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 of 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 Modell, 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 Bao, Jin Tang, Mohammad Mansourian, Adam Polcyn, IBM Storage Systems Division, San Jose, CA, Kurt Kuhn, Ken Takano, Michael F. Toney, Andreas Moser, Dieter Wolter, 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 Gbit/in² 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 Gbit/in² using magnetic media that are thermally stable. The very low signal decay rate of 0.2%/km measured on a Squid magnetometer at 350K 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 magnetic crystalline anisotropy (Ku) of the magnetic film. Although Ku as high as 3.7 x 10^6 ergs/cm² has been measured for polycrystalline HCP Co75Pd25/Co13, Ku decreases significantly when higher concentrations of Cr are added to Co and the addition of Ta is 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 anisotropy orientation in the Co-alloy. High coercive squareness [8] is necessary for good overwrite properties. The grain size distribution plays a key role in determining the thermal decay rate and medium 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. Stanislav Balda, Robert Sinclair, Stanford University, Dept. of Materials Science and Engineering, Stanford, CA, Gerard Bertero, Wei Cao, Konmig 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 reducing 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 decreases with underlayer grain size. Considering that the bicrystal grain boundaries usually are not clearly revealed in normal bright-field (BF) Transmission Electron Microscopy (TEM) images, high resolution TEM (HRTEM) images are used to accurately measure the magnetic layer size and the angles between the c-axes of adjacent grains. By carefully controlling the processing conditions, OMo underlayer grain sizes were made to vary from 10nm to 10nm. However, the corresponding Co-Co/Pd grains sizes basically remained unchanged. As the underlayer grain size decreased, the ratio of magnetic layer grain size to underlayer grain size increased from 0.86 to 1.40. HRTEM results show that the formation of bicrystal 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. Moorthy, J. S. K. Wong, J. Zheng, MCPF, Fremont, CA.
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 oriented crystallinity offer 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 of great importance. This paper is concerned with the microstructure of HF sputtered Co-CoPt films grown on silicon substrates without an intermediate layer, and as compared with those which have an intermediate buffer and seed layer. In the process of the comparison of the characteristics of the additional layers to study the crystallographic properties of the Co-alloy films, characterization, the effect of substrate temperature during deposition and the sputtering conditions on the resulting microstructure of the film is also determined.

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

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

The structural requirements for low noise magnetic recording media (small grain size, separation of magnetic particles) are in conflict with those for thermally stable magnetization (large uniform grain size, high uniform magnetic anisotropy). Magnet x'Co-alloy grain size, grain separation and anisotropy in modern thin film media are 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 in the order of 10 to 15 nm, high resolution characterization methods such as transmission electron microscopy (TEM), nanogranule 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 Mr 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 the (FePt/Al) smoothface substrates with a 60 nm NiAl seed layer and a 7.5 nm CrMo intermediate layer. The magnetic media is a CoPtCrCoPt/Co/Nb multilayered CoCoCrPt film with seven different thickness realities resulting in Mrt values from 0.1 to 0.6 mrem/cm². 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 SHELFE 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 SHELFE Program under contract DE-AC05-96OR22464 with Oak Ridge Associated Universities.

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

CHEMICAL ORDERING AND MICROSTRUCTURE OF FePt THIN FILMS WITH PERPENDICULAR MAGNETIC ANISOTROPY: EXPERIMENTAL AND THEORETICAL STUDIES
A. Brunel 1, J.-P. Jezequel 2, M. Vauzelle 2, M. Grenouille 2, J. J. P. M. Hardouin 2, C. G. Trotz 2, 1ANRSE, M. Grenoble, FRANCE; 2Aubin Marty, W. S. Ameller, David Halley, DERMIC, CENG Grenoble, FRANCE.

The L10 ordered phases in FePt, FePd, and CoPt binary alloys in thin films might be promising materials for high density storage media, due to their high magnetocrystalline anisotropy. Depending on the growth temperature, we have found that FePt films grown by EMBE 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 L10 phase with the c-axis perpendicular to the surface. Therefore the ratio between the percentage of magnetic L10 phase and the percentage of non magnetic and disordered phase 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 treatment of the growth substrate can affect the surface morphology of the growing L10 phase. Indeed, combining RHEED, STEM 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. However, the origin of the 3 equivalent variants 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 structural defects decreases as the temperature is increased and STEM images have revealed larger atomic and bi-atomic terraces. This suggests that the diffusion length of the ad-atom on the growing surface may explain the change of 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

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]-multilayers, or Fe-Co alloy films. These effects are the basis of ion beam patterning techniques proposed for future high density storage. Samples were irradiated with He, Ar, or Xe ions at energies between 30 keV and 1 MeV, with doses spanning the range 10^11 - 5 x 10^13 ions/cm². 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 chamneiling. 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 PROFILES OF MAGNETIC MULTILAYER STRUCTURE
B.K. Turner, 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 conformity 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 discussed. 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 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 20 nm Cu film, to be determined. We present evidence for surface roughening 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 8 keV and 8 keV 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 x-ray scattering and we can see the large changes in 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

PHOTOLUMINESCENCE SPECTROCOPY OF PSEUDOMORPHIC THIN FILMS OF THE INVAR ALLOY Fe₈Ni₅₋ₓ,Michael Hochstrasser 1, Ray F. Willis, The Pennsylvania State University, Physics Department, University Park, PA; Frank O. Schumman, 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 Fe₈Ni₅₋ₓ alloys deviate strongly from the 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 also been shown that Fe₈Ni₅₋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 Fe₃N₃₋ₓ alloy films pseudomorphically grown on Cu(100) over the whole emission range. We determined 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 and ARPES experiments we have further investigated to get a better insight into the changing electronic and magnetic structure of Fe₃N₃₋ₓ alloy films. We observe changes at the Fermi surface which indicate magnetic disorder. To understand the magnetic instability the 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. Demangeot, H. Dreysse, IPCMS, Strasbourg, FRANCE; A. Rakotomamonjy, Department of Physics, Florida Atlantic University, Boca Raton, FL.

Choi et al. [Phys. Rev. B 58, 1198 (1998)] have studied the magnetic properties of ultrathin Mn films on for Co/Cu(100) using the in situ magneto-optical Kerr effect (MOKE) and low energy electron diffraction. They found that a two-dimensional Mn/Cu(001) surface alloy is stabilized in the range 0.3-0.8 monolayer of Mn. Also Antel et al. tried to elucidate the origin of exchange biasing in FeMn/Cu(100) by measuring and varying dichroism. Theoretical and magnetic experiments on structures with magnetic FeMn-Cu(001) systems have been carried out using the Tight-Binding Linear Muffin-Tin Orbital (TB-LMTO) method within the density-functional theory (DFT) and its approach is described in the paper. The stability of a Mn monolayer on Co(001) versus the formation of 1 or 2ML thick Mn/Cu(001) alloys, as well as the inverted Co/Mn/Cu(001) system. The Mn-Co alloy is found to be more stable than the Mn-Co alloy on Co(001) substrate, in qualitative agreement with the experimental predictions above. In the case of 2ML thick Mn-Co alloy the magnetic configuration between Mn and Co atoms at the surface and the subsurface is found to be ferromagnetic and antiferromagnetic respectively. We report 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 atoms 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. Methaikhu, P. Vavassori, M. Grimsditch, Materials Science Division, Argonne National Laboratory, Argonne, IL; B. He, School of Applied and Engineering Physics, Cornell University, Ithaca, NY; P. Nenzi, R. Kumar, Institute of Microelectronics, Singapore, SINGAPORE.

We have investigated the size and thickness dependence of a series of Co and permalloy nanoscale arrays using the Magnetic Optic Kerr Effect (MOKE) 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 10nm to 200nm were fabricated on amorphous, on a single substrate, to avoid complications due to changes in microtography and or thickness. Identical patterns were fabricated for 30nm of Co and 20nm and 30nm of permalloy. The effect of element size 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 micromotors will be discussed. Work is Argonne National Laboratory was supported by the U.S. Department of Energy, Division of Materials Sciences, Office of Basic Energy Sciences, under contract W-31-109-ENG-38. P.V. acknowledges support from a Research grant from INFEL-Istituto Nazionale per L'Fisica della Materia.

3:15 PM G2.5/F4.5
TEM IN SUPPORT OF INDUSTRIAL RESEARCH. Philip M. Rice, Stuart S. Park, Robin F. Farr, Mary Moore, IBM Almaden Research Center, San Jose, CA; William J. Gallagher, IBM-T J Watson Research Center, Yorktown Heights, NY; Tsun Lu, 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 Focus Ion Beam (FIB) has also become an invaluable instrument due to its ability to make site specific TEM specimens from production wafers and heads. Experiments 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 element migration between multilayers will also be discussed.

3:45 PM G2.6/F4.6
ULTRATHIN ALLOY FILMS. F.O. Schumpp, Freie Universitat Berlin, GERMANY; J. Gobin, 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 Fe-Co-Ni₃₋ₓ and Fe-Ni₃₋ₓ films grown on Cu(100). For high Fe concentrations we find contracting behavior of the two systems. In the case of Fe-Co-Ni₃₋ₓ we observe a small magnetic signal associated with a moment-volume instability. However for the Fe-Co-Ni₃₋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 volumes of Fe upon alloying with Co and Ni. It is now well-established that for Fe-Co/Ni alloy can exist in two different magnetic states. These are high-spin (HS) and low-spin (LS) state, the former having the 1s vacancy and the latter not. However, we have shown that when we grow Fe-Co-Ni₃₋ₓ films on Cu(100) we observe a larger magnetic moment than bulk Fe. This moment enhancement in the case of Fe-Co-Ni₃₋ₓ alloys allows us also to resolve fine-structure in the Fe 3p emission. This was theoretically predicted, but for the experiments it was not known. We have used high-resolution photoemission 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 magnetochemistry for ultrathin films is discussed.

4:00 PM G2.7/F4.7
THE STRUCTURE OF POLYCRYSTALLINE AND EPITAXIAL GALVA-MAGNETIC MULTILAYERS AND SPIN VALVES GROWN BY SPUTTER DEPOSITION. Hong Geng, Resn Loeloe, John W. Hecken, Martin A. Cirrin, Dept of Materials Science and Mechanics, Michigan State University, East Lansing, MI; Resn Loeloe, William P. Prat Jr., Dept of Physics and Astrophysics, Michigan State University, 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 thickness and interlayer coupling as well as the magnetic properties of the constituent layer. In the present study, a number magnetic multilayers (Co/Cu)ₓ and spin-valves Cu/Mn/Cu/Pt and Pt/Au/Pt/FM/Fe/Mn) were produced by dc magnetron sputtering (0 = Permalloy, 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 [110] Nb that were deposited on (1250) Al₂O₃ substrates. The grain size, morphology, and orientation of the epitaxially grown materials 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 (CHM and HRTEM). In polycrystalline samples, CHM has revealed well-defined multilayered structures, which in most cases display columnar grains range up to 50 nm in diameter. The HRTEM study, supplemented with fast Fourier transform (FFT) analysis and image simulations, indicated that these non-equilibrium phases exist in certain regions of these spin-valve layers. In epitaxial samples, CHM and HRTEM revealed large numbers of threading dislocations in the single crystal Nb buffer layer. Subsequent sputtered of Cu, Pt and Co resulted in two growth variants predicted by the stacking sequence of the (111) epitaxial planes. Significant growing 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 CFME, and by the US NSF under grant MHSEC DMR 99-05988.

4:15 PM G2.8/F4.8
SCANNING-APERTURE PHOTOEMISSION MICROSCOPE FOR MAGNETIC IMAGING. Gary M. McClelland and Charles T. Bettner, 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, photoemission is excited by a laser focused on a 10 micron spot. A scanning aperture above the sample 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 lithographically defined CoPt multilayer thin films on sapphire have been obtained. From a cesium film, a high quantum efficiency of 0.002 was observed from 458 nm laser light. Circular dichroism of ±4% is recorded by alternating the circular polarization of the light while scanning an aperture. A scanning aperture 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 30-nm-thick CoPt structure in the film, where 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 aperture size 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 MAGNETOELECTRIC COUPLING IN ULTRATHIN Fe FILMS DEPOSITED ON GaAs[100]. Parvice Gengrass, Magali Potiero, Olivier Thomas, MATOP, CNRS, Universite Aix-Marseille III, Marseille, FRANCE; Claude Lalliau and Bruno LEPINE, Andre Guiveur, 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 strong effects on both the magnetic and electronic properties of the heterostructure. It has been shown previously that high-quality epitaxial thin films can be grown on GaAs[100] 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 bulk Fe(001) film should have fourfold symmetry. The source of this uniaxial component remains an open question. In the present measurements, an effort is made to contribute to this is the epitaxial strain, through a magnetoelectric 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 are attributable to the magnetic anisotropy of iron thin films. Moreover the sign and magnitude of the magnetoelectric coupling seem to depend on the film thickness or film strain [1]. We thus calculated the magnetic anisotropy using both bulk and thin film magnetoelectric coupling. I. 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 II B3 (Merritt)

8:30 AM *G3.1/F5.1
SPIN POLARIZED CURRENTS IN MAGNETIC TUNNEL JUNCTIONS. Peter M. Levy, Kuixing Wong, Dept. of Physics, New York University, New York, NY; Shufeng Zhang, Dept. of Physics, University of Missouri, Columbia, MO; Charles B. Sommers, Lab. Phys., Stanford, CA; Andrew C. Gossard, Technical University Budapest, Budapest, HUNGARY; Peter Weinberger, Technische Universitat Wien, Vienna, AUSTRIA.

The effects 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 the majority and minority spin channel in the electrode go in opposite directions, so as to produce important changes in the magnetoresistance for relatively small changes in the bias voltage of the interface. A positive spin selectivity at the interface, i.e. changing the type of spin-polarized electrons at the barrier, and introduces different sets of states for electrons to tunnel through. While there is no spin dependence to these insulating barriers switching, any of the type electronic, charge transfer, or 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.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 resistance are required. We show that the resistance of MTJ devices with aluminum tunnel barriers can be reliably and controllably varied over more than 8 orders of magnitude by simply varying the thickness of the aluminum layer. These requires extremely thin aluminum layers just a few nanometers 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 fringe fields from domain walls in a ferromagnetic layer acting on the other ferromagnetic layer. * Work supported in part by DARPA.

9:30 AM G3.3/F5.3

Co/A12O3/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 the case of magnetic structures, Cu interlayers grown on Co give an exponential decay of the tunneling magnetoresistance (TMR) with e1/2=20% while Cu grown on A12O3 has a much longer decay length of 0.7%km. 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 Hirokawa, Yong-Ping Xu, Charles Guenter, Tony Blind, 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 laser light has been demonstrated [2]. In this study, we produced samples of 3 nm Au/5 nm FM/GaAs [(100), 1023 = ±8, pT=1021 m2s-1] and attached two Al electrical contacts to the Au layer and one ohmic contact to the bottom of the substrate. Conventional S-V-J measurements were carried out to define the Schottky characteristics of the samples. Circularly polarised laser light (515 ≤ λ ≤ 780 nm) was used to excite electrons with a spin polarization perpendicular to the film plane in the samples. A helicity-dependent photocurrent, dependent upon the magnetisation configuration of the film [θ, η] or [θ, η/2] and the Schottky barrier height, was detected with NIPe as the FM. An almost constant difference between the helicity-dependent photocurrent for perpendicular and parallel configurations in observed as 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 suppression 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

SESSION G4/F6. Joint Session: GMR and Spin Valves I

Wednesday, April 26, 2000
Golden Gate B3 (Marrilott)

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. Myres, D.C. Ralph, Laboratory of Atomic and Solid State Physics, Cornell University, Ithaca, NY; J.A. Kuzie, 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 torque to the magnetic layers to reverse their moment, by means of 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 5-18-layer nanofabricated metal point contact is used to inject current into a Co/Cu/Co trilayer and (in work led by Kuzie, 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 achieving negative by -2 to -4 \(\mu\)A/cm² are in the point contacts and less than 10 \(\mu\)A/cm² 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 instead stimulating 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. Magan, G. Spinos, Naval Research Laboratory, Washington, DC; R.D. McMichael, P.J. Chen and W.P. Egelhoff, Jr., National Institute of Standards and Technology, Gaithersburg, MD.

The effect of annealing on the microstructure and giant magnetoresistive properties of NiO/Co/Cu/Co bottom spin valves is investigated using 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 375 °C. This decrease is attributed to a decrease in the roughness of the Cu and Co layers. In annealed specimens, grain boundary growth is also observed in the perpendicularly magnetized NiO layer at the Cu/Co interface, and the location of these grain boundary features varies with the Cu/Co interfaces. An increase in the Neel ‘orange-peel’ coupling between the ferromagnetic Cu layers, resulting from the increased roughness of the Cu/Co interfaces, accompanies the degradation of the GMR.
Cu layer thickness. The most dramatic variation occurs for Co thicknesses around 30% of the Co minority mean free path of Co (about 4 nm). This increase may be due to the magnetic behavior of the Co layer being dominated by the minority Co species. This work was supported by DARPA under contract MDA 973-97-1-1083.

2:45 PM G5.4/F7.4
MANUFACTURABILITY OF GMR HEADS. D.G. Inoue, National Institute of Standards and Technology, Materials Science and Engineering Laboratory, Gaithersburg, MD.

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 5-10nm 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 stack with a synthetic antiferromagnet as the pinned layer. Results show strong indications for the full stack 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/Anti-Ferromagnetic 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. H. Kingsley, C.A. Dykstra, and E. Dan Dahlberg, Appl. Phys. Lett. 69, 3923-3931 (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 model predictions, it does provide a more accurate determination of the average F/AF EA as the interface. As such, the F/AF EA calculated by models and studies of the systematics of the phenomenon 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 PmMn/CoFe BILAYERS DURING ANTIFERROMAGNETIC PHASE TRANSFORMATIONS. P. Rozenberg and B. Daniels, Scanning Recording Heads, Minneapolis, MN.
PmMn is one of several candidate antiferromagnetic materials for building of spin valve giant magnetoresistance (GMR) sensors used in magnetic recording heads. The deposited crystal structure of PmMn 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 [Dylikie, et al., MRS Fall '96] 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 stresses in the sample are dominated by the fcc to fct transformation. In spite of this good agreement, stress changes can be divided into three 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 a PmMn/CoFe bilayers before and after annealing. The magnetic behaviors of these films has been found to depend strongly on the PmMn composition, the behavior of the transformation has been studied for PmMn 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. Bonerich, 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 ultrathin Co films and demonstrated this technique in a pseudo spin valve. Thin films of Co, Ni_{85}Fe_{15}(Py) and Co/Cu/Co pseudo spin valves were DC magnetron sputtered deposited on obliquely 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 Co. With 7.5 nm Ta, the measured value of $H_k$, 163 mT, is comparable to that of single crystal hcp Co. A large increase in $H_{k}$ is also observed for Py films, thus the magnetocrystalline anisotropy apparently does not play a large role. Relatively narrow FMR resonance lines indicate a surprising degree of uniformity in the anisotropy. Additionally, the damping parameter is nearly isotropic. Cross-sectional HRTEM reveals the Ta/amorphous metal interface to be corrugated with ridges and grooves extending perpendicular to the Ta surface plane. The presence of roughly aligned ridges and grooves on the length scale of a few nm is expected to give rise to magnetostrictive 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 magnetostrictive charges will exist on the sides of the ridges, giving rise to a demagnetization field in the plane of the film.

SESSION 6G POSTER SESSION
POLYCRYSTALLINE METAL AND MAGNETIC THIN FILMS

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


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 microscopic changes in a function of temperature. One of the systems studied is Al-Ge. Sputter-deposited thin films of Al-Ge are high chemic 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 crystallization. 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


139
This presentation will examine experimental and modeling results concerning the thermophysical properties of polycrystalline oxides on the surface of film. 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 microstructural phonon energy transport, such as the enhanced phonon mean free path and electron-electron interactions are used to examine the thermal diffusivity in films of different microstructures as measured under various experimental situations. Modeling results will be used to generate complete simulations of the thermal behavior of Thin Film oxides with the dependence of the surface temperature. The dependence of the thermal diffusivity on grain structure, grain shape and film thickness will be examined.

G6.4
EFFECT OF WATER IN PECVD TEOS OXIDE UNDERLAYERS ON CRYSTAL ORIENTATION IN Al-AI-O/TiN/Ti/FILMS.
Kage Aoki, Tomoyuki Yoshida, Yasuichi Minakawa, Toyota Central RD Inst., Inc., Nagakute, Aichi, JAPAN.

Electromigration (EM) failures of Al-based interconnects such as Al-Cu/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-Cu films. Generally, Al-Cu/TiN/Ti interconnects are formed on plasma-enhanced chemical vapor deposited molybdenum nitride films (PECVD TEOS) oxides of 1-1/2% O2 or in a combination of CVD TEOS oxides, which usually contain water related species (H2O and SiOH). In this work, we investigated the effect of these species in oxide films on crystal orientations of sputtered-deposited Al-Cu/TiN/Ti films. In these oxide films, enhanced trench saturation in Al-Cu/TiN/Ti films was observed at various r.f. powers (60, 200 and 350 W), and some of these oxides were then annealed at various temperatures (450, 550, 650 and 800°C). These oxides were then deposited onto oxide films at 350°C by TiN/Al metal deposition on the surface free energy; this results in Ti film growth toward <0001> preferred orientation by self-assembly of Ti atoms. Furthermore, an Al-Cu/TiN/Ti/Ti film fabricated from the improved TiN 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-Cu/TiN/Ti films are expected to have a higher resistance against EM.

G6.5

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 decreases with decreasing temperature. At high temperatures the conductivity scales with the square root of T as predicted for 3D films of high disorder. The different temperature dependence of the film's resistivity is clearly attributed to their different microstructure as analyzed by transmission electron 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 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, Inan D. Bakke, Bert Lügel, Konrad M. Dirschel, 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 spectrometry ionisation technique which relies on the production of positive ions. Polycrystalline metals such as Al, Ti, W, Mo and Pt are particularly interesting materials in this respect as ionisation substantially increases their WF. We present and discuss the following experimental results: (a) the magnitude of work function changes in terms of adsorbed induced dipole, 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 time dependence of the thermionic emission of metal films at different temperatures and examined the effects of temperature, flash annealing and sputter-annal cleaning cycles via high-resolution WF topographies. Our results indicate in particular a suitable HSI target material exhibiting an increase of the H temperature dependent H2WF of the order of 1.95 eV at 800 K. Sputter-cleansed surfaces exhibit significant surface roughening even after annealing, which dramatically affects the secondary ionisation yield. We have also examined WF changes induced by oxygen adsorption on Pt(111) and carbon containing oxides (CO2) on Tungsten and Molybdenum. We observe that atomic oxygen gives similar results to O2 but has a much lower initial sticking coefficient. We report that CO2 actually lowers the WF for substrate temperatures under 650K, the peak WF changes occur at 850 K and is approximately 1/8 the height of the O2 or O peak.

G6.7
OBSERVATIONS OF DLA-TYPE GRAIN AGGREGATES IN SPUTTERED Zr FILMS. 
Elaine F. Chinaglia, Ivetz O. Oppenheim, University of São Paulo, Physics Institute, São Paulo, SP, BRAZIL.

Grain aggregates with geometry similar to that predicted by the Diffusion Limited Aggregation Model (DLA) were observed with Atomic Force Microscope AFM experiments on sputtered Zr films. The average grain diameter that forms the aggregate is the same as the average grain diameter that forms the remaining of the film. The fractal dimension (1.18±0.03) and the size (1.1±0.3 μm) of the aggregates were studied as a function of substrate temperature (300K≤T≤923K), and Ar gas partial pressure (0.8mTorr≤P≤160mTorr) during film deposition, and as a function of film thickness (50≤t≤600nm). Substrates of Si/SiO2 (Si substrates with a thick SiO2 overlayer), Sr[111], Sr[100], and amorphous-C were used. Zr-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 decreases with the increase of PZr, and were not observed for PZr≤160mTorr. We obtained aggregates with Df=1.7 (the Df value predicted by the DLA model) in films deposited with T≤Tc=0.29, where Tc is in the Zr’s melting temperature. This value of Tc/MD is very close to the value predicted in the literature (Tc/MD=0.33) for the microstructural transition of films from Zone T to Zone II. X-ray diffraction data shows that the films’ (0002) texture, and crystallite coherence length saturation value (1=2.44 Å) of Zr/SiO2 were measurable. The roughness of the films, calculated from AFM data, presents a minimum around Tc/MD=0.29. We report the first systematic observations of DLA-type grain aggregates in sputtered films. The physical and chemical interactions between the film and the substrate 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/SiO2 AND Cu/SiO2 SYSTEMS: VOID NUCLEATION AND VOID FRACTAL GROWTH.
Jing-Yeon Kwon, Kibun 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-Hee Lee, Division of Chemical Engineering, Seoul National University, Seoul, KOREA.

Agglomeration behavior of 5-50 nm Cu and Au films deposited on thermal SiO2 by using DC magnetron sputtering was investigated by Scanning Electron Microscopy (SEM). The deposited metal films were annealed at H2 and N2 environment for various times and temperatures. For the Au/SiO2 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 description of 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 fraction of vacancy satisfies DLA (Diffusion Limited Aggregation) (DLA) model. Unlike the case of Au/SiO2 system,
agglomeration started by the grain boundary growing in Cu/SiO₂ system. The final size of Cu cluster decreased as annealing temperature increased while that of S dropped. The result is explained by the difference of agglomeration sequence in each system.

G6.6

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

As the microelectronics industry continues to develop increasingly complex and more densely packed devices, the reliability of the feature sizes in existing 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 electroplating 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 electroless 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-Ni intermetallic layer 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

SOIL INFLUENCE IN HO- AND Y-DOPED (La₀.₇5₋ₓRx₀ₓ)₃₋ₓCaₓMnO₃ [R=Ho AND Y] MANGANITE THIN FILMS. M. S. Raju, Y. Randhram, V. L. Li, J. Klein, and R. Gross, II Materials Science Research Centre and Department of Physics India Institute of Technology Madras, Chennai, INDIA, "II. Physical Sciences Institute, Universite de Kiel, Kiel, GERMANY.

CMM 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 valancy of Mn (Mn⁺³/Mn⁺⁴) is required to realize the metallic behaviour as well as ferromagnetic in manganites. There exists a direct relationship between the Curie temperature (Tc) and the average ionic radius (R̅) of the rare-earth (RE) ion but which is varied by substitution of different RE ions (e.g. Ho, Dy, Pr) and also by varying divalent alkaline elements (Ca/Sr/Ba). Ferromagnetic (FM) ordering has been attributed to the exchange interaction and the valence exchange interaction is the main distorting factor in the ferromagnetic state (R̅<R̅MnO₃). The occurrence of CMR is also due to DE mechanism in conjunction with the effect of lattice distortion in these manganites. Recently, we have shown that substitution of magnetic Ho ions compared to non-magnetic Y ions at the La site in (La₀.₇5₋ₓRx₀ₓ)₃₋ₓCaₓMnO₃ (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⁺³ = 1.083 Å and Y⁺³ = 1.015 Å) and hence the internal strain leads to changes in tolerance factor is the same in both the cases. We have used the targets of 0.05 compositions of Y and Ho doped compounds in the pulsed laser deposition (PLD) of this films on polycrystalline SrTiO₃ (STO) substrate. Interestingly, the slight lattice mismatch between STO and LCMO has further decreased the Tc (strain due to mismatch) down to 125 K (160 K on the bulk compounds). In addition to decrease in Tc, the changes in resistivity are the same as that observed in the case of polycrystalline samples, i.e., Ho doped films have lesser resistivity. Studies on the effect of annealing on Tc are underway.

References:

G6.11

CVD OF THIN FILMS OF COBALT AND COPPER FILM DIFFERENT PRECURSORS: GROWTH KINETICS AND MICROSTRUCTURE. Anil U. Mane, K. Shilini, R. Lalchmii, Anjana Debi and R. Bhattacharjee, 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 precursors 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-diketone complexes, such as neocuproine, dipivaloylmethane, and diketocarbonyles. The growth rate of films of Cu and Co on various substrates such as SiO₂ glass, single crystal Al₂O₃ 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] ketylcarbonylates consist of fine grains (~ 75nm), regardless of growth temperature (180°C and above). The fine grain structure leads to continuous films at very low thickness (~ 75nm) and to conductivity approaching that of the bulk. The small grain size also leads to strong adhesion to surfaces such as SiO₂. Results of similar studies on Co will also be presented, to illustrate the influence of CVD precursors 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 Cu-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

ULTA FAST GALLIUM COATING AND THIN FILM DEPOSITION OF METALS INSULATING LAYERS. Vincent Fleury, LPMC, Ecole Polytechnique, Paris, FRANCE.

There exist many technological processes that allow to deposit metal thin films. Electrochemical techniques allow to deposit metals on substrates by two essentially different means. The first one is the electrochemical technique, by which spontaneous reduction of metal salts allows coating of both insulators and metals. This technique is used for example to tailor 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 metal insulating substrates, such as glass plates, optical 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 direct control on the deposition conditions (e.g., film texture and growth speed) which is impossible to achieve by the electrochemical 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. 89 12 644.

SESSION G7/D10: JOINT SESSION: GRAIN EVOLUTION OF METALS

Thursday March 23, 2000
Golden Glove B (Marriott)

8:30 AM-G7.1/D10.1

TEXTURE, MICROSTRUCTURE, AND ROOM TEMPERATURE RECRYSTALLIZATION IN ELECTROLEPTERED 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 (XL38) 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. The Cu interconnects are targeted being factionated, dipivaloylmethane, and diketocarbonyle Cu into dual damascene structures consisting of trenches and vias etched in SiO₂. A thin [<0.001μm] sputtered Cu seed overlay 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. Following plating, the electroplated Cu undergoes recrystallization at room temperature which advantageously produces large grains for improved electromigration resistance. This recrystallization process is likewise influenced by the damascene topography. 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

9:00 AM GT 2/D10.2
MACRO- AND MICROTEXTURE OF COPPER METALIZATION LAYERS MEASURED BY ACOM IN THE SEM. R.A. Schwaner, A. Hout, Dept. of Physics, Technical Univ. Clausthal, GERMANY; A.H. Fischer, IMMET, 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 nodes larger than 0.1 mm wide. By oscillating the specimen under the stationary primary beam, a spatial average from about 1 cm² is obtained. A uniform area scan, however, and hence an unbiased microtexture is not guaranteed. An additional inconsistency of conventional pole-figure data may be caused by the variation of information depth with specimen tilt. A further drawback is the limited availability of x-ray texture goniometers. Automated Crystal Orientation Measurement (ACOM, EBSD) in the SEM, not only in a two-dimensional scan, but also 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 not suitable, 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 direction) and the specimen (x,y)-position 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 pole figure textures. 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 textures. The benefits and limitations of texture determination by ACOM will be discussed.


9:15 AM GT 7.3/D10.3

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 SiO₂. Observations of the texture of Cu in these structures have been made non locally by broad-beam x-ray diffraction (XRD) [1]. XRD is limited by electron backscatter diffraction (EBSD), which essentially has no texture sensitivity. New texture components have been observed that originate from the sidewalls of the trenches. In this work the technique of x-ray micro-diffraction (XMRD) was applied. White x-rays were focused to a micron spot size by Kirkpatrick-Baez mirrors. The sample was stepped under the micro-beam and a Linx 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 mm to 0.5 mm and depths of either 0.5 or 1 mm were investigated. Subsequently, the lines were examined by broad-beam XRD, focused ion beam (FIB), and EBSD. We will compare the XMRD x-ray texture from XMRD to surface orientation information from broad-beam XRD and to surface orientation information from EBSD to quantify the microtexture of damascene copper.


9:30 AM GT 7.4/D10.4
THE ROLE OF ASPECT RATIO ON MICROSTRUCTURE DEVELOPMENT IN DAMASCENE PROCESSED INTER-CONNECTS: MODEL PREDICTIONS AND EXPERIMENTAL RESULTS. John E. Sanchez, Jr., Juan Dominguez, Materials Science and Engineering, University of Michigan, Ann Arbor, MI.

Surface roughness, grain size and morphology, and crystallite 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 surface and surface and/or interfacial energy may significantly affect 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 and energy minimization by examining thin film behavior on (111) surface energy minimization and size-dependent normal grain growth during the early stages of deposited film growth. In addition to the strain and normal grain growth effects, substrate effects on deposited film structure are evaluated. Surface roughness, columnar grain size and crystallographic texture were determined for sputter-deposited pure Al films on SiO₂ and Al-0.5% Cu films on SiO₂ and Ti substrates using atomic force microscopy, transmission electron microscopy, x-ray pole figure analysis, respectively. Results for sputter-deposited Al on SiO₂ substrates illustrate the surface roughness decrease during film growth up to 0.3 μm thickness due to the grain size increase and optimization of (111) texture via combined surface and interfacial energy minimization mechanisms for films on SiO₂ below 0.1 μm thickness similarly show surface smoothing as the film achieves continuity and which persists as grain growth continues and Al (111) texture evolves. Al-0.5% Cu films on Ti substrates achieve
10:45 AM G7.7/D10.7
OBSERVATION OF LONG-RANGE ORIENTATIONAL ORDERING IN METAL FILMS EVAPORATED AT OBLIQUE INCIDENCE ONTO GLASS ELECTRODES. Y.X. Zha, University of California-Davis, Dept. of Physics, Davis, CA; William J. Miller, University of California-Davis, Dept. of Chemical Engineering, Davis, CA, Nicholas J. Abbott, University of Wisconsin, Dept. of Chemical Engineering, Madison, WI.

We studied long-range orientational ordering in polycrystalline Au films (10 nm - 30 nm) that were 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 showed the strong <111> fibre texture typical of polycrystalline films, but with a restricted, three-fold symmetric, distribution of crystalline orientations about the fibre 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 for 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 measure 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. Acknowledgments: this work is supported by the National Science Foundation, grant number DMR 9520314.

11:15 AM G7.9/D10.9
PRECIPITATION IN SUB-MICRON Al/Cu INTERCONNECTS DURING ELECTROMIGRATION. C. 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/Cu interconnect segments have been performed in-situ in an SEM. On applying a sufficiently large current density, electromigration nucleates nuclei at the midpoints 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, as a superimposition 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 in Al were determined. 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 predominantly 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-gradient 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 the dependence of the temperature dependence will be presented and provide further insights into the dominant mechanisms.

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. Lee, B.C. Larson, J.D. Budi, J. 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 the beamline at the Advanced Photon Source is optimized for this 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 of strain mapping. Texture mis 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 Pt Films ON Si(001) DURING SOLID-STATE REACTION.
Patrice Germaud, Olivier Thomas, MATOP, CNRS, Université Aix Marseille III, Marseille, FRANCE; Bernard Chevrel, LMGP, CNRS, INPG, Grenoble, FRANCE; Veronique Ghez, LTPCM, CNRS, INPG, Grenoble, France; Aliette Macrocous, Shi-Li Zhang, KTH, Department of Electronics, Sweden.
Understanding stress evolution during silicide formation from thin metal films on Si is crucial to the exploitation of the silicide in integrated circuits. It requires detailed knowledge of the mechanical properties of each substance, i.e., metal(s), 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 studies on the Pt/Si(001) system. The strain in the Pt/Si/Pt/Si layers was 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. Bickel and S.M. Yalovoe, 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 deposition on Si wafers, for thicknesses <10 nm, very large compressive stress levels in the range of 1 to 5 GPa have been reported. The mechanism responsible for this has 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 deposition 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 focused 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 TaO had been formed in the early stages of deposition. 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 ratio over a broad range. Such observations were combined with high resolution electron microscopy to verify and quantify the phase relationships.

3:15 PM G8.5
STRUCTURE, STABILITY, AND MECHANICAL PROPERTIES OF METAL/AMIDE 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 45.5° rotation of the unit cells. As a result of these low-energy interfaces and the immiscibility of the layers, structures grown on the (001) out-of-plane orientation show excellent high-temperature stability. For example, W/NbN superlattices showed no detectable change in structure after annealing at 1000 °C for several hours. Mo/NbN superlattices were less stable due to the formation of a MoO layer on the interfacial plane. 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., up to 35 GPa for MoNbN vs. 3 GPa for Mo and 16 GPa for NbN. The high hardnesses were retained after annealing at elevated temperatures.