SYMPOSIUM GG

GG: Advanced Characterization Techniques for Data Storage Materials

December 2 - 3, 2003

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* Invited paper

SESSION GG1: STM-based Magnetic Characterization
Chair: Hans-Joachim Guentherodt
Tuesday Morning, December 2, 2003
Berkeley (Sheraton)

9:00 AM *GG1.1
Spin-Polarized Scanning Tunneling Microscopy as an Ultimate Tool for Future Magnetic Data Storage Technology. Roland Martin Wiesendanger, Institute of Applied Physics, University of Hamburg, Hamburg, Germany.

In order to probe and tailor magnetic properties at the spatial limit we have combined the scanning tunneling microscope (STM) with spin-sensitivity. This is achieved by the use of ferro- and antiferromagnetically coated probe tips offering a high degree of spin-polarization of the electronic states involved in the tunneling process. Magnetic domain imaging with sub-nanometer-scale spatial resolution has been demonstrated for magnetic transition metal as well as rare earth metal films. Ultra-sharp domain walls were discovered in ultra-thin iron films while for antiferromagnetic samples, the different orientation of magnetic moments could directly be made visible at the atomic level. The phenomenon of magnetic hysteresis was observed for the first time at the single-digit nanometer length scale and has directly been correlated with microscopic processes of domain nucleation and domain wall motion. We also studied magnetic vortex structures in mesoscopic-scale ferromagnetic systems which are of relevance for current developments in MRAM technology. Magnetic switching phenomena of nano-scale magnetic islands and nanoparticles were studied by time-dependent spin-sensitive STM imaging. It will be shown that granular thin films exhibit a complex magnetic switching behaviour due to the statistical distribution of grain sizes, grain shapes and inter-grain spacings. Finally, we will discuss the application of spin-sensitive STM measurements to individual atoms and molecules on magnetic substrates.

9:30 AM GG1.2
Bias-Voltage-Dependent Magnetic and Non-magnetic Corrugation in Atomic-Scale Spin-Polarized Scanning Tunneling Microscopy. Arthur Reed Smith[1], Rong Yang[1], Haiqiang Yang[1] and Walter R. L. Lambrecht[2]; [1]Physics and Astronomy, Ohio University, Athens, Ohio; [2]Physics, Case Western Reserve University, Cleveland, Ohio.

Spin-polarized scanning tunneling microscopy (SP-STM) is a very promising technique for the study of surface magnetism since detailed magnetic contrast can be obtained down to the atomic scale.[1-3] Recently, we have investigated the surface magnetic structure of Mn_3N_2 (010) - a model row-wise antiferromagnetic surface. This surface is prepared by molecular beam epitaxy with Mn effusion cell and radio frequency N plasma source. Normal in-situ STM images reveal a row structure with row spacing 6.06 *Å*. Using magnetic-coated tips, a modulation in the height of the rows is observed, resulting in a super-period of 12.12 *Å*, indicating the surface magnetic ordering. The spatially-correlated magnetic and non-magnetic components are separated using a procedure we previously developed.[3] The magnetic and electronic components are modeled using integrated local spin density of states calculated from first-principles. In previous work, we have shown good agreement of the model with the experimental data at a specific energy below the Fermi level.[3] Yet, not much has been known about the variation of the atomic-scale magnetic contrast with energy. In current work, we are investigating this energy dependence by measuring the spin-polarized and non-spin-polarized modulation amplitudes over a wide range of sample bias voltage. We find that the non-magnetic amplitude is greatest at slight positive sample voltage, whereas the magnetic amplitude is greatest at slight negative sample voltage. The ratio of these two amplitudes gives the overall tip-sample polarization. Specific features of the experimental result will be discussed, including a reversal of the polarization at positive sample voltage. In addition, the generally good agreement of these results with the polarization calculated from the energy-dependent local density of states from first-principles will also be discussed. 1. S. Heinze *etal.*, Science **288**, 1805 (2000). 2. D. Wortmann *etal.*, Phys. Rev. Lett. **86**, 4132 (2001). 3. H. Yang *etal.*, Phys. Rev. Lett. **89**, 226101 (2002).

10:15 AM *GG1.3
The Evolution of Magnetism from Adatoms to 2D Nanostructures. Harald Brune, Physics, EPFL, Lausanne, VD, Switzerland.

The magnetic anisotropy energy K is closely related to the anisotropy of the orbital magnetic moment ∆m_L. Both quantities can be measured with X-ray magnetic circular dichroism (XMCD). In the gas phase transition metal atoms have large orbital moments given by Hund's rules (m_L = 3 μ_B for Co), whereas in the bulk m_L is almost entirely quenched due to the crystal field and electron delocalization (m_L = 0.15 μ_B in hcp Co), and therefore K is small (40 μeV/atom for hcp Co). This comparison suggests spectacular magnetic properties for low coordinated atoms at surfaces. We report on the evolution of K and m_L as a function of size starting from single atoms and going up in size almost atom by atom for Co on Pt(111) using XMCD measurements carried out in-situ to self-organized growth. Monomers have giant values of K = 9.3 ± 1.6 meV and m_L = 1.1 ± 0.1 μ_B (Gambardella et al. Science 2003). These values strongly decrease as coordination goes up in dimers and trimers etc. The XMCD-results are complemented by Magneto-optical Kerr effect (MOKE) measurements for larger 2 D nanostructures confirming a strong coordination effect on the anisotropy energy. For Co/Pt(111) and for Co/Au(778) we find K = 1 meV for the low-coordinated edge atoms favoring out-of-plane magnetization, whereas due to the shape anisotropy the atoms in the interior weakly favor in-plane magnetization with K = -30 μeV (Rusponi et al. Nature Materials 2003). The temperature dependence of the susceptibility shows that mutual interactions between the monodomain particles are absent up to densities of at least 26 Tera particles/in^2.

10:45 AM *GG1.4
Ballistic Electron Magnetic Microscopy of Magnetic Nanostructures. Robert Buhrman, Cornell University, Ithaca, New York.

Ballistic electron magnetic microscopy (BEMM) is a scanning-tunneling-microscope -based technique for imaging the magnetic structure of ferromagnetic thin films and nanostructures. This technique utilizes the spin-dependent differences in the quasi-ballistic attenuation length of a hot (~ 1 eV) electron beam passing through ferromagnetic material. This allows the imaging of the local magnetic orientation of a thin film sample relative to that of a ferromagnetic "spin-analyzer" layer that is separated from the sample by a thin normal metal spacer. Since the resolution is determined by the width of the STM injected ballistic electron beam, BEMM is capable of clearly imaging magnetic changes that occur over a rather short length scale, ~ 1 nm. BEMM is distinguished by the fact that the technique does not require an external magnetic field to be applied to the sample during the measurement, and is also compatible with imaging a sample in the presence of a strong applied field. BEMM can also be employed to examine the magnetic structure of ferromagnetic layers buried as much as 20 nm beneath the surface of a thin film multilayer system. Thus BEMM is effective for studying low coercivity materials and the magnetic behavior of thin film multilayers and nanostructures of the type that may be employed, for example, in spin-valve sensors and in thin-film magnetic information storage devices. BEMM is also a powerful tool for measuring spin-dependent hot-electron transport through magnetic thin film systems of potential importance to future spintronics applications. I will review the capabilities and limitations of BEMM and discuss some examples of its application to imaging magnetic changes in thin-film nanostructures with dimensions <= 100 nm that are in some cases approaching the paramagnetic limit, and to studies of spin-dependent transport.

SESSION GG2: MFM and Optics-Based Magnetic Probes
Tuesday Afternoon, December 2, 2003
Berkeley (Sheraton)

2:00 PM *GG2.1
Precessional Magnetization Reversal in Hard-magnetic, High Density Particulate Media. Hans C. Siegmann, Stanford Linear Accelerator Center, Stanford University, Stanford, California.

The very short and intense Gaussian magnetic field pulses that can be generated in solids by passing through relativistic bunches of electrons induce precessional switching of the magnetization M in granular perpendicular magnetic media. The experiments show that the switching is stochastic, independently of specific properties of the CoCrPt-type materials and also independently of the presence of a soft magnetic under-layer. This demonstrates that the precession of the spins becomes inhomogeneous, and that the Landau-Lifshitz equation can only describe the average motion of M under these conditions. The experiments thus indicate that there is an ultimate limit to the speed of magnetic switching useable in magnetic recording at the ~ 10^{-12} sec level. It also shows that these pulses provide a new, conceptually simple technique, besides electron/hole pair excitation with pulsed lasers and spin injection, to evaluate the dynamics of ferromagnetic spins underlying all fast magnetization dynamics. This work is done in collaboration with I. Tudosa, C. Stamm, A.B. Kashuba, F. King, J. St ö hr, G. Ju, B. Lu, D. Weller.

2:30 PM *GG2.2
High Resolution and Quantitative Magnetic Force Microscopy. Hans J. Hug, P. Kappenberger and S. Martin; Institute of Physics, NCCR Nanoscale Science, Basel, Switzerland.

In the past decade magnetic force microscopy (MFM) has been an important tool to characterize magnetic media for magnetic data storage and many other applications. However, the exponential increase in storage density has led to a reduction of the dimensions of the magnetization transitions that is close or already beyond the present resolution limit of conventional MFM. Using optimized tips and operation methods we routinely obtain a lateral resolution around 10nm. MFM methods with 10nm resolution that can routinely be used are vital for the further development of magnetic data storage media. In addition to the high lateral resolution, methods for the calibration of magnetic force microscope tips [1] were developed. Such calibration methods were used to determine the uncompensated spin density at the interfaces of exchange coupled CoO/CoPt multilayers [2]. [1] P. J. A. van Schendel, H. J. Hug, B. Stiefel, S. Martin, and H.-J. Güntherodt, J. Appl. Phys. 88, 435-445 (2000) [2] P. Kappenberger, H.J. Hug, J.B. Kortright, O. Hellwog and E. E. Fullerton, submitted.

3:30 PM GG2.3
Phase separation in 30%La5/8Sr3/8MnO3 + 70% LuMnO3 bulk sample studied by scanning microwave microscopy.
Jino Lee[1], Jewook Park[1], Ahram Kim[1], Kookrin Char[1], Soonyong Park[2], Namjung Hur[2] and Sang-Wook Cheong[2]; [1]Dept of Physics, Seoul National University, Seoul, South Korea; [2]Dept of Physics & Astronomy, Rutgers University, Piscataway, New Jersey.

Using our scanning microwave microscope (SMM), we have investigated the phase separation phenomenon in 30%La5/8Sr3/8MnO3(LSMO) + 70% LuMnO3(LMO) polycrystalline pressed powder sample, in which the LSMO phase is a perovskite ferromagnetic metal while the LMO phase is a hexagonal ferroelectric insulator. Under the optical microscope using a polarized light, the sample exhibits clear grain boundaries among the grains in size of several microns. When the electrical properties of the sample were imaged using our SMM, the sample showed a significant contrast between the metallic LSMO and the insulating LMO grains. The boundaries observed by SMM agreed with those observed under the optical microscope. When we further investigated the metallic phase using a magnetic force microscope(MFM), the metallic phase identified by the SMM clearly showed ferromagnetic signal, providing solid evidence that the metallic phase is indeed the ferromagnetic LSMO. However, we have noticed a slight difference between the images generated by SMM and MFM. While the images by the MFM looked almost identical, the images measured by the SMM displayed a noticeable distinction. We believe the difference comes from the fact that the depth scales the two different microscope techniques probe are different. In other words, the MFM signals are only sensitive to the surface layer of a few tens of nm thickness, while the SMM probes a deeper region, a few microns. We infer that the contrast among the metallic LSMO grains measured by the SMM is due to the different thickness of the grains.

3:45 PM GG2.4
Spin wave propagation effects in thin NiFe films. Hans Nembach, Markus Weber, Jurgen Fassbender and Burkard Hillebrands; Fachbereich Physik, University of Kaiserslautern, Kaiserslautern, Germany.

For a deeper understanding of the magnetization dynamics and damping mechanisms in thin magnetic films spin wave propagation effects are important. These manifest themselves in a reduction of the magnetization vector magnitude within a finite sample area. In order to determine all magnetization vector components in absolute units a special calibration and measurement procedure has been performed. The measurement principle is an extension of a standard method used for the separation of the in-plane components of the magnetization vector [1,2]. It additionally leads to the polar magnetization vector component, which cannot be neglected in magnetization precession experiments. The magnetization dynamics of polycrystalline NiFe films has been investigated by means of time-resolved magneto-optic Kerr effect magnetometry and compared to numerical macrospin simulations based on the Landau Lifshitz Gilbert equation. The observed reduction of M^2 during application of a short magnetic field pulse indicates the generation of propagating spin waves. Even though M^2 is not conserved the magnetization trajectory is well described by the macrospin simulations. [1] H. F. Ding, S. Potter, H. P. Oepen, J. Kirschner, J. Magn. Magn. Mater. 212, L5 (2000). [2] R. Lopusnik, PhD thesis, University of Kaiserslautern (2001).

SESSION GG3: Electron Surface and Imaging Probes I
Wednesday Morning, December 3, 2003
Berkeley (Sheraton)

9:00 AM *GG3.1
Scanning Probe Energy Loss Spectroscopy (SPELS).
R. E. Palmer, Nanoscale Physics Research Laboratory, University of Birmingham, Edgbaston, United Kingdom.

Nanometre-scale science has the potential to contribute to a range of different application areas, including data storage, chip fabrication, catalysis and bio-sensors. While the fabrication and imaging of novel structures down to the nano and even atomic scale are both now established, significant developments are needed if surface chemical analysis is to keep pace. The Scanning Probe Energy Loss Spectrometer (SPELS) is a hybrid instrument which combines an STM tip with an electron energy analyser [2,3]. In SPELS one detects electrons backscattered from the tip-surface junction (at room temperature) when the tip is operated in field emission mode. Experiments to date have focussed on the information content of the spectra. Energy loss features in the range 1-300eV have been detected, corresponding to optical absorption spectra beginning in the IR and proceeding through the visible and UV into the X-ray region. The best energy resolution achieved so far is 0.6 eV. The plasmon modes of graphite, the Si(111)-7x7 surface and Ag clusters have all been detected, together with evidence for interband excitations. Very recent data suggests that the SPELS instrument can in addition excite *local* secondary electron emission spectra, reflecting the surface bandstructure. The prospects for local core level spectroscopy and the theoretical limits on the spatial resolution in SPELS ($\sim$1 nm) will also be discussed. 1. R.E. Palmer, S. Pratontep and H.-G. Boyen, Nature Materials, in press (July 2003). 2. B.J. Eves, F. Festy, K. Svensson and R.E.Palmer, Appl. Phys Lett **77** 4223 (2000). 3. R.E. Palmer, B.J. Eves, F. Festy and K. Svensson, Surf. Sci. **502 – 503** 224 (2002); R.E. Palmer, Surf. Interface Anal. **34** 3 (2002).

9:30 AM *GG3.2
Development of Scanning Tunneling Microscope Combined with Synchrotron Radiation Light Source for Elemental Analysis. Takeshi Matsushima, Taichi Okuda, Toyoaki Eguchi, Ayumi Harasawa, Atsushi Kamoshida, Masanori Ono, Masayuki Hamada, Toyohiko Kinoshita and Yukio Hasegawa; Institute for Solid State Physics, Univ. Tokyo, Kashiwa, Japan.

Scanning tunneling microcopy (STM) provides us information on atomic structure of material surface with high spatial resolution. But it is not easy to obtain chemical or elemental information except a few special cases using inelastic tunneling. The reason is simply because STM probes tunneling current which basically reflects valence electronic states. In order to access elemental information, one needs to probe core-level electrons like the cases of Auger electron spectroscopy and X-ray photoelectron emission spectroscopy. In this paper, we will present our recent effort for picking up core-level electrons with STM by exciting them with UV light source. We have developed a ultrahigh vacuum (UHV) STM system which can be operated with the tip and sample illuminated with synchrotron radiation (SR) light (SR-STM). Using SR light source, we can tune the energy of light to excite core electrons of the particular element and detect ensuing photoemitted electrons, advantageous for elemental identification. We first tested a performance of the system by using a standard sample of the Si(111) -7x7 surface. All experiments were made at BL-19A, Photon Factory, KEK, Japan. In spite of mechanically and electronically noisy conditions, we could successfully take atomically resolved images of the surface, implying a stability of our system as a UHV-STM. We then checked a detection of photoemitted electrons under the SR illumination. To test this, we measured photoemitted electrons as a function of photon energy from 96eV to 106eV, which crosses the Si 2p adsorption edge (99eV). We found a sharp increase in the amount of the photoelectron at the edge energy, demonstrating a capability of detecting excited core electron and thus possibility of elemental identification using this technique.

10:15 AM GG3.3
Imaging, Manipulation, and Analyzing with Nanometer Precision: Application of the Nanoworkbench. Olivier Guise[1], Hubertus Marbach[1], Jeremy Levy[2], John T. Yates[1,2] and Joachim Ahner[3]; [1]Department of Chemistry, University of Pittsburgh, Pittsburgh, Pennsylvania; [2]Department of Physics, University of Pittsburgh, Pittsburgh, Pennsylvania; [3]Seagate Technology, Pittsburgh, Pennsylvania.

It is well appreciated, that as the size of material objects approaches nanometer dimensions, the materials structural and electronic properties change. The investigation of these effects forms a broad active area of current research aimed at the optimization of nanometer sized materials properties for use in a large field of technologies including electronic devices and high-density data storage. We report the development of novel subnanometer manipulative and analytical devices for imaging, chemically analyzing and manipulation of nanometer scaled material. Two different versions of the nanoworkbench are operating currently at the Surface Science Center of the University of Pittsburgh and at the Seagate Research Center in Pittsburgh. The instrument at Seagate consists of a modified commercially available high resolution scanning electron microscope (HRSEM lateral resolution down to 1 nm at 10 KV) in combination with a set of four unique nano-manipulators, which can

be equipped with different kind of probes for nano-scale materials characterization in pressure ranges from 10^{-1} to 10^{-7} mbar. At the University of Pittsburgh a completely home-built UHV version of the nanoworkbench is in operation. Several inter-connected UHV chambers allow the in-situ deposition of thin-films and conventional surface analysis. The resolution of the SEM of the UHV system is limited to about 50 nm. We report first results obtained by using both versions of the nanoworkbench, where we succeeded in writing patterns of ultra-small carbon-containing dots (8nm in diameter) with high position accuracy (<5nm) by electron-beam-induced dissociation of carbon containing background gases. In parallel studies, the electron-stimulated carbon film creation on Si(100) using various pure hydrocarbon gases was studied in UHV by AES, TPD and XPS. The thermal stability of the carbon film and dots has been studied over the temperature range from 300K - 1400K, where the carbon converts to SiC, giving high thermal stability. We are planning to use these carbon templates for the growth of Germanium quantum dots, important for the development of novel quantum electronic devices. This work was supported by DARPA QuIST through ARO contract number DAAD-19-01-1-0650.

10:30 AM GG3.4
Characterization of Magnetic Recording Media Using Analytical Electron Microscopy. James Wittig[1], James Bentley[2] and Neal Evans[2]; [1]Electrical Engineering and Computer Science, Vanderbilt University, Nashville, Tennessee; [2]Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Composition inhomogenieties in magnetic recording media strongly influence magnetic properties such as intrinsic coercivity and magnetocrystalline anisotropy. In addition, chemical inhomogenieties that produce a paramagnetic grain boundary layer between the grains also affect the signal-to-noise ratio of the recording process. Whereas exchange decoupling decreases transition noise, non-magnetic material increases the D.C. noise of the read-back signal. Since the crystal dimensions of modern magnetic recording media are on the nanometer scale, characterization of the composition variations in the grain interiors and grain boundaries requires analytical methods with nanometer spatial resolution. These experimental data are critical for accurate micromagnetic modeling of the recording process. Over the last seven years we have optimized experimental methods specifically for elemental mapping of Co based alloy thin films using energy-filtered transmission electron microscopy (EFTEM) and spectrum imaging with a field-emission gun scanning transmission electron microscope (FEG-STEM). Extensive work has been done to understand the Cr grain boundary segregation phenomenon in sputtered CoCr(PtTa) alloys. Addition of Ta to CoCr results in greater Cr grain boundary segregation with fewer intragranular defects and higher coercivity compared to a binary alloy or an alloy of CoCrPt. Analysis of EFTEM elemental maps and high-resolution TEM images from identical areas suggests that a layer-by-layer growth mode for the CoCrTa thin film versus an island-like nucleation and growth mode for CoCrPt is responsible. The EFTEM elemental maps provide quantitative data for the dimensions of the paramagnetic grain boundary layer as well as the changes in the magnetocrystalline anisotropy of the grain interiors from Cr depletion. Recently, these experimental methods have been adapted to address the more challenging characterization problem of boron segregation in CoCrPtB sputtered thin films. Spectrum imaging using electron energy-loss spectroscopy (EELS) has proven to be superior to EFTEM for boron analysis owing to significantly greater statistics that improve the accuracy of the background subtraction. This paper will draw from these examples to describe the capabilities and limitations of analytical electron microscopy for characterization of nanostructured magnetic materials.

10:45 AM GG3.5
Structural Evolution of Epitaxial Magnetic Thin Films: a UHVTEM Study. J. Yu[1,2], Mark Yeadon[1,2], W. Tian[3], H. Sun[3], X.Q. Pan[3], C.B. Boothroyd[2], R.A. Lukaszew[4] and R. Clarke[5]; [1]Materials Science, National University of Singapore, Singapore, Singapore; [2]IMRE, Singapore; [3]Dept of Materials Science and Engineering, University of Michigan, Ann Arbor, Michigan; [4]Dept of Physics, University of Toledo, Toledo, Ohio; [5]Dept of Physics, University of Michigan, Ann Arbor, Michigan.

Understanding structure/property relationships in metal: metal oxide systems are of substantial importance in applications ranging from magnetic storage and spintronic devices to supported catalysts. Epitaxial growth can be achieved in certain metal/oxide systems and the properties of the films may strongly depend on the interfacial structure as well as growth mode. Using a modified ultrahigh vacuum transmission electron microscope (the MERLION system), we have investigated the nucleation and growth of Ni thin films on electron transparent metal oxide substrates. The system is equipped with solid source electron beam evaporators together with gas injection capability, all within the polepiece of the electron microscope which has a base pressure of 1.5x10-10Torr. The paper will focus on structural evolution in the early stages of nucleation and growth, through to the formation of continuous films. Complimentary data from STM and MOKE experiments will be presented.

11:00 AM GG3.6
Momentum-Resolved Electron Energy-Loss Spectroscopy as a Magnetic Anisotropy Probe. Yasuo Ito[1,2], Michel van Veenendaal[1,2], Brandon D Armstrong[1], Russell E Cook[2], Dean J Miller[2] and Nanda Menon[3]; [1]Physics, Northern Illinois University, DeKalb, Illinois; [2]Materials Science Division, Argonne National Laboratory, Argonne, Illinois; [3]Gatan Inc., Pleasanton, California.

Spin polarized transport across interfaces is one of the critical issues for the spin-based electronics, and understanding the basic mechanisms and limiting factors of such transport is one of the major challenges. Thus, the use of probes directly sensitive to the magnetic anisotropy of the thin film bulk and its interfaces (typically a few nm wide) are of great interest. So far, intense research activities to probe the magnetic anisotropy have been centered on polarized synchrotron x-ray spectroscopy techniques. However, the spatial resolution of these techniques is currently limited to tens of nanometers. Therefore, there is a compelling need for new techniques capable of probing the magnetic anisotropy on the few-nanometer scale or less. Momentum-Resolved Electron Energy-Loss Spectroscopy (MREELS) has been applied as a probe for nanometer-scale magnetic anisotropy. For testing the technique, α-Fe_2O_3 (hematite) was analyzed as a model sample, which has an antiferromagnetic ground state with spins on specific neighboring planes oriented in opposite directions at room temperature. MREELS is an Electron Energy-Loss Spectroscopy (EELS) technique available in Scanning Transmission Electron Microscope (STEM). It senses the oientation dependence of element-specific electronic structure of anisotropic materials. When making use of the polarization depedence of EELS on the 3d transition metal L-ionization edges, the magnetic anisotropy is expressed in the form of a magnetic linear dichric (MLD) spectrum [1]. The Fe L_{23} difference spectrum was successfully extracted from a pair of spectra acquired from a (001) oriented α-Fe_2O_3 micro-crystalline using an electron probe (less than 1 nm diameter) with different convergence angles, i.e., collecting different ratios of parallel to perpendicular components of the scattering vector. For validation, a theoretical MLD spectrum was also calculated using the atomic multiplet theory for the octahedrally coordinated Fe^{3+} ion. The experimental result agrees with the simulated MLD spectrum indicating that the spin orientation is parallel to the basal plane of the unit cell. This is also consistent with a published x-ray MLD result [2]. The STEM MREELS will provide new element-specific anisotropic magnetic structure information with the highest lateral spatial resolution (less than a few nm) among available bulk-sensitive magnetic anisotropy probes. [1] J. Yuan, N.K. Menon, J. Appl. Phys., 81, 5087 (1997). [2] P. Kuiper et al., Phys. Rev. Lett., 70, 1549 (1993). [3] This work is supported by the State of Illinois under HECA, NIU URA program, and work at Argonne, carried out in the Electron Microscopy Center, is supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences, under Contract #W-31-109-ENG-38.

11:15 AM GG3.7
Nanoscale Compositional Characterization of ONO Stacks for Charge-Storage Structures. Igor Levin[1], Mark Kovler[2], Richard Leapman[3] and Yakov Roizin[2]; [1]NIST, Gaithersburg, Maryland; [2]Tower Semiconductor Ltd, Migdal Haemek, Israel; [3]National Institutes of Health, Bethesda, Maryland.

Silicon oxide-nitride-oxide multilayers (ONO stacks) attract considerable interest for the charge storage structures in non-volatile memory devices. The critical structural and compositional parameters that affect electrical performance of ONO based devices include the physical density of the amorphous oxide/nitride layers and depth distributions of both oxygen and nitrogen atoms. In this study we applied (i) spatially-resolved electron-energy loss spectroscopy (EELS) in a transmission electron microscope and (ii) secondary ion mass spectroscopy (SIMS) to analyze elemental distributions in the differently processed ONO stacks deposited on Si. EELS measurements were made using (i) energy-filtered TEM and EELS spectrum-line acquisition in a fixed-beam high-resolution TEM equipped with a thermionic electron source, and a post-column energy filter, and (ii) EELS spectrum-imaging in a dedicated scanning transmission electron microscope (STEM) equipped with a cold field-emission source and an EELS spectrometer. Our results revealed radiation-induced nitrogen segregation to silicon/oxide interfaces; the extent of nitrogen segregation increased visibly with increasing the radiation dose. The EELS metrology was optimized to obtain artifact-free data. The results of combined EELS and SIMS chemical analyses were correlated with electrical performance of ONO-based flash-memories.

11:30 AM GG3.8
Direct Observation of Charge Transfer at MgO(111) Surface.
Arun K Subramanian[1], Laurence D Marks[1], Oliver Warschkow[2] and Donald E Ellis[2]; [1]Materials Science and Engg, Northwestern University, Evanston, Illinois; [2]Physics, Northwestern University, Evanston, Illinois.

MgO is the typical rocksalt oxide that is commonly used as a model system for testing calculations on ionic oxides. Though most studies thus far on MgO have focused on the non-polar (001) surface, the polar (111) surface has attracted attention in recent years. Experimental studies on this surface have identified various reconstructions that are remarkably stable. Critical to understanding the properties of the surface is knowledge of the electronic charge distribution. We have employed Transmission Electron Diffraction (TED) combined with Direct Methods to study the $sqrt3xsqrt3R30°$ reconstruction on the polar (111) surface of MgO. The valence charge distribution of the various surface species was directly refined from the experimental data. This is the first direct observation of charge transfer phenomenon in a surface structure. The structure we find contains a magnesium atom on top, a hole localized in the next oxygen layer and increased covalency manifested by some charge transfer from these oxygen atoms to the top magnesium atom and increased electron density between them. The MgO surface structure was separately optimized using a density functional theory surface slab model. This provides for both an independent confirmation of the experimentally determined x,y (or in-plane) atomic positions as well as the missing z atomic positions. The partial charges that we obtain for the surface atoms are in reasonable agreement with empirical bond-valence estimations.

11:45 AM GG3.9
Imaging Titanium Structures at Buried Interfaces in PEEM.
Karen M. Siegrist[1], V. Ballarotto[2], M. Breban[3], R. Yongsunthon[4] and E. D. Williams[3]; [1]Optical Technology, National Institute of Standards and Technology, Gaithersburg, Maryland; [2]Laboratory for Physical Sciences, College Park, Maryland; [3]Physics, University of Maryland, College Park, Maryland; [4]Geology, University of Maryland, College Park, Maryland.

Despite the limited (~2 nm) escape depth of photoelectrons used for imaging in photoelectron emission microscopy, PEEM has a powerful capability for imaging buried structures. We present results which demonstrate imaging of titanium lines buried under at least 0.5 μm of PECVD oxide. Physically, this result arises due to photon-stimulated charge injection from the buried substrate into the covering oxide layer, from which surface photoemission then occurs. Differences in the photoinjection rate from the surface of the Ti lines and the underlying Si substrate yield a primary contrast mechanism. Charge injection from the buried interface also results in charge trapping and generation of electric fields around the buried structures. These fields result in pronounced edge effects which clearly delineate the buried structures in images formed by photoemission from the oxide surface. The lateral surface fields generated can be stronger than the longitudinal accelerating field of the microscope, over short distances, while it was found in previous work that lateral fields as small as 5% of the total field produce strong contrast effects. PEEM is therefore a sensitive detector of potential gradients at the sample surface, but also at buried interfaces. Because field effects are nonlocal, the field-induced effects on intensity are clearly visible in images showing the buried titanium lines with an applied bias. The field-generated effects on intensity are similar in magnitude and spatial extent for both the bare surface of biased titanium lines and the same surface buried under 0.5 μm of oxide. Although this non-intrusive surface-sensitive technique has primarily been used to investigate nanometer scale surface chemistry and morphology, these results demonstrate that PEEM is a potentially powerful technique for real-time imaging of electronic devices including buried structures. Supported by the Laboratory for Physical Sciences and University of Maryland MRSEC.

SESSION GG4: Electron Surface and Imaging Probes II
Wednesday Afternoon, December 3, 2003
Berkeley (Sheraton)

2:00 PM *GG4.1
Coincidence Spectrosopy of Correlated Electron Pairs.
J. Krischner, Max-Planck-Institute fur Mikrostrukturphysik, Halle, Germany.

We observe electron-electron scattering events in which a primary electron (with some tens of eV kinetic energy) scatters from a valence band electron. If both electrons escape from the solid, they may be detected in coincidence, while both kinetic energies are determined by a time-of-flight technique. Though the time resolution of the apparatus is only of the order of half a nsec, the intrinsic interaction time is much shorter, presumably of the order of fs. This is assured by detecting only one electron pair at a time, suppressing accidental coincidences. The experiment is extremely surface sensitive since the mean free path for low energy electron is short and both electrons must escape without energy loss. We essentially probe the electronic structure of the first two monolayers, on the time-scale of the interaction time. We studied W, Cu, and LiF. The experiments were extended by developing a pulsed spin-polarized electron source and using ferromagnetic Fe single crystals. The electron-pair intensity distribution curves are dependent on the relative orientation of the primary electron spin and the sample magnetization. The dominant scattering channel produces two spin-polarized electrons in a singlet state. The results are interpreted in terms of the spin-resolved surface density of states of Fe(110). * this work was done in collaboration with S. Samarin, A. Morozov, and J. Berakdar

2:30 PM GG4.2
Visualization of electrons and holes localized in the gate thin film of Metal-Oxide-Nitride-Oxide-Semiconductor type flash memory using the Scanning Non Dielectric Microscopy.
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By applying Scanning Non Dielectric Microscopy (SNDM), we succeeded in clarifying the position where electrons/holes existed in the gate SiO2-Si3N4-SiO2 (ONO) film of the Metal-Oxide-Nitride-Oxide-Semiconductor(MONOS) type flash memory. SNDM is an atomic force microscopy measurement technique where a ring electrode is used in conjugating with the cantilever. Alternating electric field is biased between this electrode and the sample, and the capacitance variation of the surface neighborhood of the sample is detected. The charge injected in the ONO film induces a permittivity change of the ONO. The charge accumulated in the film can be detected by SNDM as a change in capacity by scanning the surface of the ONO film. Thus the visualization of the charge distribution becomes possible. In the SNDM image, a bright contrast existed in the neighborhood of the source in the channel area, when the electron was injected. On the other hand, a black contrast existed in the source neighborhood in the channel area, when the hole was injected. These images can be interpreted as the visualization of the polarization of the electron-hole pair. When the positive charge exists in the insulation film, the minus charge is introduced from the drain area in neighborhood in the channel and a positive charge-minus charge pair is formed. When the electric field is impressed from the substrate side, the capacity of this polarization becomes small. Therefore, the contrast becomes black. On the other hand, when the minus charge exists in the ONO film, the positive charge is generated from the Si substrate of p type. In this case, when the electric field is impressed from the substrate side, capacity grows, and the contrast becomes white. Therefore, the SNDM signal will be reversed by the positive and negative of the charge in the ONO film. It was also clarified that the electron is localized in the Si3N4 part in the ONO film. On the other hand, the results showed that the hole not only existed in Si3N4 but also extended to the Bottom-SiO2.

2:45 PM GG4.3
Temperature Controlled Scanning Nonlinear Dielectric Microscopy. Koya Ohara and Yasuo Cho; Research Institute of Electrical Communication, Tohoku University, Sendai, Miyagi, Japan.

Recently, ferroelectric data storage system (ferroelectric random access memory (FeRAM) or high-density ferroelectric recording system [1]) has been studied by various researchers. And investigating the characterization on surface of ferroelectric materials is becoming important in order to achieve high-density recording. On the other hand, Scanning Nonlinear Dielectric Microscopy (SNDM) has been proposed and developed by Cho *et.al.* [2] This microscopy can obtain the linear and nonlinear dielectric constant distribution (Especially the sign of the odd rank nonlinear dielectric constant tensor is correspond to the polarization direction.) of dielectric and ferroelectric materials. [3] Moreover, the formation of paraelectric surface layer on ferroelectric materials can be observed by varying the power order of nonlinearity and it is very useful for observing the state of polarization on surface of ferroelectric materials. [4] In this time, we present the new type of SNDM which can control the sample temperature in vacuo (less than 5×10^{-7} Torr). This temperature-controlled SNDM can cover the temperature range between 80K and 730K and obtain the linear and nonlinear dielectric constant distribution on ferroelectric materials caused by temperature change, especially neighborhood of the phase transition point or boiling point of water which adsorbs on surface. Using this SNDM, we have succeeded in measuring the linear and nonlinear dielectric constant distribution on $BaTiO_3$ single crystal due to the temperature change from 300K (room temperature) to 420K (above the Curie

temperature). Moreover, using higher order nonlinear dielectric microscopy, we also succeed in measuring the surface non-ferroelectric layer variation on $LiNbO_3$ and $LiTaO_3$ by temperature change. References [1] Y. Cho, K. Fujimoto, Y. Hiranaga, Y. Wagatsuma, A. Onoe, K. Terabe and K. Kitamura : Appl. Phys. Lett. 81 (2002) 4401. [2] Y. Cho, A. Kirihara and T. Saeki: Rev. Sci. Instrum. 67(1996) 2297. [3] H. Odagawa and Y. Cho: Surf. Sci. 463 (2000) L621. [4] Y. Cho and K. Ohara : Appl. Phys. Lett. 79 (2001) 3842.

3:30 PM GG4.4

Neutron Studies of Magnetic Recording Media. Stephen Lee[1], Colin Oates[1], Feodor Ogrin[2], Thomas Thomson[3], Charles Dewhurst[4], Robert Cubitt[4] and David Bowyer[4]; [1]School of Physics and Astronomy, University of St. Andrews, St. Andrews, Fife, United Kingdom; [2]Department of Physics, University of Exeter, Exeter, Devon, United Kingdom; [3]IBM, Almaden, California; [4]ILL, Grenoble, France.

There exists an enormous amount of research in the literature on both the bulk magnetic properties and the local dynamic response of magnetic recording media. In addition the physical grain structure and composition are also well-characterized by techniques such as TEM and XRD. There has, however, been relatively little work on the microscopic magnetic structure, especially regarding the magnetisation at a granular level. Small-angle neutron scattering (SANS) is an ideal tool for probing magnetic structure at a nanometre length scale, yet it is extremely difficult to perform experiments due to the tiny volume of material in the active magnetic layer. Furthermore, since the magnetic properties of thin film recording media depend critically on the sputtered multilayer structure and post-deposition processing, samples prepared especially for neutron studies may have properties which differ significantly from commercial materials. To overcome these drawbacks we have used some innovative approaches to SANS measurements to study the magnetic structure of materials intended for commercial application. Despite the inherent difficulties of the measurements it is nonetheless possible to extract a surprisingly large amount of information concerning the magnetic structure, including the size, shape and separation of the magnetic grains. One interesting result is that the extent of the magnetic core is significantly smaller than the physical grain in which it resides. We have also followed the evolution of the magnetic structure some way below saturation, which allows one to extract information on the orientation and anisotropic shape of the magnetic grains relative to the applied field.

3:45 PM GG4.5

Heteroepitaxy of InSe/GaSe on Si(111) Substrates.
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Due to its favorable structural and electrical properties, InSe is a candidate for application in a novel high-density storage medium [1]. In this paper we report on a structural study of InSe/GaSe heterostructures grown by molecular beam epitaxy on Si(111) substrates. Transmission electron microscopy indicates that these layers have good structural quality. Two-dimensional growth mode was achieved and layers have uniform thickness and flat surfaces. Two types of structural defects are present in these layers: stacking faults and dislocations. Selected area electron diffraction (SAED) shows that InSe preserves the orientation of the GaSe layer. A good quality of InSe/GaSe/Si(111) heterostructures seem to be consistent with the work of Budiman et al. [2] which shows that direct growth of InSe on GaAs(100) is very difficult, whereas an insertion of a GaSe layer leads to much better quality of InSe film. The GaSe intermediate layer seems to play a similar role in the case of Si(111) substrates, e.g. passivates the substrate dangling bonds [3]. Energy dispersive X-ray spectroscopy indicates a strong interdiffusion between Ga and In at the InSe/GaSe interface. SAED results suggest formation of the In_xGa_ySe phase. Similar interdiffusion has already been reported in the case of InSe/GaSe/GaAs(100) epitaxy [2]. [1] G.A. Gibson, et al, patent # 5,557,596. [2] Maman Budiman, Akira Yamada and Makoto Konagai , Jpn. J. Appl. Phys. 37, 4092 (1998). [3] Shuang Meng, B. R. Schroeder, and Marjorie A. Olmstead, Phys. Rev. B 61, 7215 (2000).