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ABSTRACT OF RESEARCH PROGRESS

We report continued progress in understanding the nanostructure-magnetic property relationships in sputtered, amorphous, thin films. This research has been concentrated on two categories of sputtered magnetic thin films; (1) magnetically soft films, of cobalt-based, binary (cobalt-metalloid) alloys with in plane anisotropies, and (2) magnetically hard, binary (rare earth metal-transition metal) alloy films with perpendicular anisotropies.

Major research results obtained include: (1) development of the first broadband measurement technique and apparatus for determining the complex permeability spectra of magnetic thin films in the 100KHz-200MHz frequency range; (2) processing of magnetically soft amorphous thin films with the highest values of RF permeabilities ever reported in the literature; (3) discovery of a new class of ultrasoft magnetic thin films with coercivities less than 0.1 Oe and completely reversible bias susceptibilities; (4) systemátically studied the microstructure, magnetic and magneto-optic properties of Tb-Fe and Gd-Co compositionally modulated films; (4) demonstrated that substrate bias could be used to increase the oxidation resistance and improve the magnetic and magneto-optical properties of sputtered Tb-Fe CMFs; and (5) developed a model of displacement eddy currents to resolve a long standing mystery about the origin of eddy current losses in magnetic recording head laminates.

FINAL TECHNICAL REPORT

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MAGNETIC PROPERTIES OF NANO-HETEROGENEOUS AMORPHOUS THIN FILMS

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I. OBJECTIVES OF THE RESEARCH

The overall goal of this research was to study the fabrication and properties of nano-heterogeneous, compositionally modulated, amorphous thin films with attractive properties for magnetic device applications. Devices of interest included: magnetic and magneto-optic recording media, thin film recording heads, and magnetic sensors.

The primary tasks of the program were to:

1. Study the effect of sputter deposition on: (a) film nanostructure as determined by TEM, and average composition, (b) temperature dependent magnetization and magnetic hysteresis, (c) temperature and frequency dependent permeabilities and permittivities, (d) film morphologies, and (e) film magnetization distributions.
2. Study the effect of post deposition annealing to optimize the film magnetic permeability and resistivity, and to minimize their anisotropy.
3. Study the correlation of process variables and film nanostructure, and their effect on magnetic and electronic properties.

II. STATUS OF THE RESEARCH EFFORT

A. OVERVIEW STATEMENT

The research in this program was focused on understanding the nanostructure-property relationships in magnetically soft, and magnetically hard, sputtered amorphous thin films and multilayers. For soft magnetic thin films the most important processing issues were those that influenced the development of an in-plane induced anisotropy. These issues are quite different from those that influence the development of the out-of-plane anisotropies of hard magnetic films of the type used in magnetic information storage technology. The research was concentrated on two categories of sputtered thin film systems: (1) soft magnetic thin films of Co-based alloys, and (2) compositionally modulated multilayer thin films of rare earth, and transition metals. The possibility of enhancing the magnetic response of films and multilayers in each category by understanding, and controlling their nanoscale features and morphologies was also studied.

An attempt was made to increase the initial permeability of films in the first category for application in integrated thin film recording heads and other sensors. Particular concern was given to identifying and controlling the origin of the subtle anisotropies induced during deposition in this type of film. Research on films in the second category was concerned with understanding similar issues as they affected the perpendicular anisotropy and influenced the magneto-optical response of amorphous thin films for application in magneto-optical recording.

B. SUMMARY OF SIGNIFICANT ACCOMPLISHMENTS

The following is a concise summary of the major research accomplishments of this program:

1. **Development of first broadband measurement technique and apparatus for determining the complex permeability spectra of magnetic thin films in the 100KHz-200MHz frequency range[2]**. The developed technique is theoretically capable of measuring the permeance of a 10Å permalloy film. It has subsequently been adopted by IBM, and in Japan as the standard technique qualifying thin films for use in magnetic recording heads and for comparing candidate magnetic thin films for a variety of applications.

2. **Demonstrated by TEM and related studies that the major source of the anisotropy of as-deposited amorphous soft magnetic thin films was due to growth anisotropies that resulted from self shadowing of obliquely incident atomic flux in the growing film[1,4,8]**. These experiments conclusively showed that this anisotropy could be virtually eliminated in films deposited from a normally incident sputtered flux.

2. **Produced the highest values of RF permeabilities ever reported in the literature[1,5,8,13]**. Extremely soft thin films of Co-B and Co-Fe-B with high initial permeabilities of 5,000-12,000 by annealing at elevated temperatures in rotating magnetic fields. The best binary Co-B films had permeabilities greater than 6000 with a 3 dB roll-off at 30 MHz, and the best ternary Co-Fe-B films had permeabilities of greater than 12,000 and a 3 dB roll-off at 100 MHz.

3. **A combination of TEM and AES was used to show, for the first time, the relationship between film nanoscale features and morphologies produced by the important film deposition, and post-**

deposition, parameters on the in-plane anisotropies of magnetically soft films[1,4,8,18,20,22]. These studies conclusively show that, in the as-deposited films, the major anisotropy is a dipolar anisotropy associated with the shape demagnetizing fields of the nanoscale features in the film morphology.

4. **Innovated the use of image processing techniques to quantify these results in terms of the statistical characteristics of the nanoscale morphology**[3,4,11].

5. **First demonstration of a greatly extended range of compositions in the Co-B and Co-Zr binary systems (relative to melt-quenched alloys) for which stable amorphous alloys could be retained in thin film form**[1,5,9,20,21]. Stable amorphous thin films with up to 40% B or Zr could be sputtered, compared to a maximum of about 25% B or Zr for melt-quenched ribbons. TEM revealed that sequentially sputtered films with an average composition of $\langle Co_{61}B_{39} \rangle$ or $\langle Co_{60}Zr_{40} \rangle$ were near the percolation limit and consisted of two, co-existing amorphous phases with morphological features of 1-10 nm. Surprisingly these films responded well to rotating field annealing and had attractive magnetic properties.

6. **Conducted the first extensive, systematic studies of the high frequency bias-susceptibilities of soft, nano-heterogeneous, amorphous thin films**[1,2,6,8,9,13,17,18,20].

7. **Discovered a class of ultrasoft thin films with coercivities less than 100 mOe and extremely large RF permeabilities**[5,6,13,18,20]. Nearly reversible transverse susceptibilities were produced by rotating field annealing. For small DC magnetic bias these films had very large nonlinear susceptilities, suggesting their possible application as magnetic sensors, and for low-field magnetometers.

8. **Demonstrated that, in spite of an apparent, anomalously large, magnetic loss, the dynamical response of the magnetization of soft amorphous thin films is consistent with the classical relaxation of a damped rotation**[22,23]. The damping constant of rotations in amorphous thin films is comparable to that of polycrystalline permalloy thin films.

9. **Studied the magnetization distribution in soft amorphous films by a combination of Bitter patterns, Kerr effect microscopy, and Lorentz microscopy**[1,3,8,9,10]. The results revealed that the films were nearly single domain. The thinnest films had a significant magnetization

ripple which was quantitatively analyzed, for the first time, by two dimensional, digital image processing[3]. The paper reporting this work was presented at the European Magnetics Materials and Applications Conference (EMMA) in Salford, UK in Sept. 1987, where it received the Best Poster Award.

10. **Conducted the first extensive, systematic comparisons of the microstructure, magnetic and magneto-optic properties of transition metal /rare earth alloy thin films, with compositionally modulated films of the same average composition**[7,12,14,16,19,]. This research was conducted on Gd-Co and Tb-Fe binary thin films and showed that significantly increased perpendicular anisotropies, and improved magnet-optic responses, could be developed in CMF films by optimally varying the composition and period of transition metal-rare earth metal bilayers.

11. **Demonstrated the effectiveness of substrate bias to increase the oxidation resistance and improve the magnetic and magneto-optical properties of Tb-Fe CMFs**[12,14,16].

12. Modeled the role of displacement eddy currents on the effective permeabilities of magnetic laminates of the type used in magnetic recording heads[24]. The results compared well with experimental results obtained on laminates with permalloy/silicon oxide film pairs and has **resolved a long standing mystery about the origin of anomalously eddy current losses in laminates containing films with thicknesses much smaller than a classical skin depth.**

C. SUMMARY OF RESEARCH PROGRESS

MAGNETICALLY SOFT, NANO-HETEROGENEOUS, AMORPHOUS THIN FILMS

Several types of Co-based, sputtered, amorphous, binary and ternary alloy thin films were investigated. The most interesting results were obtained with Co-B binary alloys and CoNbZr ternary alloys. The Co-B films were sequentially RF-diode sputtered from simultaneously excited elemental targets. The ternary alloy films were RF magnetron sputtered from alloy targets. Other studies were concentrated on nanoscale, compositionally modulated soft thin film systems, produced by RF magnetron sputtering alternating layers of CoNbZr alloys and silicon dioxide.

The major results of this work are summarized as follows:

This research[1,3,4,5] revealed that the major source of the anisotropy of as-deposited, sputtered, soft magnetic thin films was due to growth anisotropies that presumably have their origin in self shadowing of obliquely incident atomic flux. The formation of the resulting columnar growth structure is unavoidable because it is associated with the same kinetic conditions that favor the formation of the desired amorphous phase in which the magnetocrystalline anisotropy vanishes. Using a combination of TEM (transmission electron microscopy) and AES (Auger electron spectroscopy) it was determined that the source of the anistropy was likely due to the "internal shape anisotropy" associated with their nano-inhomogeneous morphologies. This anisotropy could be reduced to a few oersteds by simple thermal annealing.

Digital image processing was utilized in an attempt to determine the relationship between the anisotropies of these soft films and the "single particle" statistics (average size, eccentricity, orientation, etc.) of their nanoscale morphological features[4]. Surprisingly, this work revealed that systematic changes in the anisotropy of these films, produced by varying their deposition parameters, could not be correlated with changes in these statistics. These observations directly contradict the existing models and disagree with currently held beliefs.

These results suggested that the in-plane anisotropies of soft films could be the consequence of long range "morphological ordering" with a correlation length scale, too large to detect in electron diffraction, and too small to detect optically. We investigated this by digitally computing the Fourier transforms of optical images of the film TEM micrographs. The results were reported in Reference 11. This research investigated morphological order in magnetically soft, amorphous Co61B39 arising from long and short range interactions over distances of 0.2 - 1.0 nm. The results were qualitative but showed that the important changes in magnetic properties were associated with changes in the two-phase morphology. In general, decreases in the anisotropy were accompanied by increased isotropy in Fourier space. The 2D Fourier transforms of micrographs of films with the largest anisotropy reductions produced by rotating field annealing, were circularly symmetric. The results were interpreted to indicate that the reduced anisotropy results from annealing-out the preferred directions in the film plane that contained the minimum average separation between magnetic inhomogeneities.

In related research it was shown that the coercivities and anisotropies of Co-B binary alloy thin films could be reduced to values below 100 millioersteds by annealing in a rotating magnetic field[1]. The most interesting of these sputtered films were enriched by approximately 15 at % boron compared to melt quenched CoB alloys[5]. It was also established that these films had a two nanophase, amorphous morphology. Similar results were obtained for sputtered amorphous films of the binary alloy Co_xZr_y[21]. A restricted class of these soft amorphous films exhibited anomalous bias-frequency dependent susceptibilities and were designated as "ultra-soft"[6]. Ultra-soft thin films appear to have attractive properties for sensor applications. Accordingly, the Fourier transforms of their TEM micrographs were studied in an attempt to associate the onset of the ultrasoft film behavior with anneal-induced changes in film morphology. The results of this study suggest that ultrasoft properties are produced by a coarsening of the morphology associated with selective diffusion between nanoscale features in the dual nanophase structure. The difference between thermal annealing, and rotating field annealing may be due to the fact that the contribution of magnetic gradients to the diffusive driving force is anisotropic for the former, and anisotropic for the latter.

Additional studies of the bias-frequency dependent susceptibilities of the ultrasoft films were made to determine the origin of their apparently anomalous overdamped responses[13]. Recently, in this research it has been shown[23], however that these responses are not anomalously except for zero, or small bias DC magnetic bias. For biases comparable with the coercive field, the measured complex permeability spectra of soft amorphous thin films was found to agree well with that calculated from Kittel resonance theory[23].

NANO-HETEROGENEOUS, COMPOSITIONALLY MODULATED (CMF), MAGNETO-OPTICAL THIN FILMS

This research investigated the relationship of film nanostructure with the perpendicular anisotropy of sputtered thin films of transition metal (TM)-rare earth (RE) metal alloys. TM-RE alloy films are currently of great interest for their possible application as magneto-optical storage media. Their nanoheterogeneity is crucial for the development of the perpendicular magnetizations that enhance their Kerr effect magneto-optical response. It also controls their corrosive stability which is of great practical importance in this application. The effect of various sputter deposition parameters on the nanostructure, film composition, oxidation resistance, magnetization, coercivity, and magneto-optical Kerr response of these films was also investigated.

The primary concern was with the effect of sputter bias during the deposition of compositionally modulated thin films (CMFs) with modulation wavelengths of 1-15 nm. CMFs are artificially structures with ultrafine layers in which one attempts to achieve unique variations in the atomic scale pair ordering of the RE and TM species to achieve an optimum combination of desirable properties. Sputtering under a substrate bias significantly alters the amount of preferential re-sputtering of the deposited film, and alters the amount of included oxygen and sputtering gas (argon). All of these can have an important influence on the achievable properties.

The experimental research was conducted with CMFs of Gd-Co[7,14,17] and Tb-Fe[12,14,16,19] because these alloy systems hold great promise for practical storage applications. A detailed study of the effect of bias sputtering in these systems was conducted[7,12,16]. Cross section TEM, small angle X ray scattering, XPS, and AES were used to *determine sputtering effects on:* (1) the reduction of the oxygen content of these films, (2) the modified atomic clustering in the bilayers, and (3) the changes in the film nanostructure. Bias sputtered films had greatly reduced oxygen levels which, apparently, eliminated the columnar structure and improved the perpendicular anisotropy. Bias sputtered films also exhibited increased oxidation resistance and stable magneto-optical responses. The XPS data suggested that the magnetic properties of the unbiased films were dominated by the segregation of Fe atoms accompanying the oxidation of Tb near the surface.

The Tb based alloy films exhibited a larger coercivity and a larger perpendicular anisotropy than the Gd based alloy films. Accordingly an investigation of the microstructure and magnetic properties of Tb-Fe compositionally modulated films was investigated to determine their potential utility for magneto-optic recording[12,16]. Tb-Fe CMFs sputtered under -70V substrate bias had a dense, featureless structure, and a large perpendicular anisotropy. Compared to alloy films, the CMFs exhibited several anomalies; e.g. a compensation composition with increased Tb, higher Curie temperatures, and a shifted composition range for amorphization. All of these effects are consistent with the presence of atomic scale clustering in the thin layers.

The primary difference in the magnetic properties of Tb-Fe CMFs and alloy films appeared to be due to a strong atomic pair ordering associated with the anisotropic distribution of atomic pairs aligned within the layers of the CMF[19].

Compared to unbiased films, the application of substrate bias reduced the oxygen incorporated in the TbFe CMFs, eliminated the columnar void structure, and enhanced their perpendicular anisotropy and magneto-optic properties[12,16,17]. Unbiased films had a dominant in-plane anisotropy, and their magnetic and magneto-optic properties had a strong dependence on the bilayer period and film thickness. In these films Fe segregation accompanied the oxidation of Tb near the film surface. Multiple oxide regions were formed in amorphous TbFe CMFs during oxidation, and had a deleterious effect on their compositional, magnetic, and magneto-optic properties. This appeared to be a caused by the preferential oxidation and migration of Tb facilitated by the easy diffusion of oxygen through the columnar void structure of the unbiased films. These results demonstrated that the intrinsic stability of TbFe CMFs was strongly influenced by film microstructure and could be improved by the application of substrate bias during RF diode sputtering.

Although TbFe CMFs have many attractive magnetic and magneto-optic properties for application to magneto-optical recording, the research conducted on this program has shown that, without an appropriate surface passivation layer, they will be unstable due to the high affinity of Tb for oxygen.

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(2) C. Grimes, P.L. Trouilloud, and R. M. Walser, "A New Swept Frequency Thin Film Permeameter," IEEE Trans. Magn. MAG-24, 603 (1988).

(3) I. S. Jeong and R. M. Walser, "Quantitative Study of Magnetization Ripple in $Co_{61}B_{39}$ Thin Films," IEEE Trans. Magn. MAG-24, 1725 (1988).

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(9) D. Y. Kim and R. M. Walser, "Magnetic and Structural Properties of Sputtered Amorphous $Co_{100-x}B_x$ (15 at% < x < 39 at%)," In preparation.

(10) I. S. Jeong, "A Study of High Frequency Bias-Susceptibilities in Cobalt-Based Amorphous Magnetic Thin Films," Ph.D Dissertation, University of Texas at Austin, Fall 1988.

(11) A. P. Valanju, I. S. Jeong, and R. M. Walser, "Study of Multicomponent Magnetic Nanostructures with Digital Image Processing Technique," Mat. Res. Soc. Symp. Proc., 132, 173-178, (1989).

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(15) G. Choe and R. M. Walser, "Ion Beam Mixing of CoB Thin Films," Mater. Res. Soc. Symp. Proc. 157, (1989).

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(24) R. M. Walser and A. P. Valanju, "Displacement Eddy Currents in Magnetic Laminates," Accepted for Publication in J. Appl. Phys. (1992).

IV. PROFESSIONAL PERSONNEL ASSOCIATED WITH THE RESEARCH

1. Professor Rodger M. Walser - Principal Investigator
2. Ms. Alaka P. Valanju - Programmer/Analyst
3. Dr. Dae Yon Kim - Research Associate (Ph.D Degree awarded-May 1988).
4. Dr. In-Seop Jeong - Research Associate (Ph.D. Degree awarded-Dec. 1988)
5. Mr. Craig Grimes - Graduate Research Associate - (M.S. Awarded-1988).
6. Dr. Geon Choe - Research Associate - (Ph.D. Awarded- Fall 1989).
7. Dr. Ernest Louis - Research Associate - (Ph.D. Awarded-Spring 1991).
8. Dr. Young-Jin Jeon - Research Associate - (Ph.D. Awarded-Spring 1991).
9. Mr. Eui-Jung Yun - Graduate Research Associate - Ph.D. Student in Electrical and Computer Engineering.
10. Mr. Daryl Goodnight - Technical Staff Assistant V.
11. Ms. Elizabeth Savage - Technical Secretary.
12. Ms. Kay Shores - Administrative Associate.

Advanced Degrees Awarded:

1. Dr. Dae Yong Kim, Ph.D., May 1988, Thesis: A Study of the Effect of Process-Parameters on the Magnetic Anisotropy of Cobalt-Based Soft Amorphous Thin Films, Ph.D. Dissertation, University of Texas at Austin, Spring 1988.

2. Dr. In-Seop Jeong, Ph.D., December 1988, Thesis: A Study of High Frequency Bias-Susceptibilities in Cobalt-Based Amorphous Magnetic Thin Films," Ph.D Dissertation, University of Texas at Austin, Fall 1988.

3. Dr. G. Choe, Ph.D., December 1989, Thesis: "A Study of Structural Inhomogeneity and Magnetic Properties of Rare Earth-Transition Metal Compositionally Modulated Thin Films", Ph.D. Dissertation, University of Texas, Fall 1989.

4. Dr. E. Louis, Ph.D., May 1991, Thesis: "Magnetic Phase Transitions in Soft Amorphous Ferromagnetic Multilayer Thin Films", Ph.D. Dissertation, University of Texas, Spring 1991.

V. PAPERS PRESENTED AT CONFERENCES, MEETINGS, ETC.

1. I. S. Jeong and R. M. Walser, "Quantitative Study of Magnetization Ripple in $Co_{61}B_{39}$ Thin Films," European Magnetic Materials and Applications Conference, Salford, U.K., Sept. 1987.

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5. Geon Choe, M. F. Becker, and R. M. Walser, "Effect of Oxygen on Compositionally Modulated Gd-Co Thin Films," 4th Joint MMM-Intermag Conference, Vancouver, Canada, 1988.

6. A. P. Valanju, I. S. Jeong, and R. M. Walser, "Study of Multicomponent Magnetic Nanostructures with Digital Image Processing Technique," Materials Research Society Fall Meeting, Boston, Massachusetts, 1988.

7. R. M. Walser, "Model for Electronic Effects in Silicon Phase Epitaxy," Materials Research Society Fall Meeting, Boston, Massachusetts, 1988.

8. G. Choe and R. M. Walser, "Effect of Bias Sputtering on Magnetic Properties and Structure of Compositionally Modulated $Tb_{32}Fe_{68}$ Films," Intermag '89 Conference, Washington, D.C., 1989.

9. I. S. Jeong and R. M. Walser, "Magnetization Distribution in Nearly Isotropic Ultra-Soft Magnetic CoB Amorphous Thin Films," Intermag '89 Conference, Washington, D.C., 1989.

10. G. Choe and R. M. Walser, "Effect of Bias Sputtering on Magnetic Properties and Structure of Compositionally Modulated $Tb_{32}Fe_{68}$ Films," 1989 Intermag Conference on Magnetic Materials, Washington, D.C., March 28-31, 1989.

11. G. Choe and R. M. Walser, "Ion Beam Mixing of CoB Thin Films," Symposium-Materials Research Society, Boston MA, Nov. 27-Dec. 2, 1989.

12. G. Choe and R. M. Walser, "Effect of Bias Sputtering on Stability of Amorphous $Tb_{32}Fe_{68}$ Compositionally Modulated Thin Films," 34th Annual Conference on Magnetism and Magnetic Materials, Boston MA, Nov. 28-Dec. 1, 1989.

13. G. Choe, A.P. Valanju, and R. M. Walser, "Structural Inhomogeneity and Magnetic Properties of Amorphous $Gd_{17}Co_{83}$ Compositionally Modulated Thin Films," 34th Annual Conference on Magnetism and Magnetic Materials, Boston MA, Nov. 28-Dec. 1, 1989.

14. E. Louis, I. S. Jeong, and R. M. Walser, "Bias and Frequency Response of the Permeability of $CoZrNb/SiO_2$ Multilayers," 34th Annual Conference on Magnetism and Magnetic Materials, Boston MA, Nov. 28-Dec. 1, 1989.

15. G. Choe and R. M. Walser, "Structure and Magnetic Properties of Compositionally Modulated TbFe Film," 34th Annual Conference on Magnetism and Magnetic Materials, Boston MA, Nov. 28-Dec. 1, 1989.

16. I. S. Jeong, E. Louis, and R. M. Walser, "Effect of Compositional and Structural Heterogeneities on the Magnetic Properties of Amorphous CoZrNb Thin Films," 34th Annual Conference on Magnetism and Magnetic Materials, Boston MA, Nov. 28-Dec. 1, 1989.

17. A.P. Valanju, D.Y. Kim, and R. M. Walser, "Soft Magnetic Characteristics of Co/Zr Thin Film Layered Structures," 35th Annual Conference on Magnetism and Magnetic Materials, San Diego, CA, Oct. 29- Nov. 1, 1990.

18. E. Louis and R. M. Walser, "Comparison of Experimental and Theoretical Permeability Spectra of CoZrNb Amorphous Thin Magnetic Films," Second International. Symposium on Magnetic Materials, Phoenix, AZ, Oct. 14-17, 1991.

19. R. M. Walser and A. P. Valanju, "Displacement Eddy Currents in Magnetic Laminates," To be presented at Intermag Conference, St. Louis, MO, April 13-16, 1992.