SYMPOSIUM G

Polycrystalline Metal and Magnetic Thin Films

April 23 – 27, 2000

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* Invited paper

TUTORIAL

ST D-G-H: Cu INTERCONNECTS: WHAT ARE THE ISSUES?
Sunday, April 23, 2000
1:00 p.m. - 4:45 p.m.
Golden Gate B2 (Marriott)

The implementation of Cu in the metallization process of integrated circuits has redirected many research and development projects in universities, research institutes, and industry. A vast amount of data is being collected on the physical and mechanical properties of Cu thin films and lines, on various aspects of its polycrystalline nature. The implementation of Cu also goes hand in hand with the changeover from the physical vapor deposition technique to electroplating of the metal and with the introduction of chemical-mechanical polishing instead of the classical metal dry etch process. The research related to these new process steps has created a need to get more insight into other aspects of materials science.

The aim of this tutorial is to provide an interdisciplinary introduction to the latest evolution in fields relevant to Cu interconnects. An overview will be given of the state of the art of Cu metallization for high-performance Si technology. The importance of electrochemistry for the understanding of electrochemical deposition will be highlighted, and possible mechanisms of Cu corrosion will be discussed.

Instructors:
Robert Rosenberg, IBM T.J. Watson Research Center
Tom Moffatt, NIST
Vlasta Brusic, Cabot Corporation

SESSION G1/F3: JOINT SESSION: MAGNETIC RECORDING MEDIA
Tuesday Morning, April 25, 2000
Golden Gate B3 (Marriott)

8:30 AM *G1.1/F3.1
ADVANCED LONGITUDINAL RECORDING MEDIA USING HCP COBALT ALLOYS. Mary F. Doerner, Xiaoping Bian, Kai Tang, Mohammad Mirzamaani, Adam Polcyn, IBM Storage Systems Division, San Jose, CA; Kurt Rubin, Ken Takano, Michael F. Toney, Andreas Moser, Dieter Weller, IBM Almaden Research Center, San Jose, CA.

It is well recognized that thermal stability may place a limit on longitudinal magnetic recording areal density with predictions of a maximum data density limit ranging from 36 to 100 Gbits/in^2 depending on system assumptions such as bit aspect ratio and channel requirements for a given signal-to-noise ratio. Recently, IBM has demonstrated the capability to write and read data with excellent error rates at 35 Gbits/in^2 using magnetic media that are thermally stable. The very low signal decay rate of 0.63%/decade measured on a Squid magnetometer at 350 K and 500 Oe reverse field suggests that there is room for further increase in areal density using conventional Co-alloys. One of the key media improvements that contributed to achieving this good stability was an increase in the magnetocrystalline anisotropy (Ku) of the magnetic film. Although Ku as high as 3.7 x 10^6 ergs/cm^3 has been measured for polycrystalline HCP Co75Pt12Cr13, Ku decreases significantly when higher concentrations of Cr and the addition of Ta are used to reduce intergranular exchange coupling. This paper will discuss the issues associated with achieving high anisotropy in Co-alloys including the role of defects, segregation and crystallographic orientation. It is also believed that the capability of the head to write high coercivity will ultimately limit the usable Ku of magnetic media. Therefore, it is important that the media microstructure be optimized to achieve a narrow grain size distribution and good in-plane c-axis orientation in the Co-alloy. High coercive squareness (S*) is necessary for good overwrite properties. The grain size distribution plays a key role in determining the thermal decay rate and media signal-to-noise ratio. These aspects of the media microstructure will also be discussed.

9:00 AM G1.2/F3.2
EFFECT OF BORON ON CRYSTALLOGRAPHIC, MAGNETIC, AND RECORDING PROPERTIES OF COBALT ALLOY THIN FILM MEDIA. Dong-Won Park, Robert Sinclair, Department of Materials Science and Engineering, Stanford University, Stanford, CA; Marilee Schultz, Brij B. Lal, Michael A. Russak, HMT Technology, Fremont, CA.

Increasing the storage density of longitudinal thin film media requires accurate control of cobalt alloy grain size without deteriorating magnetic properties including the signal to noise ratio (SNR), overwrite (OW), and thermal stability. In order to achieve precise analysis of these characteristics, high resolution transmission electron microscopy (HRTEM), vibrating sample magnetometer, and alternating gradient force magnetometer are required, which provide microstructure, magnetic characteristics, and thermal stability, respectively. Furthermore, a dual element head consisting of an inductive and an MR head reveals magnetic recording properties. These characterization tools have been applied to investigate a series of CoCrPtTa, CoCrPtB, and CoCrPtTaB thin film longitudinal media, prepared under ostensibly identical sputtering conditions. HRTEM revealed that adding 5% or 8% boron to the magnetic layer resulted in significantly reduced average grain size compared to the media with a CoCrPtTa magnetic layer. It was also found that by adding boron, SNR and media noise properties were improved, while coercivity and OW decreased. For the medium with 8% boron in the magnetic layer, the OW value was found to be smaller than 30 dB which is required not to cause errors while reading new recorded data. In addition to this characterization, magnetic switching volume and dynamic coercivity will be also presented, which are relevant to the thermal stability of cobalt alloy longitudinal thin film media with and without the presence of boron.

9:15 AM G1.3/F3.3
TEM TO SUPPORT MAGNETIC MEDIA DEVELOPMENT IN YR2000. Warren J. MoberlyChan, Tom P. Nolan and Paul Dorsey, Komag, Inc., San Jose, CA.

In this 1 square inch space, hard drive media of 1999 stored >2 Gbytes. This requires reproducibly processing thin films with judicious control of >2000000000000/in^2 magnetic grains. Further constraints include corrosion resistance in all environments, impact resistance involving GPa stresses, >30db signal-to-noise, rapid information transfer, and wear resistance associated with heads flying at 20000 rpm and at a spacing <0.00007$^{\underline{th}}$ the width of a human hair. The price tag for a finished 3.5″ disk of media was $8 in 1999. In the year 2000, storage density must increase >100%; and price must drop. To solve this ongoing Y2K problem, statistical and efficient TEM techniques are necessary.
This paper presents traditinal and state-of-the-art TEM methods for the study & control of crystallography in all three directions in these media grains on metal or glass substrates. Electron diffraction, weak-beam dark field, and *in situ* tilting experiments provide structural analysis to advance these materials. Routinized tilting and statistical imaging enable the microscopist to see, study, and improve the spatial inhomogeneities that are necessary to isolate each bit of information. Computer processing now enhances the analysis of TEM data, enabling a synergistic relation among TEM, computers, and media processing that improves performance of all.

9:30 AM G1.4/F3.4
RELATIONSHIP BETWEEN THE GRAIN SIZE OF MAGNETIC LAYER AND THAT OF UNDERLAYER IN CoCrPtTa RECORDING MEDIA. Kai Ma, Robert Sinclair, Stanford University, Dept. of Materials Science and Engineering, Stanford, CA; Gerardo Bertero, Wei Cao, Komag Inc., San Jose, CA.

High data density and low medium noise requirements make the grain size a critical constraint in the design and manufacture of magnetic media. It is known that the microstructures of the magnetic layer are strongly dependent on those of the underlayer. A great deal of effort has been expended on decreasing the underlayer grain size in order to decrease the magnetic layer grain size. But recent investigation has shown that the grain size of the magnetic layer might not always follow that of the underlayer, e.g. decreasing at the same time. When the underlayer grain size is too small, the magnetic grain size no longer decrease with underlayer grain size. Considering that the bi-crystal grain boundaries usually are not clearly revealed in normal bright field (BF) Transmission Electron Microscopic (TEM) images, high resolution TEM (HRTEM) images are used to accurately measure the magnetic layer grain size and the angles between the c-axes of adjacent grains. By carefully controlling the processing conditions, CrMo underlayer grain sizes were made to vary from 16nm to 10nm. However, the corresponding CoCrPtTa grain sizes basically remained unchanged. As the underlayer grain size decreased, the ratio of magnetic layer grain size to underlayer grain size increased from 0.87 to 1.40. HRTEM results show that the formation and breakup of bi-crystal clusters play a determinant role in this effect. The significance of these results for the growth of the magnetic layer will be discussed.

9:45 AM G1.5/F3.5
MICROSTRUCTURE AND TEXTURAL CHARACTERIZATION OF Co-ALLOY THIN FILMS BY TRANSMISSION ELECTRON MICROSCOPY. K.M. Moulding, J.S.K. Wong, J. Zheng, MCPF,

Hong Kong University of Science and Technology, HONG KONG.

Co-alloy thin films are a popular choice for current magnetic media. As the demand for higher bit densities increases, the crystallography and microstructure of the magnetic thin film becomes increasingly important. Co-alloy films with a small grain size and a highly orientated c-axis offer the possibility of achieving a high signal to noise ratio with fewer grains per bit for future perpendicular magnetic media. In order to produce films with the desired properties, the introduction of a buffer layer and a suitable seed layer is considered to be an important process. The purpose of this paper is to investigate the microstructure of RF sputtered Co-Cr-Pt films grown on silicon substrates without an intermediate layer, and to compared with those which have an intermediate buffer and seed layer. In the process of the comparison, the roles of the additional layers to enhance the crystallographic properties of the Co-alloy films is characterized. Furthermore, the effect of substrate temperature during deposition and the sputtering conditions on the resulting microstructure of the films is also determined.

10:30 AM *G1.6/F3.6

TEM CHARACTERIZATION OF CHEMICAL COMPOSITION INHOMOGENEITY IN MAGNETIC THIN FILM MEDIA.
J.E. Wittig[1], J. Ma[1], and J. Bentley[2], [1]Vanderbilt University, Nashville, TN; [2]Oak Ridge National Laboratory, Oak Ridge, TN.

The structural requirements for low noise magnetic recording media (small grain size, separation of magnetic particles) increasingly conflict with those for thermally stable magnetization (large uniform grain size, high uniform magnetic anisotropy). Magnetic Co-alloy grain size, grain separation and anisotropy in modern thin film media are all controlled in large part by chemical composition inhomogeneities. Thus, characterization of chemical composition within and between Co-alloy grains is becoming critical for micromagnetic modeling and future media development. Since the grain size of modern recording media is on the order of 10 to 15 nm, high resolution characterization methods such as transmission electron microscopy (TEM), nanoprobe energy dispersive spectroscopy (EDS) and energy filtered imaging (EFTEM) are necessary to accurately describe the microstructure. This paper demonstrates the strengths and limitations of these characterization methods using an Mrt series of longitudinal recording media prepared for the extreme high density recording (EHDR) project of the National Storage Industry Consortium (NSIC). These EHDR disks have NiP/Al supersmooth substrates with a 60 nm NiAl seed layer and a 7.5 nm CrMo intermediate layer. The magnetic media is a $Co_{71}Cr_{17}Pt_8Ta_2Nb_2$ alloy with seven different thicknesses resulting in Mrt values from 0.1 to 0.6 memu/cm^2. Both EDS and EFTEM have been used to characterize the composition in these longitudinal media in order to understand the limitations from decreasing magnetic film thickness on the quantification capability. Research at the ORNL SHaRE User Facility was supported by the Division of Materials Sciences, U.S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp., and through the SHaRE Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities.

11:00 AM *G1.7/F3.7

CHEMICAL ORDERING AND MICROSTRUCTURE OF FePd THIN FILMS WITH PERPENDICULAR MAGNETIC ANISOTROPY. Bruno Gilles, LTPCM, CNRS Grenoble, FRANCE; Alain Marty, Yves Samson, David Halley, DRFMC, CENG Grenoble, FRANCE.

The $L1_0$ ordered phases in FePd, FePt and CoPt binary alloys in thin films might be promising materials for high density storage media, due to their high magnetocristalline. Depending on the growth temperature, we have been found that FePd alloys grown by MBE may be obtained with a long range order (LRO) varying from 0 to 1, i.e. from the disordered binary alloy to the well ordered $L1_0$ phase with the c-axis perpendicular to the surface. Therefore the ratio between the perpendicular magnetization anisotropy and the shape anisotropy may be varied from 0 to nearly 2 and materials may be obtained with either in-plane magnetization or out-of-plane magnetization. In this talk, we will focus on the growth process and show how the chemical order develops at the surface of the growing layer. Indeed, combining RHEED, STM and Auger or XPS spectroscopies we have found that a complicated segregation process occurs during growth, which may explain the development of the ordered structure with only one out of the 3 equivalent variants. This has been confirmed by experiments in which As or Te surfactants have been shown to inhibit the ordering process. TEM measurements as well as quantitative X-ray analysis have shown that the density of anti-phase boundaries is decreased when the growth temperature is increased and STM images have revealed larger atomic and bi-atomic terraces. This suggests that the diffusion length of the ad-atoms on the growing surface may explain the change in the LRO. Attention has been paid to the relaxation of strain which is in favor to the tetragonal ordered phase but it seems that it is not the effective driving force in the ordering process.

11:30 AM G1.8/F3.8

MECHANISM FOR ION BEAM MODIFICATION OF MAGNETIC PROPERTIES OF THIN FILMS AND MULTILAYERS.
J.E.E. Baglin, D. Weller, L. Folks, M. Toney, A.J. Kellock, B.D. Terris, E. Fullerton, S. Maat, C.T. Rettner and G.J. Kusinski, IBM Almaden Research Center, San Jose, CA.

We have investigated the physical mechanism whereby ion irradiation produces large changes in the magnetic properties of thin films suitable for magnetic recording, e.g. $[Co/Pt]_n$ multi-layers, or Fe-Pt alloy films. These effects are the basis of ion beam patterning techniques proposed for future high density storage. Samples were irradiated with He, N, Ar or Xe ions at energies between 30 keV and 1 MeV, with doses spanning the range 10^{14} - 5×10^{16} ion/cm^2. We then examined the dependence of the magnetic properties on ion energy, species and dose, and on the media structure, (number and thickness of layers; stoichiometry). Structural characterization was done using AFM, MFM, LTEM, XRR, RBS, and ion channelling. We attribute the magnetic effects primarily to short-range chemical disordering effects at multilayer interfaces or within alloy media, induced by ion beam mixing. The model appears to be supported by TRIM simulations.

SESSION G2/F4: JOINT SESSION: CHARACTERIZATION OF MAGNETIC THIN FILMS AND STRUCTURES

Tuesday Afternoon, April 25, 2000
Golden Gate B3 (Marriott)

1:30 PM *G2.1/F4.1

X-RAY PROBES OF MAGNETIC MULTILAYER STRUCTURE.
B.K. Tanner, T.P.A. Hase, B.D. Fulthorpe, J. Clarke and S.B. Wilkins, Department of Physics, University of Durham, Durham, UNITED KINGDOM.

We discuss the application of x-ray scattering and fluorescence to the problem of unravelling the relationship between structural and magnetic properties of magnetic multilayers. Particular attention is paid to the use of grazing incidence diffuse scatter to determine the compositional gradient, out-of-plane roughness amplitude, in-plane correlation length and fractal parameter of buried interfaces. We show how to determine the degree of conformality of the roughness in multiple layer structures such as spin-valves. Measurements of the changes in the interface structure of permalloy-copper multilayers during heat treatment and the effects of cobalt doping at the interfaces are described. The power of combining high angle diffraction and scattering with grazing incidence scattering, surface diffraction and fluorescence is illustrated in studies of Au-Fe multilayers grown by molecular beam epitaxy. Careful correlation of the structural and magnetotransport measurements provides evidence for a novel mechanism of the giant magnetoresistance in this system. Variable angle grazing incidence fluorescence studies of thin films enables the depth location of impurities, for example 1% Ni in a 20nm Cr film, to be determined. We present evidence for surfactant behaviour of Nb in the growth of Au-Fe multilayers. Soft x-ray scattering enables us to access larger areas of reciprocal space in comparison to the grazing incidence scattering of harder x-rays. We compare structural data taken from Co-Cu multilayers taken with 800eV and 8keV x-rays and show that it provides more directly a measure of interface structure with very short in-plane correlation length. By tuning to the L absorption edges of transition metals, a very substantial resonant enhancement is observed in the magnetic x-ray scattering and we show how these measurements can be used to determine the amplitude and length-scale of the pure magnetic roughness independently of the structural roughness.

2:00 PM G2.2/F4.2

PHOTOEMISSION SPECTROSCOPY OF PSEUDOMORPHIC THIN FILMS OF THE INVAR ALLOY Fe_xNi_{1-x}. Michael Hochstrasser, Roy F. Willis, The Pennsylvania State University, Physics Department, University Park, PA; Frank O. Schumann, James G. Tobin, Lawrence Livermore National Laboratory, Material Science Division, Livermore, CA; Eli Rotenberg, Lawrence Berkeley National Laboratory, Advanced Light Source, Berkeley, CA.

In the bulk, the magnetic moments of Fe_xNi_{1-x} alloys deviate strongly from at an Fe concentration of 65%, dropping quickly to zero as does the Curie temperature, at which point, a structural phase transition from fcc to bcc is observed. Recently, it has been shown that Fe_xNi_{1-x} films can be stabilized in the fcc phase when grown as ultrathin films on Cu(100). The fcc to bcc structural transformation is quenched, but the magnetic instability persists. We have investigated with angular resolved photoemission the k-space electronic structure of thin Fe_xNi_{1-x} alloy films pseudomorphically grown on Cu(100) over the whole concentration range. We observe changes in the Fermi surface which can be associated with hybridization effects of the d-bands with the sp-bands at particular positions in the k-space electronic structure. These hot-spots relate to specific spanning wave-vectors at the Fermi surface which are important in understanding quantum-well oscillations and giant magnetoresistance effects. Dispersion curve measurements of the sp-bands allow us to measure lifetime effects and exchange splitting. With spin-resolved photoemission spectroscopy particular regions in k-space have been further investigated to get a better insight into the changing electronic and magnetic structure of Fe_xNi_{1-x} fcc alloy films. We observe changes at the Fermi surface which indicate magnetic disorder. To understand the magnetic instability x-ray magnetic linear dichroism measurements have been used to measure the magnetization behavior of these magnetic thin films.

2:15 PM G2.3/F4.3

ON THE STABILIZATION OF 2D SURFACE ORDERED ALLOYS BY MAGNETIC POLARIZATION. S. Meza-Aguilar, C. Demangeat, H. Dreyssé, IPCMS, Strasbourg, FRANCE; A. Rakotomahevitra, Department of Physics, Florida Atlantic University, Boca Raton, FL.

Choi *et al* (Phys. Rev. B **58**, (1998) 5166) have studied the magnetic properties of ultrathin Mn films on fcc Co/Cu(100) using the *in situ* magneto-optical Kerr effect (MOKE) and low energy electron-diffraction. They found that a two-dimensional MnCo(001) surface alloy is stabilized in the range 0.3–0.8 monolayer of Mn. Also Antel *et al* have tried to elucidate the origin of exchange biasing in FeMn/Co(100) by MOKE and x-ray dichroism. Theoretical electronic and magnetic structure studies on MnCo and (Fe-Mn)Co systems have been carried out using the Tight-Binding Linear Muffin-Tin Orbital (TB-LMTO) method within the density-functional theory (DFT) and its local density approximation (LDA). We discuss the stability of a Mn monolayer on Co(001) versus the formation of 1 or 2ML-thick MnCo(001) alloys, as well as the inverted Co/Mn/Co(001) system case. The MnCo alloy is found to be more stable than the Mn overlayer on Co(001) substrate, in qualitative agreement with the experimental predictions above. In the case of 2ML-thick MnCo alloy the magnetic coupling between Mn and Co atoms at the surface and the subsurface is found to be ferromagnetic and antiferromagnetic respectively. We discuss the stability of a 2D ordered Fe-Mn alloy on Co(100) versus the buried Co/Fe-Mn/Co(100) alloy. As previously we found that the buried configuration is the ground state. The Mn atom -in the ground state- is always found with antiferromagnetic coupling with its nearest neighbors.

2:30 PM G2.4/F4.4

SHAPE EFFECTS ON THE MAGNETIZATION OF COBALT AND PERMALLOY NANO-SCALE ARRAYS. J. Johnson, V. Metlushko, P. Vavassori, M. Grimsditch, Materials Science Division, Argonne National Laboratory, Argonne, IL; B. Ilic, School of Applied and Engineering Physics, Cornell University, Ithaca, NY; P. Neuzil, R. Kumar, Institute of Microelectronics, Singapore, SINGAPORE.

We have investigated the shape and thickness dependence of a series of Co and permalloy nano-scale arrays using the Magneto Optic Kerr Effect (MOKE) technique and MFM. The patterns were produced by e-beam lithography combined with e-beam deposition and lift-off techniques. Fifteen 1.0 by 2.5 micron tetragonal arrays with elliptical elements with various sizes and aspect ratios in the range 100nm to 2000nm were fabricated simultaneously, on a single substrate, to avoid complications due to changes in anisotropy and or thickness. Identical patterns were fabricated for 30nm of Co and 20nm and 30nm of permalloy. The effect of element shape on coercivity, extracted from the MOKE loops for the three samples will be presented. Domain structure as inferred from MOKE, observed with MFM and estimated based on shape anisotropies will be discussed. Work at Argonne National Laboratory was supported by the U.S. Department of Energy, Division of Material Sciences, Office of Basic Energy Sciences, under contract W-31-109-ENG-38. P.V. acknowledges support by a research grant from INFM-Istituto Nazionale per la Fisica della Materia.

3:15 PM *G2.5/F4.5

TEM IN SUPPORT OF INDUSTRIAL RESEARCH. Philip M. Rice, Stuart S.P. Parkin, Robin F.C. Farrow, Mary Moore, IBM Almaden Research Center, San Jose, CA; William J. Gallagher, IBM-T.J. Watson Research Center, Yorktown Heights, NY; Tsann Lin, IBM Storage Systems Division, San Jose, CA.

Competition in the storage industry has driven storage research toward ever increasing areal densities. These increases in storage densities are derived either from improving the technology used (such as switching from MR to GMR sensors) or simply decreasing the size of the elements involved. As the size of elements decreases, the magnetic multilayers become thinner and more complex. Transmission Electron Microscopy (TEM), with its high spatial resolution, plays an increasingly important role in the characterization of both the microstructure and microchemistry of magnetic multilayers. How TEM has been used to help in the research of GMR sensors and magnetic tunnel junction (MTJ) stacks will be discussed. The Focused Ion Beam (FIB) has also become an invaluable instrument due to its ability to make site specific TEM specimens from production wafers and heads. Examples from the Magnetic RAM project will be used to show how TEM can help in the development of wafer based production. Results from analytical TEM studies of elemental migration between multilayers will also be discussed.

3:45 PM G2.6/F4.6

ULTRATHIN ALLOY FILMS. F.O. Schumann, Freie Universität Berlin, GERMANY; J.G. Tobin, Lawrence Livermore National Lab, Livermore, CA.

We investigated ultrathin alloy films in an element-specific manner by means of dichroism in photoemission (MLDAD). Specifically we investigated fcc Fe_xCo_{1-x} and Fe_xNi_{1-x} films grown on Cu(100). For high Fe concentrations we find contrasting behavior of the two systems. In the case of Fe_xNi_{1-x} we observe a small magnetic signal associated with a moment-volume instability. However for the Fe_xCo_{1-x} alloys we observe an essentially constant and strong dichroism signal for all concentrations, in particular for Fe-rich alloys. This observation can be related to the stabilization of different atomic volumina of fcc Fe upon alloying with Co and Ni. It is now well-established that fcc Fe/Cu(100) can exist in two different magnetic states. These are a high-spin (HS) and low-spin (LS) state, the former having the larger atomic volume. It is predicted that this state has a larger magnetic moment than bulk bcc Fe. This moment enhancement in the case of Fe_xCo_{1-x} alloys allows us also to resolve fine-structure in the Fe 3p emission. This was theoretically predicted, but so far the experimental observation was missing.Spin-resolving measurements on the Fe 3p level confirm the origin of the fine structure to be due to the observation of the pure spin states. The importance of this result for element-specific magnetometry for ultrathin films is discussed.

4:00 PM G2.7/F4.7

THE STRUCTURE OF POLYCRYSTALLINE AND EPITAXIAL GMR MAGNETIC MULTILAYERS AND SPIN-VALVES GROWN BY SPUTTER DEPOSITION. Hong Geng, Reza Loloee, John W. Heckman, Martin A. Crimp, Dept of Materials Science and Mechnics, Michigan State Univ, East Lansing, MI; Reza Loloee, William P. Pratt, Jr., Dept of Physics and Astronomy, Michigan State Univ, East Lansing, MI.

The study of the GMR effect in magnetic multilayers and spin-valves has shown that the electrical transport in these materials depends on layer quality and interface structures, as well as film orientation. In the present study, a number magnetic multilayers $(Co/Cu)_x$ and spin-valves (FeMn/Py/Cu/Py and Py/Ag/Py/FeMn) were produced by dc magnetron sputtering (Py = Permalloy^{TM} (NiFe)). Initial work on these structures concentrated on polycrystalline materials grown with polycrystalline Nb superconducting contacts. More recent work has focused on epitaxial structures grown on single crystal $(1\bar{1}0)$ Nb that was deposited on $(11\bar{2}0)$ Al_2O_3 substrates. The grain size, morphology, and orientation of the epitaxially grown films have been characterized using electron back scattered pattern (EBSP). The structures of both polycrystalline and epitaxial films were also characterized using a variety of cross-sectional transmission electron microscopy (TEM) approaches including conventional and high-resolution TEM (CTEM and HRTEM). In polycrystalline samples, CTEM has revealed well-defined multilayered structures, which in most cases display columnar grains range up to 90 nm in diameter. The HRTEM study, complemented with fast Fourier transform (FFT) analysis and image simulations, indicated that some non-equilibrium phases exist in certain regions of these spin-valve layers. In epitaxial samples, CTEM and HRTEM revealed large numbers of threading dislocations in the single crystal Nb buffer layer. Subsequent sputtered of Cu, Py and Co resulted in two growth variants predicated by the stacking sequence of the {111} epitaxial planes. Significant grooving occurs where these variants meet, resulting in notable thinning of the multilayer structures parallel to the growth direction, and possibly pin holes. This work was supported in part by the MSU CFMR, and by the US NSF under grant MRSEC DMR 98-09688.

4:15 PM G2.8/F4.8

SCANNING-APERTURE PHOTOEMISSION MICROSCOPE FOR MAGNETIC IMAGING. Gary M. McClelland and Charles T. Rettner, IBM Research Division, Almaden Research Center, San Jose, CA.

We have demonstrated a new technique for magnetic imaging that is ultimately capable of spatial resolution approaching 5 nm. In our instrument, photo-emission is excited by a laser focused to a 10-micron spot. A scanning aperture above the magnetic surface allows only electrons from a small selected region to reach the electron detector. The magnetization in this region is determined from the dependence of photoemission on the circular polarization of the laser. Images of 10-nm-thick Co-Pt multilayer thin films on sapphire have been obtained. From a cesiated film, a high quantum efficiency of 0.002 was observed from 458 nm laser light. Circular dichroism of $\pm 2\%$ is recorded by alternating the circular polarization of the light while scanning. The tip distance above the surface is maintained by advancing the tip until 1-nA tunneling to a positive sample is observed, then withdrawing 15 nm and switching polarity to detect photoemission through the tip. The resolution we observe agrees well with the 35-nm-sized aperture in the gold tip. From the observed noise, we project that there is enough signal to image at 5 nm resolution if a small enough aperture can be fabricated. Recent calculations show that image forces on the electron from the aperture walls act to make the effective aperture even smaller than the physical diameter. The insensitivity of the instrument to varying magnetic fields should make it ideal for time dependent magnetization measurements in an applied field.

4:30 PM G2.9/F4.9

RESIDUAL STRESSES AND MAGNETOELASTIC COUPLING IN ULTRATHIN Fe FILMS DEPOSITED ON GaAs(001). Patrice Gergaud, Magali Putero, Olivier Thomas, MATOP, CNRS, Universite Aix-Marseille III, Marseille, FRANCE; Claude Lallaizon, Bruno LEPINE, Andre Guivarch, EPSI, CNRS, Universite Rennes, FRANCE.

The growing interest in the behavior of magnetic thin films on semiconductor substrates is due in part to their potential application in spin-sensitive heterostructure devices. The metal/semiconductor interface is expected to have a strong influence on the magnetic and electronic properties of the heterostructure. It has been shown previously that high-quality epitaxial iron thin films can be grown on GaAs(001) substrates because of the small lattice parameter mismatch (1.4%). Moreover, magnetic measurements performed on Fe films thinner than 3 nm have shown that such films exhibit an in-plane uniaxial magnetic anisotropy although an ideal bcc Fe(001) film should have fourfold symmetry. The source of this uniaxial component remains an open question and one of the mechanisms which may contribute to this is the epitaxial strain, through magnetoelastic coupling. In this study, we measured using X-ray diffraction the stresses in a series of iron thin films with thicknesses varying between 1.5 and 80 nm. For the thinnest films the anisotropy of the in-plane strain has been investigated. Very small strains anisotropies are capable of modifying the magnetic anisotropy of iron thin films. Moreover the sign and magnitude of the magnetoelastic coupling seem to depend on the film thickness or film strain [1]. We thus calculated the magnetic anisotropy using both bulk and thin film magnetoelastic coupling. 1. D. Sander, Rep. Prog. Phys. 62 (1999) 1-50.

SESSION G3/F5: JOINT SESSION: MAGNETIC TUNNEL JUNCTIONS AND SPIN-DEPENDENT TRANSPORT

Wednesday Morning, April 26, 2000
Golden Gate B3 (Marriott)

8:30 AM *G3.1/F5.1

SPIN POLARIZED CURRENTS IN MAGNETIC TUNNEL JUNCTIONS. Peter M. Levy, Kuising Wang, Dept of Physics, New York University, New York, NY; Shufeng Zhang, Dept of Physics, University of Missouri, Columbia, MO; Charles B. Sommers, Lab. Phys. Sol., Universite Paris-Sud, Orsay, FRANCE; Laszlo Szunyogh, Technical University Budapest, Budapest, HUNGARY; Peter Weinberger, Technische Universitat Wien, Vienna, AUSTRIA.

The factors that govern the magnetoresistance of magnetic tunnel junctions will be reviewed. Attention will be focused on: the bonding of orbitals at the interfaces between electrodes and the insulating barrier, and the effect of electric fields on redistributing the electron charge and spin distribution at the interfaces, i.e., changing the profile of the tunnel barrier. While the effects of electric field penetration in the metallic electrodes was appreciated as early as the 1960's, we find that: 1) the field induced redistribution is larger for the electrons that spill out of the electrode at the interface into the insulating barrier, and 2) the induced changes in charge in the majority and minority spin channels in the electrode go in opposite directions, so as to produce important changes in the magnetization for relatively small changes in the charge at the interfaces. Changing barrier materials, e.g., from alumina to strontium titanate, has the putative effect of changing the type of spin-polarized electrons (s versus d) that hop from the electrodes into the barrier, and introduces different sets of states for electrons to tunnel through. While there is no spin dependence to these insulating barriers switching, say from s to d type electrons, can change the spin polarization of the tunneling current. In this way magnetoresistance is sensitive to the barrier material.

9:00 AM *G3.2/F5.2

SPUTTER DEPOSITED MAGNETIC TUNNELING JUNCTIONS. S.S.P. Parkin, M.G. Samant, L. Thomas and P.M. Rice, IBM Almaden Research Center, San Jose, CA.

Magnetic tunnel junctions (MTJ) are comprised of thin ferromagnetic electrodes separated by insulating tunnel barriers. These structures have attracted a great deal of interest in recent years because they exhibit large magnetoresistance (MR) effects in small magnetic fields at room temperature which makes them potentially useful for magnetic field sensors and non-volatile magnetic memory cells. For these applications MTJ devices with comparatively low resistances are required. We show that the resistance of MTJ devices with alumina tunnel barriers can be readily and controllably varied over more than 8 orders of magnitude by simply varying the thickness of the alumina layer. This requires extremely thin alumina layers just a few angstrom thick. Nevertheless pin-hole free layers can be formed. We discuss the properties of a variety of MTJ structures including the dependence of resistance and magnetoresistance on details of the structure, for example, the nature of the ferromagnetic electrodes. The thermal stability of these structures is important for applications: mechanisms for degradation on annealing are presented. Finally we discuss a novel indirect magnetic interaction between the ferromagnetic layers across the insulating tunnel barrier mediated by fringing fields from domain walls in one ferromagnetic layer acting on the other ferromagnetic layer. * Work supported in part by DARPA.

9:30 AM G3.3/F5.3

APPARENT SPIN POLARIZATION DECAY IN Cu DUSTED $Co/Al_2O_3/Co$ MAGNETIC TUNNEL JUNCTIONS. P. LeClair, H.J.M. Swagten, J.T. Kohlhepp, R.J.M. van de Veerdonk and W.J.M. de Jonge, Department of Applied Physics and Cobra, Eindhoven University of Technology, Eindhoven, THE NETHERLANDS.

$Co/Al_2O_3/Co$ magnetic tunnel junctions with an interfacial Cu layer have been investigated via *in-situ* X-Ray Photoelectron Spectroscopy, Auger Electron Spectroscopy, and Scanning Tunneling Microscopy as well as *ex-situ* magnetotransport measurements. In identically grown structures, Cu interlayers grown on Co give an exponential decay of the tunneling magnetoresistance (TMR) with $\xi \approx 0.26$nm while Cu grown on Al_2O_3 has a much longer decay length of 0.70nm. The difference in decay lengths can be explained by differing growth morphologies in the two cases, as monitored by the *in-situ* spectroscopies, and in this way clarifies the present disagreement in literature. For monolayer coverage of Cu, for the first time we show that tunneling spin polarization is suppressed by at least a factor of 2 compared to Co. At 2ML the tunneling spin polarization is suppressed by more than a factor of 5, while beyond approximately 5ML it becomes vanishingly small.

9:45 AM G3.4/F5.4

SPIN-DEPENDENT ELECTRON TRANSPORT IN FERROMAGNET/SEMICONDUCTOR SCHOTTKY BARRIER STRUCTURES. Atsufumi Hirohata, Yong-Bing Xu, Christian Guertler, Tony Bland, Univ of Cambridge, Dept of Physics, Cambridge, UNITED KINGDOM; Stuart Holmes, Cambridge Res Lab, Toshiba Res Europe Ltd, Cambridge, UNITED KINGDOM.

The possibility of pin-polarized electron injection from a ferromagnet (FM) to a semiconductor (SC) has stimulated a great number of researchers to realize spin-electronic devices such as spin-polarized STM [1]. One attempt to control the electron polarization in the SC using photoexcitation with circularly polarized light has recently been demonstrated [2]. In this study, we produced samples of 3 nm Au/5 nm FM/GaAs ((100), $10^{23} \leq n^+, p^- \leq 10^{25}$ m^{-3}) and attached two Al electrical contacts to the Au layer and one ohmic contact to the bottom of the substrate. Conventional *I-V* measurements were carried out to define the Schottky characteristics of the samples. Circularly polarised laser light ($515 \leq \lambda \leq 780$ nm) was used to excite electrons with a spin polarisation perpendicular to the film plane in the samples. A helicity-dependent photocurrent, dependent upon the magnetisation configuration of the film ($\vec{\sigma} \perp \vec{M}$ or $\vec{\sigma} // \vec{M}$) and the Schottky barrier height, was detected with NiFe as the FM. An almost constant difference between the helicity-dependent photocurrent for perpendicular and parallel configurations is observed at negative bias, which corresponds to the spin-dependent photocurrent passing from the SC to the FM. At an applied bias voltage approximately equal to the Schottky barrier height, a minor supression in the bias dependence of the helicity-dependent photocurrent was observed, suggesting the existence of polarized electron transport from the FM to the SC. The helicity-dependent photocurrent polarisation decreases with increasing doping density and increases with decreasing induced photon energy. These results provide clear evidence of spin-dependent electron transport through the FM/SC direct interface. [1] G. A. Prinz, *Science* **282**, 1660 (1998). [2] A. Hirohata, Y. B. Xu, C. M. Guertler and J. A. C. Bland, *J. Appl. Phys.* **85**, 5804 (1999).

SESSION G4/F6: JOINT SESSION:
GMR AND SPIN VALVES I
Wednesday Morning, April 26, 2000
Golden Gate B3 (Marriott)

10:30 AM *G4.1/F6.1

VERTICAL GMR MRAM. Gary A. Prinz, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC.

In recent years there have been announcements of programs to develop nonvolatile random access memory based upon magnetic storage of the information (MRAM). The most advanced demonstration has utilized current in-plane (CIP) transport in giant magnetoresistance (GMR) spin-valves. Slightly less developed demonstrations have shown that magnetic tunnel junctions may also provide the basis for MRAM and their vertical structures could occupy less space on a chip. In this talk, a third approach will be described, which exploits current perpendicular to the plane (CPP) transport in GMR multilayers fabricated into vertical structures.

11:00 AM *G4.2/F6.2

CURRENT-CONTROLLED DOMAIN SWITCHING IN MAGNETIC MULTILAYERS BY MEANS OF SPIN TRANSFER. E.B. Myers, D.C. Ralph, Laboratory of Atomic and Solid State Physics, Cornell University, Ithaca, NY; J.A. Katine, F.J. Albert and R.A. Buhrman, School of Applied and Engineering Physics, Cornell University, Ithaca, NY.

We demonstrate that spin-polarized currents flowing perpendicularly through magnetic multilayers can apply sufficient torques to the magnetic layers to reorient their moments, by means of a mechanism based on local exchange interactions instead of current-generated magnetic fields. The idea of the new mechanism, as predicted by J. Slonczewski and L. Berger, is that when a spin-polarized current is scattered by a magnetic layer, spin angular momentum is transferred to the layer, meaning that a torque is applied. We have observed this effect in two experimental geometries: devices in which a 5-10-nm nanofabricated metal point contact is used to inject current into a Co/Cu/Co trilayer and (in work led by Katine, Albert, and Buhrman) in Co/Cu/Co pillars approximately 100 nm in diameter. In both cases, one of the Co layers is made thinner than the other, in order to make it more easily reoriented. At low magnetic fields, we can controllably switch the magnetic moments in the two Co layers parallel with a current pulse in one direction, and switch them antiparallel with a reversed current. The asymmetry in current direction is as predicted by the spin-transfer mechanism. The current densities required for switching are $\sim 10^9$ A/cm^2 in the point contacts and less than 10^8 A/cm^2, in the pillars, well below the levels at which the devices are damaged by the current. When large magnetic fields are applied, the spin-polarized current no longer fully reverses the magnetic moment, but instead stimulates spin-wave excitations. The simple geometry of the 100-nm-pillar experiments allows for quantitative tests of the theories of the spin-transfer mechanism, and provides a new means of measuring the damping parameter controlling the motion of individual magnetic domains. We observe greater damping for domains which are exchange-coupled to a continuous film than we do for isolated domains.

11:30 AM G4.3/F6.3

EFFECTS OF ANNEALING ON THE MICROSTRUCTURE AND GIANT MAGNETORESISTANCE OF Co-Cu-BASED SPIN VALVES. M.A. Mangan, G. Spanos, Naval Research Laboratory, Washington, DC; R.D. McMichael, P.J. Chen and W.F. Egelhoff, Jr., National Institute of Standards and Technology, Gaithersburg, MD.

The effect of annealing on the microstructure and giant magneto-resistive properties of NiO/Co/Cu/Co bottom spin valves is investigated using conventional and high resolution transmission electron microscopy. The value of giant magnetoresistance (GMR) of these spin valves is observed to decrease from 12.2% to 2.7% after annealing in vacuum for 30 minutes at 335 C. This decrease is attributed to an increase in the roughness of the Co and Cu layers. In annealed specimens, grain boundary grooving is also observed in the antiferromagnetic NiO pinning layer at the NiO/Co interface, and the location of these grooves correlates with waviness in the Co/Cu interfaces. An increase in the Neel "orange-peel" coupling between the ferromagnetic Co layers, resulting from the increased roughness of the Co/Cu interfaces, accompanies the degradation of the GMR.

SESSION G5/F7: JOINT SESSION:
GMR AND SPIN VALVES II
Wednesday Afternoon, April 26, 2000
Golden Gate B3 (Marriott)

1:30 PM *G5.1/F7.1

ADVANCES IN TUNNEL JUNCTION AND SPIN VALVE TECHNOLOGY FOR MRAM AND READ HEAD APPLICATIONS. P.P. Freitas, Instituto de Engenharia de Sistemas e Computadores, Lisbon, PORTUGAL.

Tunnel junction fabrication for MRAM integration (RxA=1 to 10 kOhm x um2, TMR>40%, tAl=11A, oxide or nitride barriers), and for ultra high density read heads (RxA = 45 Ohm x um2, TMR=20%, tAl=6A) is described. The thermal stability of such devices is analized, in view of the required applications. Head and MRAM designs are discussed and results are shown for vertically integrated tunnel junction- a-Si diode memory elements, with on chip switching time down to 30ns. Ongoing progress in diode technology is discussed in order to maximize memory density. Progress in spin valve technology is reported in two areas. Increase in GMR signal from 8% to 14% is reported when inserting thin oxide layers in the middle of the free and pinned ferromagnetic layers, increasing specular scattering. Decrease in the spin valve free layer thickness(10A), and increase in sensor sensitivity is obtained by using synthetic free layers, together with synthetic AF layers.

2:00 PM *G5.2/F7.2

REAL-TIME CONDUCTIVITY STUDIES OF INTERDIFFUSION IN THIN-FILM GROWTH. William F. Egelhoff, Jr., Magnetic Materials Group, National Institute of Standards & Technology, Gaithersburg, MD.

Measurements of thin-film conductivity made in real-time during thin-film growth are well-known for providing much insight into growth modes. This insight is based on the fact that a random alloy of two metals usually has a larger electrical resistivity than either metal in pure form. We have applied this technique to investigate interdiffusion in a variety of metal-on-metal systems. Although many other experimental techniques are available for identifying interdiffusion during growth, few are as rapid and as easy to use as this one. It is noteworthy that, among the possible metal-on-metal combinations, experimental data on interdiffusion during growth is presently available for only a few systems. For example, considering two metals chosen at random from the periodic table it is very unlikely that any experimental data is available to indicate whether such interdiffusion will occur in their case. Many insights into the chemistry of interdiffusion during the growth of thin films no doubt await discovery. As one example, we find that the heat of alloying is not a particularly good predictor of the extent of interdiffusion. Often in a bi-metal system, A on B will exhibit interdiffusion while B on A will not. Clearly, kinetic factors are important. As another example, we have found experimental support for our earlier claim that oxygen can be used as a surfactant in giant magnetoresistance spin valves to suppress interdiffusion during the growth of Co on Cu. The technique is not limited to observations of interdiffusion alone. We have also used it to observe other chemical changes at metal surfaces, such as the reaction of Al with oxygen on an oxidized Ni surface to convert insulating NiO to conducting Ni metal. This talk will give an overview of and examples of the great usefulness and potential of this so-far under-utilized technique.
The author would like to acknowledge his collaborators in this work, including P. J. Chen, C. J. Powell, M. D. Stiles, and R. D. McMichael.

2:30 PM G5.3/F7.3

FIRST PRINCIPLES BASED SOLUTION TO THE BOLTZMANN TRANSPORE EQUATION FOR Co/Cu/Co SPIN Co/Cu/Co SPIN VALVES. J.M. MacLaren, Physics Department, Tulane University, New Orleans, LA; L. Malkinski and J. Wang, AMRI, University of New Orleans, New Orleans, LA.

In this work we use a first principles based semi-classical theory of electron transport based upon solutions to the Boltzmann transport theory to study the CIP and CPP transport in asymmetric Co/Cu/Co spin valves. The inputs to the Boltzmann transport theory include the Fermi energy Bloch waves, the Bloch wave velocities and scattering matrices which describe the reflection and transmission of these Bloch waves from the interfaces. These are obtained from ab-initio local spin density calculations. In particular we study the role of individual layer thicknesses on properties and compare with our Co/Cu/Co samples grown by magnetron sputtering. Our results show that signicant contributions to the GMR come from the channeling of majority electrons in the Cu spacer layer, though with contributions also arising from the adjacent Co layers. In the Co layers the contributions appear to decay with a lengthscale characteristic of the Co minority mean free path. The GMR varies slowly with Cu thickness because both the conductance and change in conductance vary similarly with

Cu layer thickness. The most dramatic variation occurs for Co thicknesses around that of the minority mean free path of Co (about 4 nm). The value of the GMR can be changed from about 5% to close to 10%. Unlike CIP, we find that for CPP the GMR ratios are larger and relatively insensitive to the layer thicknesses which simply reflects the fact that in the CPP geometry current cannot avoid the high resistance minority Co channel. This work was supported by DARPA under contract MDA 972-97-1-003.

2:45 PM G5.4/F7.4

MANUFACTURABILITY OF GMR HEADS: 10GB/IN^2 AND BEYOND. Sanghamitra Sahu, Vivien Talghader, Jian Chen, Sining Mao, Seagate Technology, Bloomington, MN.

Consistency of spin-valve stack deposition, both wafer-to-wafer and within a wafer, is key to the manufacturability of GMR heads. For typical GMR heads, film thicknesses are now in the range of 5Å-150Å and process control is becoming a challenge.
This paper discusses the importance of characterizing the thickness and uniformity of each material in the spin-valve stack within the context of single layer sheet films. For this study, single layer sheet films and spin-valves were deposited in a multi-target cluster tool. The sheet resistance and thickness of the single layer sheet films were measured using a 4-point probe and ellipsometry respectively. Results were then correlated to the sheet resistance and magnetic performance (i.e., GMR ratio, exchange field, and magneto-static coupling field) of the full spin-valve stacks with a synthetic antiferromagnet as the pinned layer. The $1\sigma = 1\%$ uniformity realized for the full stacks is a strong indicator of a manufacturable process.
Model calculations of the sheet resistance and GMR were carried out to provide theoretical understanding of the film thickness dependence. The calculated results are well correlated with the experimental measurements. The model is based on band structures and implicitly includes the momentum-dependent reflection and transmission coefficients at the interfaces.
Results from both experiments and calculations show that process control and repeatability (both within a wafer and from wafer to wafer) are crucial to the spin-valve manufacturability, offering a critical challenge to the disc drive industry as it heads into the next generation of GMR heads.

3:30 PM *G5.5/F7.5

EXCHANGE ANISOTROPY DETERMINED FROM REVERSIBLE PROCESSES. E. Dan Dahlberg, School of Physics and Astronomy, University of Minnesota, Minneapolis, MN.

A microscopic understanding of the Ferromagnetic/AntiFerromagnetic Exchange Anisotropy (F/AF EA) which arises at the interface between a ferromagnet and an antiferromagnet has been elusive since its discovery. Simple models of the F/AF EA would predict energies roughly a factor of a hundred times larger than those determined by measurements of the shift in magnetization hysteresis loops. However, a hysteresis loop is generally determined by irreversible processes. Using two different reversible magnetic techniques, we obtain F/AF EA which are up to an order of magnitude larger than the hysteresis loop method.(B.H. Miller and E. Dan Dahlberg, Appl. Phys. Lett. 69,3932 3931 (1996) and Valter Strom, B.J. Jonsson, K.V. Rao, and Dan Dahlberg, J. Appl.Phys. 81, 5003 5005 (1997)) Although the F/AF EA determined by reversible magnetic rotations is still less than simple models would predict, it does provide a more accurate determination of the average F/AF EA at the interface. As such, the F/AF EA calculated by models and studies of the systematics of this phenomena should be compared only to the F/AF EA determined by reversible processes.

4:00 PM G5.6/F7.6

CHANGES IN STRESS AND MICROSTRUCTURE IN PtMn/CoFe BILAYERS DURING ANTIFERROMAGNETIC PHASE TRANSFORMATIONS. S.P. Bozeman and B.J. Daniels, Seagate Recording Heads, Minneapolis, MN.

PtMn is one of several candidate antiferromagnetic materials for biasing of spin valve giant magnetoresistive (GMR) sensors used in magnetic recording heads. The as-deposited crystal structure of PtMn is face-centered cubic (fcc), which is *not* antiferromagnetic, and it is commonly annealed to transform it to the face-centered tetragonal (fct) structure, which *is* antiferromagnetic. This phase transformation is accompanied by significant increases in stress and substantial changes in microstructure. Changes in the thin film stress during the fcc to fct transformation have been reported previously (Daniels, et al., MRS Fall '99) and can be up to 1.5 GPa. Stress vs time data from *in situ* annealing experiments were shown to fit well with the Avrami equation, suggesting that the changes in stress are dominated by the fcc to fct transformation. In spite of this good agreement, stress changes can originate from components related to (1) the phase transformation, (2) interdiffusion, and (3) grain growth. In order to resolve the effect of microstructural changes on the stress in these films, we have obtained XRD data for PtMn/CoFe bilayers before and after annealing. Further, since the magnetic behavior of these films has been observed to depend strongly on the PtMn composition, the behavior of the transformation has been studied for PtMn compositions over a range of ~10 atomic percent.

4:15 PM G5.7/F7.7

LARGE ANISOTROPY INDUCED BY OBLIQUE SPUTTERING OF Ta UNDERLAYERS. J.E. Bonevich, R.D. McMichael, C.G. Lee, P.J. Chen, W. Miller and W.F. Egelhoff, Jr., National Institute of Standards and Technology, Materials Science and Engineering Laboratory, Gaithersburg, MD.

Applications of the giant magnetoresistance (GMR) effect rely on precise magnetization control of the constituent layers. A number of control schemes have been developed including the use of antiferromagnetic materials for exchange biasing and the use of hard magnetic materials. We have obliquely sputtered Ta underlayers to produce unusually strong uniaxial anisotropy in ultra-thin Co films and demonstrated this technique in a pseudo spin valve. Thin films of Co, $Ni_{80}Fe_{20}$(Py) and Co/Cu/Co pseudo spin valves were DC magnetron sputter deposited on obliquely (60° to substrate normal) sputtered Ta underlayers and capped with Au. The anisotropy field values for the films deposited on obliquely sputtered Ta are quite large compared to magnetocrystalline anisotropy fields of Py or fcc Co. With 7.5 nm Ta, the measured value of $\mu_0 H_a$, 163 mT, is comparable to that of single crystal hcp Co. A large increase in $\mu_0 H_a$ is also observed for Py films, thus the magnetocrystalline anisotropy apparently does not play a large role. Relatively narrow FMR resonances also indicate a surprising degree of uniformity in the anisotropy. Additionally, the damping parameter is nearly isotropic. Cross-sectional HRTEM reveals the Ta/transition metal interface to be corrugated with ridges and grooves extending perpendicular to the Ta incidence plane. The presence of roughly aligned ridges and grooves on the length scale of a few nm is expected to give rise to magnetostatic anisotropy. Because the periodicity of the grooves is not significantly larger than the magnetostatic exchange length, ≈ 4 nm, the magnetization will not be able to follow the contours of the surface and magnetostatic charges will exist on the sides of the ridges, giving rise to a demagnetization field in the plane of the film.

SESSION G6: POSTER SESSION: POLYCRYSTALLINE METAL AND MAGNETIC THIN FILMS

Wednesday Evening, April 26, 2000
8:00 PM
Salon 1-7 (Marriott)

G6.1

IN-SITU ELECTRICAL RESISTIVITY MEASUREMENTS OF Al-Ge FILMS IN THE TEM USING A MODIFIED HEATING HOLDER. Marcel Verheijen, Johan Donkers, Philips CFT, Eindhoven, THE NETHERLANDS; Frank Thomassen, Jan van den Broek, Richard van der Rijt, Rine Dona, Casper Smit, Philips Research, Eindhoven, THE NETHERLANDS.

A TEM specimen holder has been developed for the measurement of the electrical resistivity of a TEM sample as a function of temperature. A custom TEM heating holder was modified for this purpose. Eight feed-through wires were passed through the specimen holder, (i) providing current to the heater, (ii) allowing for the measurement of the hot stage temperature and (iii) enabling the measurement of the electrical resistivity of the sample. This configuration creates the opportunity to directly correlate changes in the resistivity to microstructural changes as a function of temperature. One of the systems studied is Al-Ge. Sputter-deposited thin films of Al-Ge are high ohmic and amorphous. Upon heating a large decrease in resistivity occurs at 490 K. The microstructure of films of several thicknesses has been studied as a function of temperature and has been recorded on videotape, while simultaneously acquiring resistivity data. These in-situ TEM studies show that the decrease in resistivity is caused by crystallisation. During this transition demixing occurs, resulting in crystalline Al and Ge phases. Interpretation of the recorded images allowed for elucidating the crystallisation mechanism.

G6.2

Abstract Withdrawn.

G6.3

HIGHLY LOCALIZED QUANTITATIVE MEASUREMENTS AND SIMULATIONS OF THE THERMOPHYSICAL PROPERTIES OF POLYCRYSTALLINE ALUMINUM FILMS. C.J.K. Richardson and J.B. Spicer, The Johns Hopkins Univ, Dept of Materials Science and Engineering, Baltimore, MD.

This presentation will examine experimental and modeling results concerning the thermophysical properties of polycrystalline aluminum films. It is well known that the thermophysical properties of these films apparently vary with film thickness and method of deposition. Models incorporating the effects of microscopic electron-phonon energy transport, such as Umklapp electron-phonon scattering and electron-defect interactions are used to examine the thermal diffusivity in films of different microstructures as is measured under various experimental situations. Modeling results will be used to generate complete simulations of ultrafast time-resolved thermal spectroscopy observations that result from ultrafast laser pulse heating of a material surface. Measurement of thermal diffusion resulting from this laser heating over a few hundred picoseconds provides information about the thermal diffusivity of the first 100 nanometers of the sample surface. The dependence of the thermal diffusivity on grain texture, grain shape and film thickness will be examined.

G6.4
EFFECT OF WATER IN PECVD TEOS OXIDE UNDERLAYERS ON CRYSTAL ORIENTATION IN Al-ALLOY/TiN/Ti FILMS. Koyu Aoki, Tomoyuki Yoshida, Yasuichi Mitsushima, Toyota Central R&D Labs., Inc., Nagakute, Aichi, JAPAN.

Electromigration (EM) failures of Al-based interconnects such as Al-alloy/TiN/Ti are an important reliability problem in submicron ULSI technology. The EM resistance is known to be improved by enhancing <111> crystal orientation of Al-alloy films. Generally, Al-alloy/TiN/Ti interconnects are formed on plasma-enhanced chemical vapor deposited tetraethylorthosilicate (PECVD TEOS) oxides or O_3-assisted thermal CVD TEOS oxides, which usually contain water related species (H_2O and SiOH). In this work, we investigated the effect of these species in oxide films on crystal orientations of sputter-deposited Al-alloy/TiN/Ti films.
In this experiment, PECVD TEOS oxide films (800 nm) were deposited at various r.f. powers (60, 200 and 350 W), and some of these oxide films were then annealed at various temperatures (450, 550, 650 and 900°C). Then Ti and Al-alloy/TiN/Ti films were sputter-deposited onto these oxides at 350°C. FT-IR and SIMS measurements revealed that the concentration of water related species in the oxide films decreased as either the r.f. power or the annealing temperature increased. Also, x-ray diffraction measurements revealed that the crystal orientation of the Ti films was the most highly <0001> preferred at a certain concentration of the water related species. The following mechanism is proposed for the improved Ti orientation. H_2O vapor desorbed from oxide films at 350°C prior to sputtering of Ti films adsorbs on the oxide surface to reduce the surface free energy; this results in Ti film growth toward <0001> preferred orientation by self-assembly of Ti atoms. Furthermore, an Al-alloy/TiN/Ti film fabricated from the improved Ti film showed high TiN <111> and Al <111> orientations; the full width at half maximum value of the Al 111 x-ray rocking curve was almost 1°. Interconnects fabricated from the improved Al-alloy/TiN/Ti films are expected to have a higher resistance against EM.

G6.5
ELECTRICAL AND MICROSCOPIC INVESTIGATION OF E–GUN EVAPORATED TITANIUM THIN FILMS. K. Hofmann, B. Spangenberg, M. Luysberg, H. Kurz, RWTH Aachen, Institute of Semiconductor Electronics II, Aachen, GERMANY.

The work examines the temperature dependence of the resistivity of titanium thin films, when the films which have a thickness of 10 nm are evaporated by e–gun with different rates. The titanium film evaporated with a rate of 1 Å/sec exhibited a decreasing resistivity with falling temperature as it is expected for metal films. At high temperatures the resistivity decreases linearly with the temperature until T=25 K, after which the resistivity remains constant down to T=5 K. Whereas the resistivity of the 10 nm thin film prepared with a rate of 0.2 Å/sec rises with decreasing temperature. At low temperatures the conductivity scales with the square root of T as predicted for 3D films of high disorder. The different temperature dependence of the two films' resistivity is clearly attributed to their different crystal structure as analyzed by transmission electron–beam microscopy. Both films are polycrystalline, but the film grown with the higher rate contains a fiber texture. The film deposited with a low evaporation rate consists of small coherent crystalline grains of only 3 nm size. This confirms the high disorder of the film implied by the electrical measurements.

G6.6
WORK FUNCTION STUDY OF POLYCRYSTALLINE METALS USING A UHV SCANNING KELVIN PROBE. Uwe Petermann, Iain D. Baikie, Bert Lägel, Konrad M. Dirscherl, Robert Gordon Univ, Dept of Applied Physics, Aberdeen, UNITED KINGDOM.

We have undertaken a study of high work function (wf) surfaces as part of an ongoing project searching for efficient target materials for use in Hyperthermal Surface Ionisation (HSI), a new mass spectroscopy ionisation technique. HSI relies on high wf surfaces for the production of positive ions. Polycrystalline metals as Re, W, Mo and Pt are particularly interesting materials in this respect as oxidation substantially increases their wf. We present and discuss the following experimental evidence: a) the magnitude and sign of wf changes in terms of adsorbate induced dipoles, b) the effect of molecular hydrogen exposure on the clean surface, and c) the effect of subsequent oxygen exposure. Using a novel UHV Scanning Kelvin Probe we have followed the oxidation kinetics of polycrystalline metals at different temperatures and examined the effects of oxidation, flash annealing and sputter-anneal cleaning cycles via high-resolution wf topographies. Our results indicate in particular Re as a suitable HSI target material exhibiting a wf increase of 1.05 eV at 300 K increasing to 1.95 eV at 800 K. Sputter-cleaned surfaces exhibit significant surface roughening even after annealing, which dramatically affects the second oxidation phase. We have also examined wf changes associated with atomic oxygen (using N_2O) and carbon containing oxides (CO_2) on Tungsten and Molybdenum. We observe that atomic oxygen gives similar results to O_2 but has a much lower initial sticking coefficient. We report that CO_2 actually lowers the wf for substrate temperatures under 650K, the peak wf changes occurs at 850 K and is approximately 1/3 the height of the O_2 or O peak.

G6.7
OBSERVATIONS OF DLA-TYPE GRAIN AGGREGATES IN SPUTTERED Zr FILMS. Eliane F. Chinaglia, Ivette C. Oppenheim, University of São Paulo, Physics Institute, São Paulo, SP, BRAZIL; Mauricio U. Kleinke, University of Campinas, Gleb Watagin Institute of Physics, Campinas, SP, BRAZIL.

Grain aggregates with geometry similar to that predicted by the Diffusion Limited Aggregation Model (DLA) were observed with Atomic Force Microscopy (AFM) in magnetron-sputtered Zr films. The average grain diameter that forms the aggregates is the same as the average grain diameter that forms the remaining of the film. The fractal dimension ($1.46 \leq D_F \leq 1.83$) and the size (1 to 30μm) of the aggregates were studied as a function of substrate temperature ($300K \leq T_S \leq 923K$), and Ar gas partial pressure ($0.8mTorr \leq P_{Ar} \leq 16mTorr$) during film deposition, and as a function of film thickness ($28nm \leq t \leq 250nm$). Substrates of Si/SiO_2 (Si substrates with a thick SiO_2 overlayer), Si{111}, Si{100}, and amorphous-C were used. DLA-type aggregates were observed in Zr films deposited on all substrates, except in those deposited on amorphous-C. Independent of the film's thickness grain aggregates were present. The size of the aggregates decrease with the increase of P_{Ar}, and were not observed for P_{Ar}=16mTorr. We obtained aggregates with $D_F \approx 1.7$ (the D_F value predicted by the DLA model) in films deposited with $T_S/T_M=0.29$, where T_M is the Zr's melting temperature. This value of T_S/T_M is very close to the value predicted in the literature ($T_S/T_M=0.30$) for the microstructural transition of films from Zone T to Zone II. X-ray diffraction data shows that the films' {0002} texture, and crystalline coherence reach saturation values for $T_S/T_M \geq 0.29$. The roughness of the films, calculated from AFM data, presents a minimum around $T_S/T_M=0.29$. We report the first systematic observations of DLA-type grain aggregates in sputtered films. The physical and chemical interactions that lead to their formation are not yet understood, and are a challenging problem. Work supported by FAPESP (Fundação de Amparo à Pesquisa do Estado de São Paulo).

G6.8
AGGLOMERATION MODEL OF Au/SiO_2 AND Cu/SiO_2 SYSTEMS: VOID NUCLEATION AND VOID FRACTAL GROWTH. Jang-Yeon Kwon, Ki-Bum Kim, School of Material Science and Engineering, Seoul National University, Seoul, KOREA; Seok-Hong Min, Department of Metallurgical Engineering, Kangnung National University, Kangnung, KOREA; Ji-Hwa Lee, Division of Chemical Engineering, Seoul National University, Seoul, KOREA.

Agglomeration behavior of 5∼50 nm Cu and Au films deposited on thermal SiO_2 by using DC magnetron sputtering was investigated by Scanning Electron Microscopy (SEM). The deposited metal films were annealed at H_2 and N_2 environment for various times and temperatures. For the Au/SiO_2 system, void nucleation was observed at the initial stage. Then the void grew with the shape of branches (gyration or snowflake). As annealing time increases, the size of branches increased, while the density of void nuclei has not changed. The coalescence of void branches was observed with further annealing. Agglomeration process finished when the void branches covered the whole film. Avrami equation, well-known formula for the phase transformation, can describe the fraction of agglomeration as a function of annealing time. Especially growth morphology of void follows the fractal mode with the dimension of 1.7. This means that the motion of vacancy satisfies the assumptions of Diffusion Limited Aggregation (DLA) model. Unlikely the case of Au/SiO_2 system, agglomeration started by the grain boundary grooving in Cu/SiO_2 system. The final size of Cu cluster decreased as annealing temperature increased, while that of Au cluster unchanged. The result is explained by the difference of agglomeration sequence in each systems.

G6.9
EFFECT OF INTERMETALLIC FORMATION ON THE RELIABILITY OF THIN LAYERED SOLDER JOINTS IN BALL GRID ARRAY PACKAGES. Lorraine C. Wang, Reinhold H. Dauskardt, Stanford University, Dept. of Materials Science and Engineering, Stanford, CA.

As the microelectronics industry continues to develop increasingly complicated devices with extremely small feature sizes, creating reliable interconnect systems becomes a significant challenge. Currently, there is significant interest in using electroless processes to produce the metal thin film stacks that make up the bond pads in solder ball grid array (BGA) packages. These electroless processes produce a more uniform film thickness, but the resulting metal stacks have exhibited less mechanical reliability than that of traditional electrolytic stacks. For surface mount applications, this can be a critical problem because solder in the BGA configuration does not only provide an electrical connection but also serves as the sole mechanical connection between the bond pads on the chip package and those on the circuit board. This presentation discusses a system consisting of an eutectic Pb-Sn solder sandwiched between stacks of Cu, Ni, and Au thin films. During the solder reflow process, the Au layer is absorbed and a Ni-Sn intermetallic layer forms between the underlying Ni layer and the solder. After aging at an elevated temperature, an additional Au-Sn intermetallic forms on the Ni-Sn intermetallic. Previous work has shown that the presence of these intermetallics strongly influences the fracture behavior of the solder joints. A fracture mechanics approach was used to measure the adhesion between the solder and bond pads. The samples were tested in cyclic fatigue as well as monotonic loading conditions. These results are discussed in terms of the microstructures present in order to determine possible relationships between the intermetallic formation and the fracture behavior in these joints.

G6.10
STRAIN EFFECTS IN Ho- AND Y- DOPED ($La_{0.7-x}R_x$) $Ca_{0.3}MnO_3$ (R=Ho AND Y) MANGANITE THIN FILMS.
M.S.R. Rao[1], V. Ravindranath[1], Y. Lu[2], J. Klein[2] and R. Gross[2].
[1]Materials Science Research Centre and Department of Physics Indian Institute of Technology Madras, Chennai, INDIA, [2]II. Physikalisches Institut, Universität Köln, Köln, GERMANY.

CMR manganites have attracted great deal of interest in recent years and the understanding of the underlying physical mechanisms offer a great deal of scope to probe in to many aspects of manganites. Mixed valency of Mn (Mn^{3+}/Mn^{4+}) is required to realize the metallic behaviour as well as ferromagnetism in manganites. There exists a direct relationship between the Curie temperature (T_C) and the average ionic radius ($<r_A>$) of the rare-earth (RE)-ion (Ex. La) which is varied by substitution of different RE-ions (Ex. Ho, Dy, Pr) and also by varying divalent alkali elements (Ca/Sr/Ba). Ferromagnetic (FM) ordering has been attributed to the double exchange (DE) interaction between the valence electronic states of Mn^{3+}-O^2-Mn^{4+}. The occurrence of CMR is also due to DE mechanism in conjunction with the effect of lattice distortion in these manganites[1]. Recently, we have shown[2] that substitution of magnetic Ho- ions compared to non-magnetic Y-ion at the La site in ($La_{0.7-x}R_x$)$Ca_{0.3}MnO_3$ (R = Ho and Y) causes a large decrease in resistivity and the changes are more marked with increase in dopant concentration. This observation is very interesting from the view point that both Ho and Y have almost identical ionic radii ($Ho_{i.r.}$ = 1.018Å and $Y_{i.r.}$ = 1.015Å) and hence the internal (lattice) strain leading to changes in tolerance factor is the same in both the case. We have used the targets of 0.05 compositions of Y and Ho doped compounds in the pulsed laser deposition (PLD) of thin films on polished $SrTiO_3$ (STO) substrate. Interestingly, the slight lattice mismatch between STO and LCMO has further decreased the T_C (strain due to mismatch) down to 125 K (160 K on the bulk compounds). In addition to decrease in T_C, the changes in resistivity are the same as that observed in the case of polycrystalline samples, ie., Ho doped films have lesser resistivity. Studies on the effect of annealing on T_C are underway.
References:
[1]H.Y. Hwang et al. Phys. Rev. Lett. 75 (1995) 914.
[2]M.S. Ramachandra Rao et al. Submitted to Phys. Rev. Lett.

G6.11
CVD OF THIN FILMS OF COBALT AND COPPER FROM DIFFERENT PRECURSORS: GROWTH KINETICS AND MICROSTRUCTURE. Anil U. Mane, K. Shalini, R. Lakshmi, Anjana Devi and S.A. Shivashankar, Indian Institute of Science, Materials Research Centre, Bangalore, INDIA.

The growth of thin films by CVD depends on all parameters that influence chemical reactions. Of these, the composition and molecular structure of the CVD precursor employed may be expected to be very significant. We have investigated the growth kinetics of thin films of Cu and Co from a variety of the respective β-diketonate complexes, such as acetylacetonates, dipivaloylmethanates, and ketocarboxylates. The growth rate of films of Cu and Co on various substrates such as SiO_2, glass, single crystal Al_2O_3 and Si{100}, as a function of substrate temperature, reactor pressure, and carrier gas hydrogen or argon flow rate was measured. Film microstructure was studied using optical microscopy, SEM, and STM. Electrical conductivity of films was measured both as a function of temperature and of film thickness. It was found, for example, that films of Cu deposited from Cu{II} ketocarboxylates consist of fine grains {$\sim$ 75nm}, regardless of growth temperature {180°C and above}. The fine grain structure leads to continuous films at very low thickness {$\sim$ 75nm} and to conductivity approaching that of the bulk. The small grain size also leads to strong adhesion to surfaces such as SiO_2. Results of similar studies on Co will also be presented, to illustrate the influence of CVD precursor on the micro/nanostructure of films and their properties. By suitable choice of CVD precursor and of the process parameters, we have been able to obtain films of the recently-identified ϵ-Co phase. Using similar considerations, it is possible by CVD to obtain metal films with microstructures appropriate for devices and structures with very small dimensions.

G6.12
ULTRA FAST GALVANIC COATING AND THIN FILM DEPOSITION OF METALS ON INSULATING SUBSTRATES. Vincent Fleury, LPMC, Ecole Polytechnique, Paris, FRANCE.

There exist many technological processes that allow to deposit metals on substrates. Electrochemical techniques allow to deposit metals on substrates by two essentially different means. The first one is the electroless technique, by which spontaneous reduction of metal salts allows coating of both insulators and metals. This technique is used for example to taylor domestic mirrors. The second technique is galvanic deposition on a metal surface, by which a conducting material, used as electrode, is uniformly coated. Metal foils can be recovered from such deposition techniques.
I present a new electrochemical technique which is a *galvanic* technique that allows to coat uniformly with metals *insulating* substrates, such as glass plates, optic fibers, etc. This apparent paradox is resolved by use of a new type of cell which will be presented. This galvanic technique of deposition allows a fine control on the deposition conditions (esp. grain texture and growth speed) which is impossible to achieve by the electroless technique. The technique allows to form very thin metal coatings in the sub-micron range, hence to produce also very thin metal foils (especially, of copper and silver). Patent filed on October 11th 1999 under No. fr 99 12 644.

SESSION G7/D10: JOINT SESSION:
GRAIN EVOLUTION OF METALS
Thursday Morning, April 27, 2000
Golden Gate B2 (Marriott)

8:30 AM *G7.1/D10.1
TEXTURE, MICROSTRUCTURE, AND ROOM TEMPERATURE RECRYSTALLIZATION IN ELECTROPLATED COPPER FOR ADVANCED INTERCONNECTS. M.E. Gross, Bell Labs, Lucent Technologies, Murray Hill, NJ.

Cu is rapidly being adopted as the primary metallization for interconnects in extreme large scale interconnect (XLSI) devices. The interfaces, microstructure, and texture of the Cu all factor into producing an interconnect that is reliable at dimensions from 0.16μm at the lowest metallization level to several microns at the uppermost levels. Cu interconnects are today being fabricated by electroplating Cu into dual damascene structures consisting of trenches and vias etched in SiO_2. A thin (<1,000Å) sputtered Cu seed layer over a refractory metal-based diffusion barrier serves as the cathode for plating. The texture of the barrier layer as well as the topography of the damascene structure both influence the texture of the electroplated Cu. A new sidewall texture component was identified in damascene Cu samples.[1] Following plating, the electroplated Cu undergoes recrystallization at room temperature that advantageously produces large grains for improved electromigration resistance. This recrystallization process is likewise influenced by the damascene topography.[2] Interestingly, the recrystallization of the EP film at room temperature can also drive grain growth in sputtered Cu underlayers that can be as much as twice the thickness of the EP layer. In this talk, I will review recent results on various aspects of the texture, microstructure, and room temperature recrystallization of electroplated Cu in damascene and dual damascene architectures, with a consideration of the underlying mechanisms. [1]C. Lingk, M.E. Gross, W.L. Brown, Appl. Phys. Lett. 74, 682 (1999). [2]C. Lingk, M.E. Gross, J. Appl. Phys. 84, 5547 (1998).

9:00 AM G7.2/D10.2

MACRO- AND MICROTEXTURE OF COPPER METALLIZATION LAYERS MEASURED BY ACOM IN THE SEM. R.A. Schwarzer, A. Huot, Dept. of Physics, Technical Univ. Clausthal, GERMANY; A.H. Fischer, RM MET, Infineon Technologies GmbH, Munich, GERMANY.

Control of (crystal) texture is important for process optimization of metallization layers. Conventional pole-figure measurement by x-ray diffraction (XRD) is limited to areas larger than 0.1 mm wide. By oscillating the specimen under the stationary primary beam, a spatial average from about 1 cm^2 is obtained. A uniform area scan, however, and hence an unbiased macrotexture is not guaranteed. An additional inconsistency of x-ray pole-figure data may be caused by the variation of information depth with specimen tilt. A further draw-back is the limited availability of x-ray texture goniometers.
Automated Crystal Orientation Measurement (ACOM, EBSD) in the SEM is now widely used for studying microtexture in interconnects on a grain specific scale. Commercial EBSD systems with digital beam scan, however, are not made for scans across large specimen areas at low SEM magnifications. A mechanical stage scan (OIM), on the other hand, is too slow, although the scanned field size is only limited by the travel of the stage.
With the ACOM system named ORKID, both the position of the pattern center (which marks the reference directions) and the specimen-to-screen distance are calibrated automatically, as well as the lens focus is corrected dynamically from scan point to scan point when the beam travels across the steeply tilted surface [1]. Therefore specimen areas as large as with XRD can be scanned to acquire large populations of grains, in addition to mapping small areas at single line width resolution, without sacrificing accuracy of orientation measurement or spatial resolution. At present, speed of ACOM with digital beam scan exceeds 20,000 orientations per hour.
From the ACOM database, the ODF and pole figures have been calculated for comparison with x-ray measurements. The benefits and limitations of texture determination by ACOM will be discussed.
[1] R.A. Schwarzer: Micron 28(1997)249-265

9:15 AM G7.3/D10.3

MICROTEXTURE OF ELECTROPLATED COPPER INTERCONNECTS. R. Spolenak[1], D.L. Barr[1], M.E. Gross[1], W.L. Brown[1], A.A. Macdowell[2], N. Tamura[2], R. Celestre[2], H.A. Padmore[2], B. Valek[3], J. C. Bravman[3], P. Flinn[3], T. Marieb[4], B.W. Batterman[5] and J.R. Patel[5]. [1]Bell Labs, Lucent Technologies, Murray Hill, NJ, [2]Advanced Light Source, Lawrence Berkeley National Lab., Berkeley, CA, [3]Dept. of Materials Science and Engineering, Stanford University, Stanford, CA, [4]Intel Corporation, Portland, OR, [5]ALS/LBL, Berkeley, CA & SSRL/SLAC, Stanford University, Stanford CA.

Future generations of ULSI technology will rely heavily on micron and sub-micron Cu interconnections fabricated in damascene architecture. Damascene Cu structures are typically formed by electroplating Cu into trenches etched into SiO_2. Observations of the texture of Cu in these structures have been made non locally by broad-beam x-ray diffraction (XRD) and locally by electron back-scatter diffraction (EBSD). New texture components have been observed that originate from the sidewalls of the trenches[1]. In this work the technique of x-ray micro-diffraction (XRMD) was applied. White x-rays were focused to a micron spot size by Kirk Patrick-Baez mirrors. The sample was stepped under the micro-beam and a Laue image was obtained at each sample location utilizing a CCD area detector. In order to demonstrate the effect of sidewall on the texture, lines with widths ranging from 0.3 μm to 5 μm and depths of either 0.5 μm or 1 μm were investigated. Subsequently, the lines were examined by broad-beam XRD, focused ion beam (FIB), and EBSD. We will compare the local volume information from XRMD to average volume information from broad-beam XRD and to surface orientation information from EBSD to quantify the microstructure of damascene copper.
[1]C. Lingk, M.E. Gross, W.L. Brown, Appl. Phys. Lett. 74, 682 (1999)

9:30 AM G7.4/D10.4

THE ROLE OF ASPECT RATIO ON MICROSTRUCTURE DEVELOPMENT IN DAMASCENE PROCESSED INTERCONNECTS: MODEL PREDICTIONS AND EXPERIMENTAL RESULTS. John E. Sanchez, Jr., Juan Dominguez, Materials Science and Engineering, University of Michigan, Ann Arbor, MI.

Advanced interconnect structures consist of damascene-processed trench metallization lines in which the aspect ratio, defined as trench depth to linewidth, is significantly greater than 1. Concern for interconnect reliability requires the optimization of both line grain size and texture orientation. Given the anisotropic surface and interfacial energies of the FCC Al and Cu choices for metallization, minimum energy orientations of bamboo grains in the damascene lines are a function of aspect ratio (β) and damascene processing details. We have modeled the competition between driving forces arising from surface trench + bottom interface energy minimization and trench sidewall interface energy minimization. Energetic competitions between variously oriented bamboo grains are illustrated. Results of global energy minimization indicate that (111) out-of-plane + (110) sidewall oriented grains are preferred when the aspect ratio is less than 2, while (110) out-of-plane + (111) sidewall oriented grains are preferred when the aspect ratio is greater than 2. A preferred (100) out-of-plane + (100) sidewall texture orientation is predicted at the transition aspect ratio = 2. Grain texture evolution in high aspect ratio trenches is therefore driven by the minimization of trench sidewall interfacial energy, whereas the grain structure in within low aspect ratio lines is determined by surface and trench bottom interface energy minimization. Comparisons of this model are made to recent experimental results of crystallographic texture and grain size in damascene-processed Al and Cu interconnects. The drag effect of grain boundary grooving on bamboo boundary mobility is described. The surface groove drag places a minimum energetic driving force required for bamboo boundary motion which is dependent on damascene line aspect ratio. The groove drag therefore limits in general the competition between misoriented bamboo grains. However the global minimum energy grain orientations, (111) out-of-plane + (110) sidewall at low aspect ratio and (110) out-of-plane + (111) sidewall at high aspect ratio, are maintained. The effects of groove drag and damascene sequence processing options on damascene microstructure development are described.

10:15 AM G7.5/D10.5

CHARACTERIZATION OF THE MICROSTRUCTURE OF Cu-Al ALLOY/SiO_2 INTERFACES. Pei-I Wang, S.P. Murarka, G.-R. Yang, E. Barnat and T.-M. Lu, Center for Integrated Electronics, Electronics Manufacturing and Electronic Media, Rensselaer Polytechnic Institute, Troy, NY.

Low resistivity ($< 6\ \mu\Omega$-cm)Cu-Al alloys have been recommended for application as the diffusion barriers/adhesion promoters for advanced copper based metallization schemes. This approach to barrier formation is to generate an ultra-thin interfacial layer through Cu alloying without significantly affecting the resistivity of Cu. In this paper the microstructure of the bilayers of Cu/Cu-5at.%Al and vice versa sputter deposited on SiO_2 before and after thermal annealing is investigated by x-ray diffraction (XRD) and transmission electron microscopy (TEM). The x-ray diffraction spectra of Cu-5at.%Al on SiO_2 show that the addition of Al into Cu tends to favor the Cu (111) texture. The Al peak appears on x-ray diffraction spectra after annealing indicates that Al segregates at the interface to promote the interfacial reaction of Cu-Al alloy/SiO_2 interface. These results and TEM microstructure data will be presented and discussed, and will show that films of Cu doped with Al appear to act as a suitable barrier and adhesion promoter between SiO_2 and Cu.

10:30 AM G7.6/D10.6

EARLY STAGES OF SURFACE AND MICROSTRUCTURE DEVELOPMENT DURING GROWTH OF POLYCRYSTALLINE THIN FILMS. John E. Sanchez, Jr., Adriana E. Lita, Materials Science and Engineering, University of Michigan, Ann Arbor, MI.

Surface roughness, grain size and morphology, and crystallographic orientation are important microstructural features of deposited polycrystalline films that often determine the performance and reliability of microelectronic devices. Factors such as surface curvature, grain boundary curvature and surface and/or interfacial energy minimization may separately drive the evolution of structure, while processing and substrate material effects further complicate understanding of film evolution. We evaluate the competing effects of surface curvature minimization, grain boundary groove pinning, surface energy minimization and size-dependent normal grain growth during the early stages of deposited film growth. In addition the effects of substrate material properties such as crystallographic texture and surface energy on deposited film structure are evaluated. Surface roughness, columnar grain size and crystallographic texture were determined for sputter deposited pure Al films on SiO_2 and Al-0.5% Cu films on SiO_2 and Ti substrates using atomic force microscopy, transmission electron microscopy and x-ray pole figure analysis, respectively. Results for sputter deposited Al on SiO_2 substrates illustrate the surface roughness decrease during film growth up to 0.3 μm thickness due to the grain size increase and optimization of Al (111) texture via combined normal and secondary grain growth mechanisms. Results for Al-0.5% Cu films on SiO_2 below 0.1 μm thickness similarly show surface smoothing as the film achieves continuity and which persists as grain growth continues and as Al (111) texture evolves. Al-0.5% Cu films on Ti substrates achieve continuity at a film thickness below 10 nm, and maintain a smaller grain size than Al-Cu films on SiO_2. The development of 5 degree offset Al (111) texture on SiO_2 occurs prior to film continuity, seeding the (111) offset texture in the fully continuous film. In contrast, the 10 nm fully continuous Al films on Ti substrates are randomly oriented with exact Al (111) texture evolving due to combined normal and secondary grain growth. These results suggest that film bulk processes such as size and orientation dependent grain growth, rather than surface capillary forces, are primarily responsible for polycrystalline film surface and structure development.

10:45 AM G7.7/D10.7
OBSERVATION OF LONG-RANGE ORIENTATIONAL ORDERING IN METAL FILMS EVAPORATED AT OBLIQUE INCIDENCE ONTO GLASS. David L. Everitt, X.D. Zhu, Univ of California-Davis, Dept of Physics, Davis, CA; William J. Miller, Univ of California-Davis, Dept of Chemical Engineering, Davis, CA; Nicholas J. Abbott, Univ of Wisconsin, Dept of Chemical Engineering, Madison, WI.

We studied long-range orientational ordering in polycrystalline Au films (10 nm - 30 nm) that are evaporated at oblique incidence onto a glass substrate at room temperature. By measuring the averaged optical second-harmonic response from the films over a 6-mm diameter region, we observed a transition from the expected in-plane mirror symmetry at 10 nm to a surprising three-fold in-plane rotational symmetry at 30 nm. X-ray pole figure analysis performed on these films showed the strong $< 111 >$ fiber texture typical of fcc films, but with a restricted, three-fold symmetric, distribution of crystallite orientations about the fiber axis.

11:00 AM G7.8/D10.8
GRAIN BOUNDARY CURVATURE IN POLYCRYSTALLINE METALLIC THIN FILMS. Alexander H. King, Purdue University, School of Materials Engineering, West Lafayette, IN.

Annealed thin films are typically observed to have mean grain diameters that are approximately equal to the film thickness. The standard explanation of this sheet thickness effect is that it results from a balance of grain boundary curvature in two different directions which, in turn, results from pinning at grain boundary grooves. TEM experiments have been performed to assess this model, and it is found that the predicted curvature about axes in the film plane, is absent. Alternate explanations of the sheet thickness effect are considered. Acknowledgement: this work is supported by the National Science Foundation, grant number DMR 9530314.

11:15 AM G7.9/D10.9
PRECIPITATION IN SUB-MICRON Al(Cu) INTERCONNECTS DURING ELECTROMIGRATION. C.A. Volkert, C. Witt and E. Arzt, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

Studies of θ-phase precipitation during annealing and electromigration of passivated sub-micron Al(0.5wt.%Cu) interconnect segments have been performed in-situ in an SEM. On applying a sufficiently large current density, precipitates nucleated and grew at the anode ends of the segments and dissolved at the cathode ends. By reversing the direction of the current, this process could be reversed. The kinetics of nucleation, growth, and dissolution were studied at 250°C for a range of current densities, as well as in the absence of an applied current. In all cases, the behavior was well described by a model in which the motion of Cu in solution is driven by both the electromigration force and by the solute concentration gradient. A clear barrier to nucleation was observed, at a supersaturation of roughly twice the equilibrium solubility, after which precipitate growth was diffusion rate-limited. By comparing precipitate dissolution kinetics with and without an applied current, values for the effective charge and the diffusion coefficient of Cu were determined. Since the measured Cu diffusion coefficient was several orders of magnitude larger than that in the bulk lattice, and since the interconnect segments have an almost perfect bamboo structure, it is likely that Cu diffusion occurs predominately along the interfaces. The precipitates formed at different sites at the anode ends during each current cycle, suggesting that the microstructure does not determine the nucleation site. It was also observed that precipitates dissolved without leaving voids behind, indicating that the Al moved backwards to replace the Cu either due to a stress-gradients or due to coupling between the Cu and Al fluxes. The implications of these results on the understanding of precipitation, particularly in small dimensions, will be discussed. In addition, it is hoped that results of the temperature dependence will be presented and provide further insights into the dominant mechanisms.

11:30 AM G7.10/D10.10
SIMULATION OF THE $TiAl_3$ FORMATION AT INTERFACES IN INTERCONNECTS. X. Federspiel, M. Ignat, INP, Grenoble, FRANCE; C. Bergman, P. Gas, LMCT, CNRS, Marseille, FRANCE; R. Olligier, CEA, Grenoble, FRANCE; H. Fujimoto, T. Marieb, Intel Components Research, Hillsboro, OR.

It is well known that solid state reactions forming intermetalics in interconect systems, will degradate their electrical reliability and produce mechanical stresses which in certain circumstances will induce damage. For example islands of a $TiAl_3$ compound can be nucleated at the interfaces of stacks of Al and Ti thin films under the effect of heating, producing the above mentioned awkward effects. A solution to avoid these undesirable effects is to produce, prior to the definitive manufacture of a device, a controlled thin layer of the compound, which will remain stable during any further thermo-mechanical solicitation. We have analysed the kinetics of $TiAl_3$ formation, from diferent sort of Al/Ti layered systems. Some of them presented diferent Al/Ti thickness ratio, while others were obtained with intentionally induced pollutions during the films obtentions. A numerical model for $TiAl_3$ growth based on interfacial diffusion mechanisms was confronted to in-situ experiments and observations by TEM, to SIMS analysis and DSC experiments.

SESSION G8: STRESS AND MECHANICAL PROPERTIES OF THIN FILMS
Thursday Afternoon, April 27, 2000
Golden Gate B1 (Marriott)

1:30 PM *G8.1
RAMAN SPECTROSCOPY: A UNIQUE TOOL FOR THE STUDY OF THIN FILMS. Ingrid De Wolf, IMEC, Leuven, BELGIUM.

Raman spectroscopy is a non-destructive optical technique, which can give information on different properties of films, such as composition, crystal structure (crystalline - poly-crystalline - amorphous), crystal orientation, crystal phase, mechanical stress, temperature, and in some cases doping. The spatial resolution of a conventional micro-Raman spectroscope is about 1 um. As such, this technique is unique for the study of different kinds of films. In this paper, these different applications of Raman spectroscopy will be briefly discussed using examples from microelectronics. Special attention will be given to the usage of micro-Raman spectroscopy to measure the local mechanical stress in and near thin films. Mechanical stresses develop at various stages of IC processing. These stresses depend not only on the mechanical and thermal properties of the different films, such as their intrinsic stress and their thermal expansion coefficient, but also on the film dimensions and pitch. With the continued miniaturization and densification of IC devices, the effects of local mechanical stress on reliability of the device often become more important. For example, metal films will introduce stress in the substrate. This stress is especially high near the side and edges of the lines. Reduction of the spacing between two metal films will result in an overlap of these stress fields, with possible defect generation in the substrate as a result. Conventional techniques to measure stress, such as X-ray diffraction, can not really be used to look at these local stress fields. Raman spectroscopy, on the other hand, is very useful if a Raman signal of the film or of the surrounding substrate can be measured. The effects of miniaturization on local stress, as studied with Raman spectroscopy, will be discussed using examples from metal lines with different size and pitch.

2:00 PM G8.2
LOCAL TEXTURE AND STRAIN MEASUREMENTS OF POLYCRYSTALLINE METALS USING X-RAY MICROBEAM. Jin-Seok Chung, University of Illinois at Urbana-Champaign, Urbana, IL; Gene E. Ice, B.C. Larson, J.D. Budai, J.Z. Tischler, Oak Ridge National Laboratory, Oak Ridge, TN; Nobumichi Tamura, Advanced Light Source, Berkeley, CA.

The 3rd generation synchrotron radiation sources and recent developments in x-ray optics have made intense sub-micron x-ray probes possible to study material properties. X-ray microdiffraction using white beam synchrotron radiation is particularly useful to study strain distribution and texture, which are keys to understand grain growth and evolution of polycrystalline materials. The x-ray microbeam station on MHATT-CAT beamline at the Advanced Photon Source is optimized for that purpose. The key elements in the setup are a small-displacement x-ray monochromator, a non-dispersive Kirkpatrick-Baez mirror system, and a CCD with a minimal spatial distortion. Routine measurement of local texture and strain is made possible by automatic indexing of white beam Laue pictures. With a focused x-ray beam, 2-dimensional mapping of texture and strain is possible in a sub-micron scale. With a triangulation technique, it is also possible to do a limited extent of non-destructive depth profiling or 3D mapping. Texture map and deviatoric strain tensors were obtained from deformed metal samples to demonstrate the capability of x-ray microbeam analysis.

2:15 PM G8.3
IN SITU CURVATURE AND DIFFRACTION STUDIES OF Pd FILMS ON Si(001) DURING SOLID-STATE REACTION.
Patrice Gergaud, Olivier Thomas, MATOP, CNRS, Universite Aix Marseille III, Marseille, FRANCE; Bernard Chenevier, LMGP, CNRS, INPG, Grenoble, FRANCE; Veronique Ghetta, LTPCM, CNRS, INPG, Grenoble, FRANCE; Aliette Mouroux, Shi-Li Zhang, KTH, Department of Electronics, SWEDEN.

Understanding stress evolution during silicide formation from thin metal films on Si substrates is crucial for the application of silicides in integrated circuits. It requires detailed knowledge of the mechanical properties of each substance, i.e. metal, silicide(s) and Si, in the reacting system. In situ curvature measurement generally gives access to the overall force in the layered stack, whereas in situ X-ray diffraction (XRD) provides information about the mechanical strain in each crystalline sublayer. In this study, we performed both in situ XRD and curvature measurements on the Pd/Si(001) system. The strain in the Pd and Pd2Si phases was first determined by XRD. Whereas the strain in the silicide decreased all along the solid-state reaction, the stress in the metal first increased and then relaxed. These strains were then translated into force per unit width and compared to the in situ curvature measurement results. A good qualitative agreement between these two different measurements was found. A high compressive stress in the silicide at the very first stage of the reaction was also confirmed. Finally, we used a qualitative model proposed by Zhang and d'Heurle in order to describe the experimental observations and to extract physical parameters such as relaxation rates both in the metal and in the silicide. These results were discussed and compared to other experimental studies.

2:30 PM G8.4
IMPURITY EFFECTS IN THE INITIAL STAGES OF REFRACTORY METAL SPUTTER DEPOSITION STUDIED VIA REAL-TIME IN SITU X-RAY METHODS. J.C. Bilello and S.M. Yalisove, Center for Nanomaterials Sciences, Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI.

In the initial stages of Ta, W or Mo polycrystalline metallization on Si wafers, for thicknesses < 10nm, very large compressive residual stresses in the range of 1 to 5 GPa have been reported. The mechanism(s) responsible for this have been variously attributed to impurities, which may arise from either the native oxide on the substrate, or contamination of the sputter target, or residual gases in the sputter chamber, or any combination of the preceding. Since the adhesion of a film is directly related to the properties of the interfacial zone, and furthermore, because the thickness scale of technological interest for metallizations continues to diminish, the present work focussed on identifying and understanding the role of impurities in causing the initially high residual stresses in the sputtering of these refractories, with special emphasis on Ta. High-resolution x-ray diffraction studies, combined with chemical analysis, showed that a mixture of crystalline Ta and amorphous Ta-O had been formed in the early stages of growth. Real-time x-ray diffraction data was collected in situ from the growing Ta film and enabled us to monitor and ultimately control the amorphous-crystalline phase ratios over a broad range. Such observations were combined to with high resolution electron microscopy to verify and quantify the phase relationships.

3:15 PM *G8.5
STRUCTURE, STABILITY, AND MECHANICAL PROPERTIES OF METAL/NITRIDE SUPERLATTICES. Scott A. Barnett, Department of Materials Science and Engineering, Northwestern University, Evanston, IL.

In this talk, superlattice thin films consisting of metal layers alternated with nitride layers, deposited by reactive magnetron sputtering, are described. Results for epitaxial and polycrystalline films, with individual layers typically 1 to 100 nm thick, are presented and compared. When combining BCC metals, e.g. Mo and W, with rocksalt-structure nitrides, e.g. NbN and ZrN, there is a good structural match on the (001) planes with a 45 degree rotation of the unit cells. As a result of these low-energy interfaces and the immiscibility of the layers, structures grown with a (001) out-of-plane orientation show excellent high-temperature stability. For example, W/NbN superlattices showed no detectable change in structure after annealing at 1000C for several hours. Mo/NbN superlattices were less stable due to the formation of a MoNbN ternary interfacial phase. For equal superlattice layer thicknesses, the initial layers were consumed entirely to form the ternary. On the other hand, for Mo-rich structures, the superlattices were stable, presumably because no further reaction occurred after the NbN layers were consumed to form MoNbN. These superlattices exhibit large hardness enhancements compared to the constituent materials, e.g. as high as 35 GPa for Mo/NbN vs. 3 GPa for Mo and 16 GPa for NbN. The high hardnesses were retained after annealing at elevated temperatures.

3:45 PM G8.6
IN SITU STRESS MEASUREMENT DURING THE GROWTH OF Cu/Ni MULTILAYERS. O. Thomas, T. Bigault, F. Bocquet, MATOP-CNRS, EDIFIS-CNRS, Faculté des Sciences et Techniques de Saint Jérome, Marseille, FRANCE; A. Marty, CEN-SP2M, Grenoble, FRANCE; B. Gilles, LTPCM-CNRS, St Martin d'Hères, FRANCE.

Ni epitaxial films grown on Cu (001) display an interesting range of magnetic anisotropy behaviour. These have been interpreted as a result of a competition between magneto-elastic coupling and shape anisotropy. Magneto-elasticity in ultra-thin layers is still under debate. The detailed investigation of residual strains in ultra-thin Ni films deposited on Cu is necessary to understand these magnetic properties. We present stress and RHEED measurements performed in situ during the MBE growth of Ni/Cu multilayers of variable period. The stress evolution is compared with the case of (111)-textured Cu/Ni multilayers grown by sputtering [1]. The results are discussed in terms of coherency stress and plastic relaxation in both Cu and Ni. Some parameters that can influence the stress behaviour have been studied: deposited thickness, deposition rate and underlayer in-plane parameter. [1] V.Ramaswany, B. Clemens, and W. D. Nix, "Stress evolution during growth of sputtered Cu/Ni multilayers", to appear in Mater. Res. Soc. Proc. (1998)

4:00 PM G8.7
STRUCTURAL AND ELASTIC RESPONSE OF Mo/Ni MULTILAYERS TO ION IRRADIATION. Jérôme Pacaud, Franck Martin, Anny Michel, Christiane Jaouen, Lab de Métallurgie Physique, Poitiers, FRANCE; Philippe Djémia, Francois Ganot, Lab. de Propriétés Mécaniques et Thermodynamiques des Matériaux, Villetaneuse, FRANCE.

Studies of elastic properties of metallic superlattices have revealed strong deviations with respect to predictions based on continuum elasticity in some systems. Brillouin spectroscopy studies have particularly given evidence of a marked softening of the shear elastic constant c_{44} at small modulation wavelengths. Although some studies have been devoted to structural investigations in connection with mechanical properties, the origin and the existence of the elastic anomalies in superlattices and nanocrystalline materials is still not fully understood. The Mo/Ni system, when grown as a multilayer with a period in the range of nanometers exhibits a clear softening of the c_{44} elastic constant. The goal of the present study is to understand the origins of such behaviour for this system by combining mechanical properties studies and detailed structural characterization. The employed techniques are Brillouin scattering, x-ray diffraction and high resolution electron microscopy. The use of ion irradiation in the energy range of 100 keV permits to introduce well controlled structural modifications (stress relaxation, interdiffusion) wether preserving the composition modulation or not. Mo/Ni multilayers with modulation period in the range 3-90 nm were grown by alternately depositing layers of Ni and Mo on Si or single crystal sapphire substrates using ion beam sputtering technique. The as-deposited multilayers exhibits a modification of the elastic properties as the period of the modulation changes. An unexpected anomalous behaviour is also observed in the Mo layers. Ion irradiation has been performed on a 7.7 nm period multilayer at different doses. The as-deposited, the stress-relaxed and the final homogeneous solid solution are then compared in term of structure and elastic response. All the results obtained during these investigations tend to show an out-of-equilibrium presence of nickel in the molybdenum layers even in the as-deposited films. This put forward the interplay between interfacial mixing and stress relaxation.