). A.S. Ozcan, K.F. Ludwig Jr., Boston University, Physics Department, Boston, MA; K.P. Rodbell, C. Lavoie, C. Cabral Jr., J.M.E. Harper, IBM T.J. Watson Research Center, Yorktown Heights, NY.

We have studied the formation of titanium silicides in the presence of an ultra-thin layer of Ta interposed between Ti and Si. In-situ x-ray diffraction (XRD), resistance measurements and light scattering were used to study the thin film reactions in real time. On both poly-Si and Si(001) substrates Ta thickness was varied from 0 to 1.5 nm while Ti thickness was held constant at ~27 nm. The time-resolved XRD shows that the volume fraction of C40 and metal-rich silicide phases grows with increasing Ta layer thickness. Increased Ta layer thicknesses also cause delayed growth of the C49 disilicide phase. Among the Ta thicknesses we examined, 0.3 nm is the most effective one in lowering the C49-C54 transformation temperature. Films with Ta layers thicker than 0.5 nm do not completely transform into the C54 phase. The texture of the C54 phase is very sensitive to the Ta thickness. The C54 disilicide film is predominantly (010) textured for the Ti / 0.3 nm Ta sample on both poly-Si and Si(001). Pole figure analysis also shows an in-plane orientation for the film on Si(001) with C54[001] ‖ Si [110]. This orientation is consistent with a template mechanism acting between the $< 300 >$ planes of Ti_5Si_3 phase and the $< 010 >$ planes of the C54 disilicide phase. The final C54 texture is significantly different for Ta layers thinner or thicker than 0.3 nm. This suggests that the most effective thickness for lowering the C54 formation temperature is related to the development of a strong (010) texture. We have observed similar relationships between Ta concentration, C54 texture and transformation temperature in Ti films alloyed with Ta.

11:15 AM J2.5
THE EFFECT OF PLASTIC STRAIN ON GRAIN GROWTH AND TEXTURE IN Al FILMS. C.A. Volkert and D. Tse, Max Planck Institute for Metals Research, Stuttgart, GERMANY.

Stored energy due to plastic deformation is known to play a major role in grain growth in bulk metals. However, the effect of deformation on grain growth in thin metal films has not been systematically investigated despite the fact that films experience large deformations during annealing due to their thermal expansion mismatch with surrounding materials. The goal of this work is to investigate the role of small strains (~1%), comparable to those experienced during heating of metal films on substrates, on grain growth in Al films. The

samples, which were 0.5 μm thick Al films on polyimide substrates, were strained in a microtensile tester during annealing at temperatures up to 300°C and were subsequently characterized using FIB microscopy, x-ray diffraction, and EBSD. It was found that tensile straining increased the average grain size by up to a factor of three over the unstrained films. Results on the effect of applied strain on grain size and film texture will be presented. In addition, a model will be discussed to account for the observation that thin film grain sizes scale with the film thickness.

11:30 AM J2.6
INTERFACE AMPORPHIZATION AND TEXTURE OF Co FILMS ON DIFFERENT METAL SUBSTRATES. D.K. Sarkar, M. Falke, G. Beddies and H.-J. Hinneberg, Institute of Physics, Chemnitz University of Technology, Chemnitz, GERMANY.

Thin films of different metals (M) of thickness around 10 nm are deposited on Si(100) substrates and a ~30 nm Co film is deposited on these metal films using the magnetron sputtering method. An experiment has been carried out to understand the role of the thickness of the under layer metal films by varying the thickness of Zr films from 0.5 nm to 10 nm. Cross Sectional Transmission Electron Microscopy (XTEM) shows the presence of a ~2 nm buried ultra thin amorphous interlayer at the interface between the Co layer and the M-layers. X-Ray Reflectivity (XRR) is used to determine the electron density of this buried ultra thin amorphous interlayer. X-Ray Diffraction (XRD) is used to determine the crystalline quality of the deposited Co film on these various metal film substrates. Pole figure measurements confirm that the Co film is highly textured on such metallic substrates. Theoretically, the heat of mixing (-DH) has been calculated for Co-M systems. The local temperature rise is estimated using the average heat capacity and the calculated heat of mixing of the amorphous interface compound (Co_3M). The local temperature rise due to the amorphous phase formation and hence better mobility of the further deposited metal atoms is the cause of highly textured Co thin films on such metal substrates.

11:45 AM J2.7
TEM STUDIES OF THE IN-SITU HEATING OF NANO-STRUCTURED Cu. Pei-I Wang, Y.-P. Zhao, E. Barnat, J. Senkevich, A. Vijayaraghavan, and T.-M. Lu, Center for Integrated Electronics, Electronics Manufacturing and Electronic Media, Rensselaer Polytechnic Institute, Troy, NY.

Transmission electron microscopy (TEM) is often used for the structural and elemental analysis of nano-electronics materials. It has been shown that in-situ heating in a TEM is useful for dynamic observation of intermediate products in chemical reactions and of complicated processes of crystal growth. In this study, various recently fabricated nanometer-sized structures, isolated Cu nano-rods, and ultra-thin Cu films deposited onto Si oxide membranes with different adhesion promoters, were heated in-situ using electron beam. Their microstructural changes during heat treatments were investigated. We found that the grain boundaries gradually migrated and were eventually eliminated throughout the Cu nano-rods with sufficiently long hold times on heating. Our observations also show that upon heating, the Cu ultra-thin film wets the substate surface with the adhesion layer compared to de-wetting of the Cu film directly deposited onto the Si oxide substrate. These results and TEM microstructure data will be presented and discussed. We demonstrate that the present technique can provide valuable information on defects-elimination of Cu nano-rods and the wetting of thin films on dielectric surface.

SESSION J3: COPPER FILMS
Chairs: Patrick W. DeHaven and John E. Sanchez
Tuesday Afternoon, April 2, 2002
Nob Hill A/B (Marriott)

1:30 PM *J3.1
TEXTURE DEVELOMENT IN Cu ALLOY FILMS. K. Barmak, A. Gungor, Carnegie Mellon Univ, Dept of MS&E, Pittsburgh, PA; C. Cabral Jr., J.M.E. Harper, IBM T.J. Watson Research Center, Yorktown Heights, NY.

It is believed that the addition of alloying elements will become necessary for tailoring the microstructure (i.e., grain size and crystallographic orientation or texture) and thus the reliability and functionality of Cu interconnections as line widths approach 100 nm. In this work, the impact of nine alloying elements on the texture of Cu films was investigated. Five of the alloying elements, Al, In, Mg, Sn, and Ti form compounds, while the other four, Ag, B, Co, and Nb do not form compounds with Cu. Alloy films, approximately 500 nm thick, and containing nominally 1 and 3 at% solute were electron beam evaporated onto thermally oxidized Si wafers. Isothermal anneals were carried out at 400C for five hours, while constant-heating rate treatments were done at 3C/s to 650C, 750C, and 950C. In all cases, annealing, whether isothermal or constant-heating-rate, resulted in strengthening of the < 111 > fiber texture when compared with the as-deposited state. For the isothermal anneal at 400C, the strongest < 111 > fiber texture was found in Cu(3 at% Ti), while for the constant-heating-rate treatments, the strongest < 111 > fiber texture was found in Cu(3 at% Nb) annealed to 950C. Furthermore, in all cases, the < 111 > fiber texture was found to be stronger for the nominally 3 at% alloy when compared with the 1 at% alloy. In addition to discussing the behavior of the alloys, the merits of the three techniques for texture analysis - conventional theta/two-theta x-ray diffraction, the pole plot, and pole figures in combination with the orientation distribution function - will be discussed.

2:00 PM J3.2
DILUTE Cu(Ti), Cu(In), Cu(Sn) AND Cu(Al) THIN FILMS: GRAIN GROWTH, TEXTURE, AND RESISTIVITY. A. Gungor, K. Barmak, Carnegie Mellon Univ, Dept of MS&E, Pittsburgh, PA; C. Cabral, IBM T.J. Watson Research Center, Yorktown Heights, NY; A. Ozcan, Boston Univ, Dept of Physics, Boston, MA; C. Lavoie, J.M.E. Harper, IBM T.J. Watson Research Center, Yorktown Heights, NY.

Annealing dilute Cu(Ti), Cu(In), Cu(Sn) and Cu(Al) alloy films at 400C for 5 hrs resulted in the strengthening of the < 111 > fiber texture when compared with the as-deposited state, with Cu(Ti) showing the strongest texture of all films. Annealing also resulted in a decrease of electrical resistivity and the growth of grains, with Cu(Ti) and Cu(In) showing the lowest resistivity and Cu(In) and Cu(Sn) exhibiting the largest grain size. The study of these four alloy films was motivated by the fact that the performance and reliability of metal interconnections are increasingly dependent on aspects of microstructure such as grain size and crystallographic orientation (texture) as line widths approach 100 nm, with the expectation that the control of these microstructural parameters in Cu will require the addition of alloying elements. The alloy films, approximately 500nm in thickness and containing nominally 3 at% solute, were electron beam evaporated onto thermally oxidized Si wafers. Pure evaporated, approximately 500 nm thick Cu film samples were used as controls. Four point probe resistance measurements, in situ synchrotron and conventional x-ray diffraction, transmission electron microscopy, Rutherford back scattering and particle induced x-ray emission were used to characterize the films and to follow the changes in film microstructure, phase constitution and resistivity upon annealing. The behavior of these four alloy films will be compared and contrasted with that of Cu(Nb), Cu(B) and Cu(Co) studied previously.

2:15 PM J3.3
BEHAVIOR OF Cu IN NARROW STRUCTURES. Sywert Brongersma, Martin Nolan, Iwan Vervoort, Karen Maex, IMEC, BELGIUM.

In the past we have established a thorough understanding of grain growth in electroplated blanket Cu films by studying changes in stress, sheet resistance, desorption, impurity levels, and grain structure as a function of several deposition parameters. It encompasses a first stage of growth that is controlled by grain boundary self-diffusion, a stagnation due to the build-up of Sulfur in the grain boundaries, and a continuation of growth when the Sulfur is removed at higher temperatures. The incorporation of impurities due to the use of several organic additives in the plating chemistry plays a crucial role in controlling the importance of these three stages. However, the effect of these additives on the plating dynamics becomes increasingly important when going to narrower structures. Therefor we have now studied the behavior in damascene lines (0.13-10 um wide; 0.5-1.0 um deep) in detail and found it to be very different indeed. Not only is there an unexpected acceleration of grain growth, but also the magnitude of changes in resistance and crystal orientations are strongly linewidth dependent. In fact, the as-deposited resistance of Cu in the lines can be up to 40% higher than bulk values while a typical value of 20% is observed for blanket films. Measurements on impurity levels in the lines are ongoing. The data obtained so far can be explained in terms of two factors. Firstly there are significant changes in the as-deposited grain structure due to the bottom-up or superfilling, which is induced by the organic additives. Secondly, the spatial confinement has a strong influence on grain sizes and orientations obtained after various annealing conditions.

2:30 PM J3.4
THE EFFECTS OF DEPOSITION CHEMISTRY ON TEXTURE AND MICROSTRUCTURE DEVELOPMENT IN COPPER. Matthew M. Nowell, Jeffrey K. Farrer, TSL/EDAX, Draper, UT.

The electrical chemical plating (ECP) of copper for interconnect lines in high performance integrated circuits has become routine in the semiconductor industry. However the effects of additives to the deposition baths on the resulting microstructure are not well understood. In this work, copper films were deposited with differing

bath chemistries and then patterned into integrated circuit devices. The texture and microstructure of the copper contact pads and interconnect lines of various sizes were then characterized using orientation imaging microscopy. The effects of the differing bath chemistry and additives on the resulting microstructures were examined.

3:15 PM *J3.5
TEXTURE AND MICROTEXTURE OF COPPER FILMS PREPARED BY THE SELF-ION ASSISTED DEPOSITION TECHNIQUE ON BARRIER LAYERS WITH DIFFERENT STRUCTURE. Oleg Kononenko, Victor Matveev, Institute of Microelectronics Technology and High Purity Materials, RAS, Chernogolovka, Moscow Region, RUSSIA; Andrei Vasiliev, Moscow State Institute of Radioengineering, Electronics and Automation (Technical University), Department of Condensed Matter Electronics, Moscow, RUSSIA; David Field, School of Mechanical and Materials Engineering, Washington State University, Pullman, WA.

Copper films were deposited onto silicon wafers with different barrier layers by the self-ion assisted deposition technique at the 6 kV bias. Polycrystalline TaN and amorphous TaSiN barrier layers were deposited by E-beam gun. Thickness of copper films was 0.3 and 1.0 μm. After deposition, films were annealed under vacuum. Texture of the films was studied by XRD. Microstructural analysis of the copper films was performed by orientation imaging microscopy. The through thickness tilt of the boundary is unknown, but is assumed to be normal to the specimen surface in this investigation. Results of the structural analysis will be discussed from the viewpoint of electromigration reliability.

3:45 PM J3.6
INFLUENCES OF HYDROGEN ON THE EVOLUTION OF THE ELECTRICAL RESISTIVITY OF ULTRA-THIN SPUTTERED COPPER FILMS. E.V. Barnat, P.-I. Wang, D. Nagakura, T.-M. Lu, Rensselaer Polytechnic Institute, Department of Physics, Applied Physics, and Astronomy and Center for Integrated Electronics, Troy, NY.

Recently there has been much interest in the electrical characteristics of very thin copper films deposited on an insulating substrate for advanced integrated circuit applications. In this paper, comparisons are made between the evolution of the electrical resistivity of copper films sputtered in an atmosphere containing hydrogen to films grown in an atmosphere of argon in the range of 3 to 100 nm. Measurements of the electrical resistivity, obtained in real time, indicate that the films grown in the hydrogen-containing atmosphere are comparable to the films grown in argon at the initial stages of growth, while possessing lower resistivities at the latter stages of growth. Furthermore, measurements made of a transient behavior of the electrical resistivity immediately after deposition indicate that the films are in a meta-stable state. The degree of change and the time in which the films undergo the change in the electrical resistivity are shown to depend on the presence of hydrogen during growth, with the films grown in a hydrogen atmosphere undergoing a more dramatic drop in resistivity. X-ray diffraction spectra show that the transient behavior of the resistivity after deposition corresponds to changes in the microstructure, while transmission electron microscope images are used to investigate the influence hydrogen has on the microstructure of the sputtered copper films.

4:00 PM J3.7
MOLECULAR DYNAMICS SIMULATION OF COPPER THIN FILM GROWTH ON β-Ta SUBSTRATE. Youhong Li, James B. Adams, Chemical and Materials Engineering Department, Arizona State University, Tempe, AZ.

Understanding the copper thin film growth mechanism is very important for predicting correct thin film structure. Tantalum can be used both as a diffusion barrier and adhesion layer for copper metallization in the semiconductor industry. Experiments showed that b-Ta (200) substrate promotes the (111) texture growth in copper films grown above it. In this study, we use Molecular Dynamics method with our embedded atom method (EAM) Cu-Ta potential developed by force matching method (FMM) to simulate initial copper thin film growth on b-Ta substrate. Both Cu/Ta interfacial structures and copper film structure are investigated.

SESSION J4: POSTER SESSION
TEXTURE AND MIROSTRUCTURE IN ELECTRONICS AND MAGNETIC FILMS
Chair: Kris J. Kozaczek
Tuesday Evening, April 2, 2002
8:00 PM
Salon 1-7 (Marriott)

J4.1
GROWTH AND PHYSICAL PROPERTIES OF HIGHLY (100)-ORIENTED PZT THIN FILMS ON $LaNiO_3$ BUFFERIZED PLATINIZED SILICON AND SILICON WAFERS. J.H. Chu, J. Yu, X.J. Meng, J.L. Sun, Z.M. Huang, S.L. Guo and D.Y. Tang, National Laboratory for Infrared Physics, Shanghai Institute of Technical Physics, Chinese Academy of Sciences, Shanghai, CHINA.

Ferroelectric thin films based on $Pb(Zr,Ti)O_3$ (PZT) have demonstrated a promising large field of applications, such as ferroelectric memories, piezoelectric micromotors, pyroelectric focal plane array detectors, and electro-optic devices. Due to its directional spontaneous polarization, exhibiting consequently large anisotropy in physical properties, the preferred crystallographic orientation of those thin films exerts an essential influences on device performances. On one hand, using metallic oxides electrodes such as $LaNiO_3$, $(La,Sr)CoO_3$, et al., the challenging problems of polarization fatigue and imprint can be well overcome. On the other hand, the utilization of $LaNiO_3$ as buffer layer is also an useful technique to induce a preferred crystallograpic orientation of ferroelectric films, besides controlling the film deposition parameters. In this study, highly-(100) oriented PZT thin films were derived on $LaNiO_3$ bufferized platinized silicon and silicon wafers by chemical solution deposition processes. X-ray diffractometer, atomic force microscopy, spectroscopic ellipsometry (SE), RT66A ferroelectric analyzer and HP4194A impedance analyzer were used to characterize microstructures, optical and electrical properties. On both $Pt(111)/Ti/SiO_2/Si$ and Si wafers, the highly (100)-oriented $LaNiO_3$ thin films were obtained via a simple metalorganic solution decomposition technique, and then PZT thin films were deposited by a modified sol-gel process. The orientation of PZT films has been found to vary with the used process parameters. The orientation mechanisms are discussed closely relating to lattice mismatch and thermal expansion coefficient mismatch between ferroelectric film and substrate. Optical properties of the $PZT/LaNiO_3/$ platinized silicon heterostructures have been charaterized by SE technique, in which their dielectric functions can be described by classical Lorentz-Drude model. Ferroelectric hysteresis and C-V bufferfly loop are further given, and relationship of those electrical properties with the film microstructures such as crystallite orientation, crystallite size, etc are also discussed. At last, the possibility of these heterostructures applied in thermal microsensors are also analysed.

J4.2
THE INITIAL GROWTH STAGES AND CRYSTALLIZATION MECHANISM OF Bi-BASED FILMS. Oleg Kononenko, Alexandra Andreeva, Alexandr Ilin, Victor Matveev, Institute of Microelectronics Technology and High Purity Materials, RAS, Chernogolovka, Moscow Region, RUSSIA.

In the present work initial stages of the growth of Bi, Bi-Sb films produced by thermal evaporation (TE) and self-ion assisted deposition (SIAD) were investigated. Bi films on amorphous substrates are formed by the Volmer-Weber mechanism and are characterized by different orientations of facetted islands. A preferential island orientation and stable facetted morphology are observed for crystalline substrates. For amorphous substrates a relative number of islands with the (111) R orientation increases with temperature. The island interaction during crystallization on cold substrates occurs without coalescence. Upon condensation on a warm substrate (100°), the facetted islands are larger due to coalescence and recrystallization processes. In this case the continuous films formed highly irregular grains of different grain sizes whereas large grains are usually (111) R oriented. The annealing leads to a more perfect film texture and an increase in the number of grains with the (111) R orientation. The formation of continuous films prepared by TE on a cold crystalline substrate proceeds by the vapor-crystal mechanism without coalescence, and preferentially coherent twin Σ=3 grain boundaries are observed. For the Bi films obtained by SIAD a clearer facetted morphology and larger islands are observed. A decrease of the substrate temperature leads to an increase of the growth center density and a decrease of the island density with the (111) R orientation. The microstructure perfection of the continuous films is observed with an increase of their thickness up to 200-300 nm. For the Bi-Sb films, the island facetted morphology is not so clear as for pure Bi films which is connected with partial coalescence by the liquid fluidity mechanism and lower anisotropy of the Sb lattice compared to the Bi one. At all substrate temperatures, the film grain orientation (111) R is stronger for SIAD films.

J4.3
POLE FIGURE ANALYSIS OF EPITAXIAL FILMS OF ZnO:2wt%Al GROWN ON SAPPHIRE SUBSTRATES BY RF MAGNETRON SPUTTERING. P. Kuppusami, Indira Gandhi Centre for Atomic Research, Kalpakkam, INDIA; S. Fiechter, K. Ellmer, Dept. Solarenergieforschung, Hahn- Meitner- Institut, Berlin, GERMANY.

ZnO oxide with a room temperature band gap of 3.37 eV, similar to that of GaN, has recently attracted attention as a useful material for short wavelength light-emitting devices. In order to realise these applications, the films must be grown epitaxially on suitable single crystal substrates. Among several single crystal substrates reported so far, sapphire (Al_2O_3) has been relatively inexpensive and a widely used substrate material in semiconductor industry. However, the large mismatch between lattice constants of sapphire and ZnO could produce films with a significant crystal mosaicity. There has been a considerable attempt to circumvent this difficulty by optimising the deposition parameters used in the deposition techniques. We report here the texture analysis of ZnO: 2wt%Al films deposited on (11.0) oriented or a-plane sapphire and (00.1) oriented or c- plane sapphire substrates by radio frequency magnetron sputtering from ZnO:2wt%Al target. Conventonal x-ray diffraction analysis of the films showed the following orientation relationships:(00.1) ZnO$\|$(11.0)Al_2O_3 on a-plane sapphire and (00.1) ZnO$\|$ (00.1)Al_2O_3 on c-plane sapphire substrates. To study the in-plane orientation of the films on the substrates, x-ray pole figure measurements were carried out using the (10.1) reflection of ZnO. The films grown on a- plane sapphire substrates are found to have a clear six fold symmetry of ZnO (10.1) reflections along the surface normal. These aligned in plane domains become dominant with increase in substrate temperature. There was also no indication of rotation domains or twinning domains in the films grown on a-plane sapphire. On the other hand, the films grown on c-plane sapphire was found to have two kinds of rotation domains rotated by 30° and their formation is significantly influenced both by substrate temperature and oxygen content. The role of these domains on the electrical properties will be further discussed.

J4.4
ANOMALOUS GRAIN GROWTH IN SPUTTERED CoCrMn THIN FILMS. Hajung Song, Soon-Ju Kwon, POSTECH, Dept of Materials Science and Engineering, Pohang, KOREA; Kyung-Ho Shin, KIST, Nano Device Research Center, Future Technology Research Division, Seoul, KOREA.

Anomalous grain growth is observed in sputtered hcp CoCrMn thin films. The grain size is about 250 nm wide and a few micrometers long in the film of only 50 nm thickness. Selected area diffraction (SAD) technique indicates that the perpendicular direction (to the substrate) of grains is mainly parallel to the zone axis of hcp $< 100 >$ and hcp $< 110 >$, which is consistent with texture measurement by x-ray diffraction. SAD also indicates that short axes of elongated grains is the c-axis of hcp structure, i.e. grains are elongated in the orthogonal direction to the c-axis. In addition, several these large elongated grains compose a grain bunch, within which grains are parallel to each other. Phi scan at grazing incidence x-ray diffraction (GID) geometry reveals that these grain bunches are, as a whole, randomly oriented, but neighboring bunches are misoriented about 10-20 degrees with each other, which is consistent with TEM observation. The anomalous grain growth of CoCrMn thin film is closely related to the nucleation mechanism and starts from very early stage of film formation, which is different from the generally known secondary (or abnormal) grain growth. Qualitatively, it is understood based on the surface energy minimization and the details will be discussed.

J4.5
MAGNETIC FILM CHARACTERIZATION BY RBS, NRA, HFS AND PIXE. Luncun Wei, Charles Evans & Associates, Sunnyvale, CA.

Characterization of thin magnetic coating layer is always challenging, combination of different analytical methods are required to characterize layer structure and composition. In the present paper, RBS, PIXE, hydrogen forward scattering (HFS) and nuclear reaction analysis (NRA) are used to measure three typical magnetic film structure and coating layer. Carbon, oxygen, nitrogen contents are measured by deuteron NRA and hydrogen content is measured by HFS. Magnetic layers beneath carbon cover layer are characterized by RBS, Ni, Co, Fe, Cr contents are also checked with PIXE. Our analytical results show that combination of these four methods can give complete and precise layer structure and composition.

J4.6
INFLUENCE OF LINE-WIDTH ON MICROSTRUCTURE AND TEXTURE OF DAMASCENE COPPER INTERCONNECTS. Kabir Mirpuri, Jerzy Szpunar, McGill Univ, Dept of Metals and Materials Engineering, Montreal, CANADA; Kris Kozaczek, Hypernex Inc, State College, PA.

The structure, texture and grain boundary character distribution in the copper interconnects for the line-widths varying from 0.35 to 100 μm was studied. Field Emission Gun -SEM orientation imaging microscopy was used in this investigation. The shape of grains changed with increasing line-width. The grains were confined to grow along the trench length in the submicron lines wherein a bamboo grain structure was observed with only one grain along the width. More than one grain along the width were observed in the lines starting from 1 μm. The grain size increased with increasing line-width. The mean grain size was found to be 0.23 μm in the smallest lines with width 0.35 μm and as high as 4.33 μm in the lines with width 100 μm. The grain size distribution was monomodal and skew symmetric with an inclination towards smaller size. The inverse pole figure and the area fraction of the grain boundaries with different misorientation angle has been determined for all the line-widths. The strength of the (111) component increased with increasing line-width from 0.5 μm and decreased with increasing line-width from 0.35 to 0.5 μm. The lines with widths 0.35 and 0.4 μm had the (111) component in the direction transverse to the trenches much stronger than the normal direction. The lines with higher width (36, 40 and 100 μm) had (111) fiber texture. Low angle grain boundaries were present in smallest amounts in all the line-widths. Grain boundaries with misorientation angle lying between 46-60° were present in the highest amount. The $\Sigma 3$ and $\Sigma 9$ coincidence site lattice (CSL) boundaries were present in significant number in all the investigated lines. Twins were observed in all the lines.

J4.7
RATE-EQUATION BASED MODELLING OF ION BEAM ASSISTED HOMOEXPITAXY ON LOW-INDEX SURFACES OF Ag AND Cu. J. Sillanpää, N. Grønbech-Jensen, University of California, Davis, Dept. of Applied Science, Davis, CA; I.T. Koponen, University of Helsinki, Dept. of Physical Sciences, Helsinki, FINLAND.

We study surface growth on several low-index surfaces of Ag and Cu with rate-equation simulations. We take into account adatom and island diffusion, adatom detachment, vacancy diffusion and aggregation of vacancy islands and the recombination of adatoms and vacancies. We demonstrate how different processes affect the microstructure of the film and assess their relative importance. We compare results obtained with potential barriers from surface embedded-atom method (SEAM) calculations with those obtained using barriers from effective method theory (EMT), and discuss the suitability of these models for calculating barriers for surface simulation programs.

J4.8
MAGNETIC PROPERTIES AND MICROSTRUCTURES OF ANNEALED FeBSi THIN FILMS PREPARED BY DC MAGNETRON SPUTTERING. Jong-Wook Hong, Jong-Wan Park, Division of Materials Science and Engineering, Hanyang University, Seoul, KOREA; Taesuk Jang, Division of Metallurgical and Materials Engineering, Sunmoon Uni., Asan Si, Choongnam, KOREA.

Recently magnetostrictive materials have drawn significant interest in sensor and actuator applications. Metglas ribbons (Fe-based amorphous alloys) are the best-known candidates for magnetostrictive sensors in an electronic article surveillance (EAS) system due to the low coercivity and high saturation magnetization of the amorphous films. But at present, these alloys are available only in the form of thin ribbon ranging from 15 to 50 μm in thickness, which is a limit to the micro sensor application. Consequently, magnetoelastic materials in the form of thin film need to be developed for the miniaturization of sensors. Therefore, in this study, we examined the microstructure and magnetic properties of the annealed amorphous FeBSi films in order to understand the relationships between the composition, microstructure and magnetic properties for micro sensor application. The specimens with 1000 Å in thickness were prepared by DC magnetron sputtering and annealed at relatively high vacuum furnace to prevent oxidation. The changes in microstructure after annealing the thin films were characterized by X-ray diffraction pattern and transmission electron microscope. The intensity of magnetization was enhanced by a meta-stable phase about 20% at 400°C, which was reduced gradually by the crystallization from 500°C. However, the coercivity became higher extremely by about 250 Oe from 400°C to 500°C, and then the formation of stable Fe-B compounds which occurred during annealing at 600°C caused the coercivity to decrease by about 150 Oe. Our results indicate that amorphous thin films can undergo crystallization in response to oxidation at temperatures well below the bulk crystallization. In addition, two phases with different microstructure effect on the coercivity and magnetization separately in the amorphous FeBSi thin films, so we will be able to control the magnetic properties by the annealing temperature.

J4.9
THE DEPENDENCE OF TEXTURE BEHAVIOR IN Zr AND ZrN ON DIFFUSION-BARRIER PROPERTIES. Chuan-Pu Liu, Heng-Ghieh Yang, Department of Materials Science and Engineering, National Cheng-Kung University, Tainan, TAIWAN.

ZrN has a lower negative formation energy and a lower resistivity than TaN. Therefore, Zr/ZrN system could be a good candidate for the diffusion barriers in IC technology. In this paper, Zr or ZrN were deposited on Si(001) substrates by DC magnetron sputtering and

their properties and structures were analyzed by α-Step, 4-point probe, electron probe microanalysis, X-ray diffraction and transmission electron microscopy. The results indicate that both the resistivity and texture of the resulting films are sensitive to substrate bias, temperature and orientation. For example, the preferred orientations of Zr films change from (002) to random orientation when the substrate bias increases from 0 to 140V while the resistivity becomes lower. The mechanism of texture formation is discussed. Subsequently, we apply the different textured-Zr(N) films on Cu/Zr(N)/Si systems to examine the dependence of film texture on the diffusion-barrier properties. The failure mechanisms from different structures are discussed. The results imply that the appropriate texture structures of Zr and ZrN can be obtained by controlling substrate bias, temperature and orientation.

J4.10
VOID AND HILLOCK FORMATION IN Cu FILMS. Yasuyuki Kusama, David P. Field and Tejodher Muppidi, School of Mechanical and Materials Engineering, Washington State University, Pullman, WA.

Structure evolution during thermal cycling of Cu films and lines can lead to stress voiding and hillock formation that adversely affect manufacturability of Cu based IC interconnect lines. The position and character of these voids and hillocks is a function of Cu microstructure among other factors. To determine the local structure of Cu films where voids or hillocks preferentially form, several pad regions of test structures processed using conventional techniques (barrier, seed, ECD, CMP) was completely characterized using orientation imaging in the SEM. All films consisted of strong {111} fiber textures with virtually no twin grain boundaries observed in the microstructure. The structures were then heated to 450°C and cooled again in a positive pressure Ar environment. The positions of the resulting voids and hillocks were correlated with the original microstructure. The results show a preference for voiding at triple junctions consisting of high angle < 111 > tilt boundaries. Percolation theory is used to aid in interpretation of results.

J4.11
SURFACE EVOLUTION OF ZnO/SAPPHIRE(0001) FILMS DURING HIGH TEMPERATURE ANNEALING. In Woo Kim, Seok Joo Doh, Department of Materials Science and Engineering, POSTECH, Pohang, KOREA; Jung Ho Je, Department of Materials Science and Engineering, POSTECH, Pohang, KOREA and Materials Science Division, Argonne National Laboratory, Argonne, IL; Tae Sik Cho, Department of Materials Science and Engineering, Sangju National University, Sangju, KOREA; Byung Mook Weon, Gong-Seog Park, and Soo-Deok Han, Display Device Research Lab., LG Electronics Inc., KOREA.

The surface evolution of ZnO/sapphire(0001) heteroepitaxial thin films during high temperature annealing has been studied using real-time synchrotron x-ray scattering and atomic force microscope. We found that the evaporation as well as grain growth plays a crucial role for the evolution of textured ZnO/sapphire(0001) film surface. The preferential evaporation on protruding grains at high temperature makes initial epitaxially textured feature of ZnO surface flat, and measured evaporation rate was 0.33Å/min at 800°C. Active grain growth at high temperature generate pores, which located between growing grains on the surface of the ZnO/sapphire(0001) films. Finally we discuss the detailed microstructural evolution of epitaxially textured ZnO/sapphire(0001) surface at high temperature from the viewpoint of kinematics.

J4.12
THE IMPORTANCE OF THE TEXTURE ON THE LIFETIME DETERMINATION OF THE THIN FILM INTERCONNECTS WITH BAMBOO STRUCTURE BY THE VOID INTERGRANUAL MOTION. Ersin Emre Oren, Tarik Omer Ogurtani, Middle East Technical Univ, Dept of Metallurgical and Materials Engineering, Ankara, TURKEY.

In these studies irreversible thermokinetic formulation of the generalized forces and conjugate fluxes are used associated with the void - grain boundary interaction. A well - posed moving boundary value problem describing the kinetics of void intergranual motion is solved by using indirect boundary element technique for various combinations of texture and electromigration forces in thin film metallic interconnects with bamboo structure. Extensive computer simulations have been performed on the configurational changes associated with voids during the intergranual motions in two dimensional space, under the actions of capillary effect, and electron wind in unpassivated interconnects. As a result, very rich interaction morphologies such as penetration, trapping, detrapping, fragmentation and daughter void formation are observed and their respective threshold values and failure lifetimes are determined. The texture studies in this work show that there are two different and very distinct modes of failure, namely: the grain boundary carving or tearing mode, and the interconnect edge cutting mode by the oblique slit formation on the wind-side of the grain boundary. Similarly, the grain texture on the both sides of the grain boundaries containing trapped void or cavity is very important parameter dictating not only the mechanism of the fatal open circuit configurations but also determining the failure lifetime of the sample at least by a factor of three.

SESSION J5: ELECTRONIC THIN FILMS I
Chair: Mark D. Vaudin
Wednesday Morning, April 3, 2002
Nob Hill A/B (Marriott)

8:00 AM *J5.1
ON THE ANALYSIS AND MANIPULATION OF CRYSTALLOGRAPHIC ORIENTATION IN SLS-PROCESSED Si, Cu, AND Al FILMS. James S. Im, B.A. Turk, J.B. Choi, P. Ch. Van der Wilt, J. Nakayama, M.A. Crowder, R.S. Sposili, and A.B. Limanov, Program in Materials Science and Engineering, Department of Applied Physics and Applied Mathematics, School of Engineering and Applied Science, Columbia University, New York, NY.

The sequential lateral solidification (SLS) process is a pulsed-laser based thin-film-processing method that can be utilized in order manipulate the microstructure of thin films. Depending on the details of the process (i.e., the shape of the patterned beamlets and the microtranslation sequence between the laser pulses), it is possible to obtain a variety of microstructures ranging from grain-boundary-location-engineered polycrystalline films to location-controlled single-crystal regions. SLS is noteworthy because it can offer more flexibility for explicit microstructural manipulation and optimization of the films than can be attained using direct deposition and/or various solid-phase-phenomena based processes (such as grain growth of polycrystalline films or solid-phase crystallization of amorphous films). In this paper, we will review the previous work and present recent findings related to the subject of crystallographic orientations in SLS-processed Si, Cu, and Al films. SLS of Si films is being investigated for manufacture of high-performance TFTs, while SLS of Cu and Al films is being pursued for improving the materials interconnect performance. It is known that effects arising from the texture of the films can play a role in some of these applications. The samples were processed using an excimer-laser-based SLS system with projection optics, and were subsequently analyzed using TEM, SEM, EBSP, and AFM techniques. In addition to presenting the results of the microstructure and orientation analyses, we will also discuss: (1) the phenomenon of continuous change in crystal orientation during lateral solidification of the films, (2) the effect of pre-existing textures in polycrystalline precursor films, as well as (3) various technical schemes for further selecting and controlling the final texture of the SLS processes films which can range from engineering of the precursor material to exploiting various aspects of melting and solidification phenomena.

8:30 AM J5.2
TEXTURE TAYLORING IN LASER-CRYSTALLIZATION OF SILICON THIN FILMS ON GLASS SUBSTRATES. M. Nerding, S. Christiansen, Institute of Microcharacterization, Univ. Erlangen, GERMANY; R. Dassow, K. Taretto, J.R. Köhler, Institute of Physical Electronics, Univ. Stuttgart, GERMANY; H.P. Strunk, Institute of Microcharacterization, Univ. Erlangen, GERMANY.

We investigate the texture of polycrystalline silicon thin films. These films are produced by laser crystallization of amorphous silicon on glass substrates by scanning a frequency doubled $Nd:YVO_4$-laser beam with a wavelength of 532 nm. Transmission electron microscopy reveals that the grains have an average width between 0.25 - 5 μm depending on the crystallization parameters and a length of several 10 μm. Electron back-scattering diffraction experiments indicate a textured grain population in the poly-Si films. Type and extent of texture depend on the thickness of the amorphous silicon layer, on the repetition rate of the laser pulses and on whether or not an additional buffer layer is present on the glass substrate. For crystallization on a silicon nitride buffer layer or crystallization with high repetition rates we observe a strong preferential <111> orientation of the surface normal. Crystallization of amorphous silicon with film thicknesses below 100 nm leads to a marked <110> surface normal with a preferential alignment of the grains along <100> within the films. We can explain texture formation in terms of interface energies and lateral overgrowth and thus provide a means for tayloring of the texture and consequently of the grain boundary population.

8:45 AM J5.3
TEXTURE OF POLYCRYSTALLINE MoS_x THIN FILMS REACTIVELY SPUTTERED FROM A MOLYBDENUM TARGET IN Ar-H_2S ATMOSPHERES. Volkmar Weiss, Klaus Ellmer, Hahn-Meitner-Institut, Dept. Solare Energetik, Berlin, GERMANY.

The van der Waals type layered semiconductors MX_2 (M = Mo, W; X = S, Se) have been proposed as absorber materials in thin film solar cells and proved to be photoactive when grown with texture promoting agents like Ni, CrNi or Co. Since reactive magnetron sputtering from metallic targets in mixtures of argon and a reactive gas (e.g. O_2, H_2S) is a well established technique for thin film deposition of oxides and sulfides we investigated this method for the deposition of MoS_x films. The depositions on glass, quartz and pyrolytic carbon substrates were performed at substrate temperatures up to 800 K and have been carried out from a molybdenum target with 4 inch diameter at total sputtering pressures of the Ar-H_2S mixtures from 0.1 - 0.4 Pa. Depending on the H_2S partial pressure MoS_x films with x = 0.5 - 1.8 were prepared. The highest sulfur content of the films could be achieved only for H_2S-to-Ar flow ratios higher than 1.4. The films showed n-type semiconductivity behaviour and had resistivities from 0.5 - 2.5 Ω·cm for $x \geq 1.0$. Films with thicknesses < 300 nm exhibited a pronounced (001)-texture of the 2H-MoS_2 phase crystallites as revealed by conventional angle dispersive XRD. However, from the position of the (002) reflection an enlargement of the c-lattice constant by up to 11% was inferred. For thicker films or films deposited at low deposition rates a crossover from the (001) to the (100) texture was observed, which is believed to be due to intrinsic stress in the films. The texture change has been confirmed by scanning electron microscope analysis. Furthermore, the film growth especially the texture crossover from the (001) to the (100) orientation was investigated in situ by energy dispersive X ray diffraction (EDXRD) at the synchrotron radiation source HASYLAB (Hamburg/Germany).

9:00 AM J5.4
RAPID LOW COVERAGE BIAXIAL TEXTURE DEVELOPMENT IN MgO FILMS DURING GROWTH ON AMORPHOUS SUBSTRATES BY ION BEAM-ASSISTED DEPOSITION.
Rhett Brewer and Harry A. Atwater, California Institute of Technology, Pasadena, CA.

High-quality, biaxially textured MgO can be grown on amorphous Si_3N_4 using ion beam-assisted deposition (IBAD). Previous studies of texture evolution have hinted that biaxial texture develops during the nucleation stage preceding MgO film coalescence, but these studies have lacked the sensitivity to determine the nature of the texture evolution in this regime. We report a previously unseen, dramatic precoalescence texture evolution in IBAD MgO based on quantitative texture measurements at very low coverage derived from reflection high energy electron diffraction (RHEED) based biaxial texture analysis techniques. We find specifically that the earliest stage of MgO growth exhibits an almost random out-of-plane texture up to a thickness of 3.5 nm. During the next 1 nm (~5 monolayers) of additional growth, a sudden and striking onset of texture evolution is observed, resulting in a transition from very broad out-of-plane distribution to a well-oriented fiber texture. The in-plane orientation distribution exhibits a similarly rapid evolution over the same thickness interval. We have examined film morphological evolution during this sharp biaxial texture transition using RHEED, transmission electron microscopy, and atomic force microscopy. *In situ* RHEED measurements of biaxial texture made during film growth, while varying the Ar^+ energy from 300 to 1000 eV, the substrate temperature from 25° to 600°C, and the ion/MgO molecule flux ratio from 0.2 to 0.6, illuminate biaxial texture development mechanisms and suggest strategies for biaxial texture optimization. IBAD MgO films are potentially important as a buffer layer for integration of active oxide materials such as $BaTiO_3$ with a Si integrated circuit compatible process. Issues associated with the biaxial texture of $BaTiO_3$ grown on IBAD MgO template layers will be discussed.

9:15 AM J5.5
TEXTURE AND MICROSTRUCTURE OF YBCO FILMS: HIGH ACCURACY CHARACTERIZATION OF MISORIENTATIONS BY EBSD.
John A. Sutliff and Xiaodong Han, HKL Technology, Burnt Hills, NY; J.L. Reeves, IGC SuperPower, Schenectady, NY.

Eliminating microstructural barriers to supercurrent is key to making the high temperature superconductor $YBa_2Cu_3O_x$ (YBCO) more favorable for commercial applications in the electric power industry. Crystallographic boundaries (grain, sub-grain, and twin) are some of the possible barriers to current flow. The number density, spatial distribution, and misorientation character of crystallographic boundaries are the critical microstructural parameters that need to be controlled via processing to achieve consistent high performance material. We demonstrate the quantification of the above-mentioned parameters with high accuracy using novel methods in automated Electron Backscatter Diffraction (EBSD) technology. Particularly, the use of improved diffraction pattern feature detection allows very low misorientation structures to be revealed. Measurements of microstructure and texture from YBCO films on both single crystal and polycrystalline substrates will be presented.

9:30 AM J5.6
GROWTH OF VO2 THIN FILMS ON GERMANIUM, SAPPHIRE AND LITHIUM NIOBATE SUBSTRATES BY PULSED LASER DEPOSITION.
Guy Garry, Olivier Durand, Albert Lordereau, Ceramics & Packaging Dept, Thales Research & Technology, Orsay, FRANCE.

Vanadium dioxide (VO2) thin films have been deposited on Ge (111), Al2O3 (00.1) and LiNbO3 (00.1) substrates using a Pulsed Laser Deposition technique. Growth conditions were optimized to obtain epitaxial growth. X-ray diffraction analysis revealed the highly (010) texture of VO2 thin layers on all three substrates. However these observations also showed that the monoclinic structure of this oxide becomes tetragonal when the in-plane lattice parameter mismatch value is the lowest (case of LiNbO3). This suggests the existence of stresses at the interface which force the material into the highest symmetry state. Such behavior is also observed on bilayers MgO (111) / VO2 (010) deposited on Al2O3 (00.1) for VO2 layers of thicknesses less than 20 nm. To explain this effect one has also to take into account the influence of the linear expansion coefficient differences between the substrates and the thin films which induce thermal stresses during cooling. In all cases, DC resistivity and optical properties in the 0.2 to 18 μm range have been measured as a function of temperature between room temperature and 100°C. These physical properties are discussed in relation to the crystalline quality of VO2 thin films.

SESSION J6: ELECTRONIC THIN FILMS II
Chair: Stuart Wright
Wednesday Morning, April 3, 2002
Nob Hill A/B (Marriott)

10:15 AM *J6.1
MODEL RELATING THIN FILM PZT CRYSTALLOGRAPHIC TEXTURE TO FERROELECTRIC SWITCHING PERFORMANCE.
G.R. Fox, Ramtron International Corporation, Colorado Springs, CO; S. Summerfelt, Texas Instruments Corporation, Dallas, TX.

The fundamental physical property used for data storage in a ferroelectric non-volatile memory (FRAM) is the remanent polarization of a ferroelectric capacitor. In order to sense the data state of the capacitor, the charge pulse generated by either a switching or non-switching ferroelectric polarization is detected. Since the polarization of the ferroelectric is a vector property, the switchable polarization response of a ferroelectric capacitor is necessarily dependent on the texture of the ferroelectric material used to make the capacitor. A series of Ca, Sr, and La doped $PbZr_{0.4}Ti_{0.6}O_3$ (PZT) capacitor test samples were produced with texture ranging from nearly complete {111} texture to random orientation. The volume fraction of the textured material was quantified by X-ray diffraction. An experimental correlation between texture and the switchable polarization was established using pulse switching measurements. A mathematical model was developed to explain the dependence of the switchable polarization on the crystallographic texture. It was found that the dependence of the saturated switchable polarization on texture required that there was no 90° domain reorientation. The impact of these findings will be related to the performance of FRAM.

10:45 AM J6.2
STRUCTURE AND MORPHOLOGY OF REACTIVELY SPUTTERED $In_{0.9}Sn_{0.1}O_x$ LAYERS.
R. Mientus, Opto-Transmitter-Umweltschutz-Technologie e.V., Berlin, GERMANY; I. Sieber, K. Ellmer, Hahn-Meitner-Institut, Dept. Solarenergieforschung, Berlin, GERMANY.

Indium-tin oxide (ITO) films are widely used as transparent conductive electrodes, especially in flat panel displays based on liquid crystals, micro plasmas or light emitting polymers. The technique, mostly used for the deposition of ITO films is DC magnetron sputtering from ceramic targets ($In_2O_3$90%$SnO_2$10%). The ceramic targets are expensive and fragile. Therefore, for mass production, DC or pulsed DC reactive magnetron sputtering from a metallic InSn target would be very promising. In this work, the influence of discharge parameters like oxygen partial pressure, discharge power and plasma excitation on the structure and morphology of the deposited films is investigated. The films were deposited onto unheated substrates with a balanced magnetron source of 2 inch diameter and with a target-to-substrate distance of 60 mm. The oxygen partial pressure was varied in order to deposit $In_{0.9}Sn_{0.1}O_x$ films with $0 \leq x \leq 1.76$. The composition x was measured by Rutherford backscattering (RBS). For low x values, these films are metallic and opaque, while at $x \approx 1.5$ the layers exhibit good properties as transparent and conducting electrodes. Further increase in x leads to

transparent insulating films. The morphology of the films, investigated by SEM and AFM shows significant variations with the composition x. While the metallic films consist of coarse globular grains, made up of polycrystalline indium-tin, a small addition of oxygen leads to nearly amorphous metal-oxide mixtures with smooth surfaces. Around x≈1.5 the films are polycrystalline with the cubic In_2O_3 bixbyite structure and show the resistivity minimum. The cross sectional morphology of these films shows a columnar structure of broad bundles (≈250 nm), composed of narrow needle-like crystallites of about 20 nm diameter. At very high oxygen partial pressures, the grain size decreases while the strain and the resistivity increase significantly.

11:00 AM J6.3
TRANSMISSION ELECTRON MICROSCOPY STUDY OF ULTRAVIOLET-ASSISTED PULSE LASER DEPOSITED INDIUM TIN OXIDE FILMS. Nabil D. Bassim, Valentin Craciun, and Rajiv K. Singh, University of Florida, Department of Materials Science & Engineering, Gainesville, FL.

Indium tin oxide is a useful material because of both its optical and electrical properties as a transparent conducting oxide. In order to reduce the processing thermal budget and enhance compatibility of these films for such applications as transparent electrodes for solar cells and flat panel displays, lower deposition temperatures are desirable. The addition of a non-thermal energy source during deposition, in this case a mercury lamp, has the ability to lower the required substrate temperature during processing while increasing the oxygen content of the deposited film through the added contribution of ionized species into the deposited material. We investigated the microstructure of UV-assisted pulsed laser deposited indium tin oxide films as a function of substrate temperature and oxygen pressure in the deposition chamber by transmission electron microscopy and related this microstructure to the electrical and optical properties of the films, namely sheet resistance, hole mobility, refractive index and extinction coefficient. Further comparisons were made between the UV and non-UV films to judge the effectiveness of the UV radiation in achieving desirable properties.

11:15 AM J6.4
UTILIZATION OF A NEW LIQUID-STATE PRECURSOR FOR THE DEPOSITION AND SYSTEMATIC STUDY OF THE INTERRELATIONS OF STRAIN, TEXTURE AND TRANSPARENT CONDUCTING PROPERTIES IN MOCVD DERIVED EPITAXIAL CdO THIN-FILMS. Andrew W. Metz, Kenneth R. Poeppelmeier, Tobin J. Marks, Department of Chemistry and the Materials Research Center, Northwestern University, Evanston, IL; John R. Ireland, Melissa A. Lane, Carl R. Kannewurf, Department of Electrical and Computer Engineering and the Materials Research Center, Northwestern University, Evanston, IL.

Epitaxially grown transparent conductors have received little attention in the literature owing to assumptions that grain boundary interactions in degenerately doped materials should have little effect on properties. Recent work has shown that more study is needed to elucidate these effects. We have synthesized a new low-melting, highly volatile and thermally stable cadmium MOCVD precursor for the detailed study of the effects that strain, texture, and microstructure can have on the properties of high-mobility cadmium-based TCOs. MOCVD-derived films of CdO on MgO(100) show significant lattice compression that relaxes to bulk values in thicker films owing to the large (8.7%) lattice mismatch. This relaxation in lattice parameter is accompanied by increased crystallinity and texture, having dramatic effects on the optical and charge transport characteristics of these films. Mobilities as high as 320 $cm^2/V*s$ have been achieved at relatively low growth temperatures.

11:30 AM J6.5
INFLUENCE OF OXYGEN ADDITION AND SUBSTRATE TEMPERATURE ON TEXTURED GROWTH OF Al DOPED ZnO THIN FILMS PREPARED BY RF MAGNETRON SPUTTERING. P. Kuppusami, Indira Gandhi Centre for Atomic Research, Kalpakkam, INDIA; K. Diesner, I. Sieber, K. Ellmer, Hahn-Meitner-Institut, Dept. Solarenergieforschung, Berlin, GERMANY.

Aluminium doped zinc oxide films have been increasinly used as transparent electrodes in flat panel displays and thin film solar cells owing to higher carrier concentration, electrical conductivity and mobility in comparison with those of undoped ZnO films. The high carrier concentration in these films is attributed to the electrons donated from $Al3^+$ ions on the substitutional sites of $Zn2^+$ ions. The electrical and optical properties of ZnO:Al films are not only determined by the dopant concentration in the films but also by an interplay between the texture, microstructure and the state of strain achieved in them. These properties are strongly influenced by process parameters. We report here the influence of oxygen content in the range 0.04-0.4% and substrate temperature in the range 300-723K on the textured growth of ZnO:2wt%Al on soda lime glass substrates grown by partially reactive radio frequency magnetron sputtering. The films grown with oxygen content of 0.2% show minimum resitivity of $1.2x10^{-2}$ Ω·cm and about 85% optical transmission. The films grown with oxygen content less than 0.2% have low transmission due to metallic inclusions in the films. Higher oxygen contents in the sputtering atmosphere (>0.4%) lead to films with a high resistivity and high optical transmission. Obviously, the dopant Al is also oxidised leading to a low carrier concentration. SEM micrographs indicate columnar growth and a smooth surface for the films grown with 0.1-0.3% oxygen content. The films grown at low substrate temperatures with higher resistivities ($8.65x10^{-3}$ Ω·cm at 373K) show better transmission for uv-visible and near infrared region than the films obtained at high substrate temperature with low resistivities ($1.5x10^{-3}$ Ω·cm at 673K). It is seen from the SEM micrographs that columnar growth becomes more dense and dominant and the surface becomes more rough with increasing substrate temperature.

SESSION J7/E4: JOINT SESSION
MICROSTRUCTURAL AND PROCESSING ASPECTS OF MAGNETIC THIN FILMS
Chairs: Li Tang and Jerzy A. Szpunar
Wednesday Afternoon, April 3, 2002
Nob Hill A/B (Marriott)

1:30 PM J7.1/E4.1
GROWTH AND TEXTURE OF GaMnAs GROWN ON GaAs BY MOVPE. Kerstin Volz, Michael Lampalzer, Torsten Torunski, Wolfgang Stolz, Philipps University Marburg, Materials Science Center, Marburg, GERMANY; Leander Tapfer, PASTIS CNRSM, Brindisi, ITALY.

Magnetic semiconductors are important for the progress of magneto-optics and spin-opto-electronics. In this study, GaMnAs films have been grown successfully on GaAs substrates under non-equilibrium conditions by metal organic vapour phase epitaxy (MOVPE). The layers exhibit a high structural perfection, as will be shown by X-ray diffraction and mainly by high resolution transmission electron microscopy (TEM). Depending on the growth conditions, clusters are found in these films. These clusters are responsible for the ferromagnetic coupling of the films, also observed at temperatures exceeding room temperature. The clusters are shown to have hexagonal NiAs crystal structure and contain a considerable amount of Ga. Despite of the different crystal structure from the surrounding matrix, the hexagonal GaMnAs clusters as well as the surrounding GaAs:Mn matrix grow defect-free. A heteroepitaxial relationship to the cubic matrix crystal can be observed for all clusters. Depending on the growth temperature and the substrate orientation, different cluster shapes as well as different epitaxial relationships of the clusters to the surrounding GaAs:Mn are found. The texture of these films will be shown in dependence on the growth conditions and will be correlated to the magnetic properties.

1:45 PM J7.2/E4.2
BULK VERSUS SURFACE MAGNETIC TEXTURE IN THIN FILMS OBTAINED BY PULSED LASER DEPOSITION. Monica Sorescu, Duquesne University, Pittsburgh, PA; A. Grabias, Institute of Electronic Materials Technology, Warsaw, POLAND; D. Tarabasanu-Mihaila and L. Diamandescu, Institute of Atomic Physics, Bucharest, ROMANIA.

Pulsed laser deposition has been extensively used in obtaining thin films of ferroelectrics and superconductors. Recently, it became a promising method for fabricating thin films from several classes of magnetic materials, such as metallic glasses. In this regard, laser ablation deposition of iron oxides and intermetallics both with and without substitutions represent a challenging approach and was undertaken in the present study. Using X-ray diffraction, transmission and conversion electron Mössbauer spectroscopy, the magnetic properties of these systems are related to those of the corresponding bulk materials. In this study we present a direct comparison between the properties of bulk magnetic systems and those of corresponding thin films obtained by pulsed laser deposition: $Fe_{50}Co_{50}$, Fe_3O_4, Fe_3O_4:Co and Fe_2O_3:Cr. Using complementary techniques, we present results on the bulk and surface hyperfine magnetic fields, site populations and magnetic texture. Our results support an increase in the hyperfine field values at the surfaces, a more pronounced out-of-plane magnetic texture in the films, as well as a perfect transfer of stoichiometry and substitution level from target to substrate materials.

2:00 PM J7.3/E4.3
EVOLUTION OF FREE VOLUME IN ULTRASOFT MAGNETIC FeZrN FILMS DURING THERMAL ANNEALING. N.G. Chechenin[a], A. van Veen[b], H. Schut[b], A.R. Chezan[a], D.O. Boerma[a], T. Vystavel[a],

J. Th. M. de Hosson[a]; [a]Materials Science Centre, University of Groningen, Groningen, NETHERLANDS; [b]Interfaculty Reactor Institute, Delft University of Technology, Delft, NETHERLANDS.

Ultrasoft magnetic films with low a coercive magnetic field of ~1 Oe, an induced uniaxial magnetic anisotropy of 10-50 Oe and a high saturation magnetization of ~1.5-2 T, are required for future high frequency applications in the GHz region. We obtain excellent magnetic parameters in FeZrN films, deposited by DC reactive sputtering on different substrates at various temperatures. The grain size is a crucial parameter of the microstructure of a magnetic film: nanometer size of the grains is required for averaging out the magnetocrystalline anisotropy and for reduction of the intrinsic stray field. The films were studied with XRD, TEM and positron beam analysis. The films with the best soft magnetic properties were almost amorphous: the grain size estimated using XRD was about 2-3 nm, while HRTEM showed only a weak ordering. The main emphasis of this study was on nano-grain formation, grain growth and evolution of the intergranular free space during a thermal treatment in the temperature range up to 300°C. The microstructural evolution was investigated by HRTEM and by Lorentz TEM using an in-situ heating stage. Positrons proved to be a sensitive probe for the free volume in the near-amorphous and recrystallized films. Due to the nanometer size of the grains nearly all positrons diffuse to the grain boundary and are trapped and annihilated there. The size and character of the free volume on an atomic scale was monitored for films with different deposition parameters and its evolution is followed during thermal annealing, where two competing processes are involved: the reduction of the free space by the formation of dense grains and by a decrease of intergranular space during the grain growth on one side, and an increase of the free volume due to nitrogen release, on the other side.

2:15 PM *J7.4/E4.4

MICROSTRUCTURAL EFFECTS IN SINGLE LAYER AND ANTIFERROMAGNETICALLY-COUPLED MAGNETIC MEDIA. Mary Doerner, Kai Tang, Qi-Fan Xiao, IBM Storage Technology Division, San Jose, CA; Shaun McKinlay, Robert Sinclair, Stanford Univ, Stanford, CA.

Microstructural control for magnetic recording media is becoming ever more critical with the rapid decrease in bit dimensions and the threat of thermal instability. The crystallographic texture and grain size of the Co-alloy magnetic layer is obtained through epitaxy with the underlying layers. Improvement of the grain size distribution and texture in the Cr-alloy underlayer is therefore a key priority, however, previous researchers have shown that the epitaxial relationship between the underlayer and magnetic layer is not perfect. This can result in an uncontrolled microstructure in the magnetic layer. One example of this effect and its explanation is discussed for CoPtCrB on Cr-alloy underlayers. Antiferromagnetically-coupled (AFC) media are used to improve thermal stability. Since at least part of the increase in stability is obtained through increased thickness of the top magnetic layer, optimum performance is obtained for microstructures that maintain recording properties as a function of magnetic layer thickness. The implications of this will be discussed and demonstrated for different microstructures.

3:15 PM J7.5/E4.5

QUANTITATIVE NANOMETER-RESOLUTION MAPPING OF BORON IN MAGNETIC RECORDING MEDIA BY ANALYTICAL ELECTRON MICROSCOPY. J. Bentley, N.D. Evans, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN; J.E. Wittig, J.F. Al-Sharab, Vanderbilt University, Nashville, TN.

Measurements of composition at the nanometer scale are critical for structure-property correlations aimed at understanding the performance of Co-Cr-based thin-film longitudinal magnetic recording media. We have previously established and applied reliable methods for mapping Cr segregation by energy-filtered transmission electron microscopy (EFTEM). Of particular interest is the extension of such work to map B distributions. Two methods are being employed: EFTEM mapping with a Gatan imaging filter (GIF) on a LaB_6 Philips CM30 and spectrum imaging in the scanning transmission electron microscopy (STEM) mode on a Philips CM200FEG with Emispec ESVision and a GIF. For B measurements, the presence of M-edges from 3d transition metals (TM) results in low signal-to-background (S/B) and a background shape that does not follow an AE^{-r} inverse power law. Previous quantitative analysis of TM borides was successful with the use of a log-polynomial (LP) background fitting procedure in which a low-order polynomial was fit to log(intensity) versus log (energy-loss). For Co-Cr-Pt-B media, limited success has been achieved employing such an approach with spectrum imaging in STEM. However, Cr increases and Co decreases concurrent with local B enrichment appear to subtly change the background shape, leading to limited reliability at low B levels, even with LP background fitting. In EFTEM methods, multiple acquisitions are required to increase the detected signal in order to overcome the low S/B statistical limitations. In addition, rather than using the conventional 3-window method, custom scripts within the Gatan DigitalMicrograph software have been developed for optimized acquisition and LP background fitting from an extended set of energy-filtered images. Mapping B at ~1-nm resolution by EFTEM and spectrum imaging has clearly indicated inter- and intra-granular segregation. Continuing work on the remaining issues for robust quantitative B concentration mapping will be fully described. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC. Support from an IBM Faculty Partnership is also gratefully acknowledged.

3:30 PM J7.6/E4.6

SPIN-DEPENDENT QUANTUM SIZE EFFECTS IN THIN IRON MICROCRYSTALS. Ryszard Zdyb, Ernst Bauer, Arizona State University, Dept of Physics and Astronomy, Tempe, AZ.

The spin-dependent quantum size effects (QSE) of slow electrons reflected from atomically flat Fe microcrystals with well-defined thicknesss are studied by spin-polarized low energy electron microscopy. From the energy values of the extrema of the QSE oscillations between 0 and 20 eV the band structure of the exchange-split sp band along the [110] direction is derived. It agrees well with that calculated by Hathaway et al [1]. From the amplitudes of the oscillations as a function of energy and thickness the inelastic mean free paths (IMFP) of spin-up and spin-down electrons are obtained. Although their magnitudes agree qualitatively with recent calculations [2] their energy dependence does not: they increase from 5 - 6 eV up to the threshold of sp excitations at about 8 eV and then decrease again. This discrepancy is attributed to the simplified description of the sp contributions in the calculations and indicates that the excited state band structure has to be taken into account in the theory of the IMFP. [1] K.B. Hathaway et al, Phys. Rev. B31, 7603 (1985) [2] Jisang Hong and D.L. Mills, Phys. Rev. B62, 5589 (2000)

3:45 PM J7.7/E4.7

COMPARISON OF Pb, O_2, AND Ag SURFACTANTS IN SPUTTERED Co/Cu MULTILAYERS. Brennan L. Peterson, Bruce M. Clemens, Robert L. White, Stanford University, Department of Materials Science and Engineering, Stanford, CA.

The addition of so-called surfactants to thin films in order to improve their structural properties is an interesting and potentially, a particularly useful area of study. In the case of Co/Cu growth, a variety of surfactants have been used, in a variety of growth systems. Our specific interest is in sputter deposited systems, where surface stresses, intermixing, growth rates, and kinetics can be significantly different than in evaporated and CVD films. Because of the variety of surface growth phenomena involved, several techniques are necessary for a good understanding of the processes which govern growth. In order to quantify the effect of surface stresses and intermixing, in-situ stress measurements were undertaken. In order to quantify the structure of the film, *ex-situ* low angle x-ray scattering studies were combined with *in-situ* STM. In this study, we focused particularly on the difference between metallic surfactants (Ag, Pb) and oxygen. DC magnetron sputtered [Co/Cu] multilayers were deposited on SiO_2 and Pt/Al_2O_3 substrates. Typical rates were ~1Å/s. The Pt seed layers were grown at high temperatures, and provide a single crystal metal template for growth, with wide (~500Å) terraces. Surfactants were added at interfaces and at the base, in order to determine where surfactants were most efficient. Oxygen was introduced during growth at various partial pressures, as well as puffed onto interfaces. Both lead and silver move effectively to the surfaces, but nowhere near as efficiently as earlier work in evaporated films had shown. The addition of a partial pressure of oxygen during growth had the largest effect in increasing smoothness and layer to layer roughness correlation. At interfaces, the oxygen effect was minimal. We also compare simple models of the effect of surfactants on nucleation and growth, especially at the non-equilibrium rates of sputter deposition.

4:00 PM J7.8/E4.8

CONTROL OF NOVEL MAGNETIC PROPERTIES IN COBALT-BASED THIN FILMS BY USE OF MOCVD. Mariana Chioncel, Feodor Ogrin, Brian Ruthven, Peter Haycock, Jilan Shah, Anthony Wright, Gillian Maddison, Samantha Stockton, Keele University, School of Chemistry and Physics, Staffordshire, UNITED KINGDOM.

The magnetic properties of thin films can be modified significantly from those of the bulk material through careful control of the growth process. We have made use of conventional and assisted metal-organic chemical vapor deposition to control the occurrance of inverse magnetic hysteresis in cobalt-based films. Changing the process parameters, including the balance between photolytic and pyrolytic decomposition of the cyclopentadienylcobalt dicarbonyl precursor enables the development and control of a range of anomalous

magnetic properties. The occurrence of inverted hysteresis is now well established, but extant data on other magnetic properties of such systems is scarce. We present here the results of hysteresis measurements, remanence curves, ferromagnetic resonance studies, magneto-optic magnetometry and magnetotransport experiments on samples which exhibit large, fully inverted hysteresis loops when the magnetic field is applied perpendicular to the plane of the film. The anomalous properties can be correlated with the occurrance of a certain microstructure and texture, namely amorphous, randomly shaped grains of dimension around 150 nm. The remanence curves indicate that there is competition between different anisotropies in the film, each favouring a different orientation of the magnetization with respect to the applied field. Ferromagnetic resonance studies have confirmed the unconventional anisotropy and shown the presence of an extra, strong spin mode in the range of orientations within which the inverted hystesis is exhibited. Magnetotransport measurements have shown that there appears to be a strong in-plane moment re-orientation taking place when the hysteresis loop contains a section with negative dynamic susceptibility. In this paper we bring together the results from a wide range a measurements and discuss these in the light of the microstructural and textural features of the films. We also indicate how to control the microstructure through the growth process parameters.