DOE/ER/45297-24

Technical Progress Report

ARTIFICIALLY STRUCTURED MAGNETIC MATERIALS
DOE Grant Number DE-FG02-87ER45297
April 1, 1990 - September 30, 1990
(submitted September 28, 1990)

DOE/ER/45297--24
DE91 000600

Attention: Dr. Claude Herrick

Charles M. Falco, Principal Investigator
Department of Physics; Optical Sciences Center
PAS Building 81
University of Arizona
Tucson, Arizona 85704
(602) 621-6771
(602) 621-4356 FAX

I. INTRODUCTION

This document reports the progress made during the first six months of the current three-year DOE grant on "Artificially Structured Magnetic Materials." However, because some of the results of our previous three-year DOE grant on "Artificially Structured Superconductors" continue to emerge, both topics are addressed in this Progress Report.

Following this Introduction, the remaining sections of this report describe progress with DOE funding during the current calendar year; description of the research to be conducted during the remaining six months of the current grant year; a description of the status of the graduate students working on this research; lists of the invited talks, seminars and colloquia, of other recognition of our research, and of the publications crediting DOE sponsorship; and a summary of current and pending federal support. Since the research proposed to be conducted during the next 2½ years is described in detail in our DOE proposal, it is only briefly reviewed here.

Because of the specific capabilities given by significant enhancements to our thin film preparation and analysis facilities (e.g. two heavily instrumented Molecular Beam Epitaxy machines, recent acquisition of an atomic-resolution Transmission Electron Microscope, etc.), our research efforts have been increasingly directed toward studies of magnetism at surfaces and interfaces. Thus, our DOE-funded research program for the next three years involves studies of such magnetic thin films and superlattices.

Excellent progress and recognition of our DOE-funded research continues to be achieved, as evidenced by the 23 publications, 9 invited talks, and 6 contributed abstracts thus far in 1990 which credit DOE support, and appointment of the PI to several editorial boards and superconductivity panels as detailed below.

II. RESEARCH ACCOMPLISHMENTS: JANUARY - OCTOBER, 1990

A. Introduction

Substantial progress was made on our studies of the deposition, characterization and physical properties of metallic multilayers and superlattices during 1990 with DOE funding. This work resulted in 23 publications, 9 invited talks, and 6 contributed abstracts written/published thus far since January 1990 which credit DOE support. Some of these results are briefly described in the sub-Sections below.

B. Fabrication of Multilayers by Sputtering and MBE

Approximately 2½ years ago we obtained our first MBE system, which contains Reflected High and Low Energy Electron Diffraction (RHEED and LEED), X-ray Photoelectron Spectroscopy (XPS), ion mill Auger Electron Spectroscopy (AES), and Ion Scattering Spectroscopy (ISS). This machine is devoted to the fabrication and study of ultra-thin metallic films and superlattices. A large number of samples (>100) already have been made with this MBE system; both ultra-thin films and multilayers. During the current year we added an "experimentation" chamber of our own design to this machine. This new chamber contains Surface Magneto-Optic Kerr Effect (SMOKE) instrumentation for *in situ* studies at $<5x10_{-11}$ torr of the magnetic properties of our MBE-grown magnetic films. We also added a four-hearth electron beam gun this year which, along with our existing two single-hearth guns and two Knudsen cells, now allows us to grow 28 different two-at-a-time combinations of materials without having to break vacuum.

A year ago we obtained a second, Riber 1000, MBE machine, donated by Hughes' Malibu Research Laboratories. We refurbished and reconfigured this machine over the past year with funds from the university, and it is now functional. This machine contains scanning Auger and Secondary Ion Mass Spectroscopy (SIMS) for studies of the films grown in it, as well as of samples grown in our other MBE and sputtering machines.

During the past year we also added additional controls and instruments to our two computer-controlled, multi-target sputtering systems. Thus, with our two MBE machines and two computer-controlled sputtering machines, we are extremely well equipped to grow and study ultra-thin films and multilayers of magnetic materials.

C. Sample Characterization Techniques

C-1. Introduction

Very recently (August 1990) the AFOSR transferred to my lab a two-year-old JEOL 2000FX atomic-resolution Transmission Electron Microscope (TEM). At the same time they also transferred a high quality JEOL 5200 Scanning Electron Microscopy (SEM). Both of these microscopes are scheduled for installation in my lab by JEOL technicians the last week in September. These electron microscopes, of current value approximately $0.5M, clearly are significant additions to our analytical capabilities. With them we intend to make atomic resolution, cross-sectional studies of the composition and structure of our multilayers.

One of the most serious problems in research involving artificially structured magnetic materials (either superlattices or ultra-thin films) is adequate characterization of the samples produced. For example, information is required with better than a few Ångstroms resolution about the sharpness, chemical purity, and electronic properties of the interfaces. Given the importance of this problem, it is necessary to have techniques for determining details of the composition profile at the interfaces (i.e. distance of interdiffusion, roughness, presence of oxides or intermetallic compounds, etc.). Standard surface analysis techniques such as Auger or Ion Scattering Spectroscopy have sufficient, or nearly sufficient, depth resolution when applied to composition analysis of surfaces. However, the problem of characterizing multilayers is intrinsically different, since we require information on the composition profile of interfaces located within the sample, rather than on the surface. If ion milling is used to expose these interfaces, the lattice is disrupted and the interfaces are smeared out. For this reason we have been investigating alternative ways of obtaining composition information near interfaces.

In the remainder of this Section we describe, in order, recent results obtained with x-ray diffraction, Scanning Tunneling Microscopy (STM), and Rutherford backscattering (RBS).

C-2. X-Ray Diffraction

Recently we completed construction of our own low-angle diffractometer, which now enables us to characterize all of our samples using this technique, rather than a selected few. Software for computer-control of this instrument has been modified and upgraded this year. Because of the power of this technique, this diffractometer has been kept very busy the past year characterizing our multilayer samples. However, because it uses a sealed-tube x-ray source, it typically takes at least 12 hours to obtain a complete spectrum. A very useful future upgrade would be to obtain a rotating anode x-ray source. This would reduce data acquisition time to a much more satisfactory hour or so.

C-3. Scanning Tunneling Microscopy (STM)

Considerable research using our STM has been conducted this year. We used our STM studies to refine our MBE growth parameters. This enabled us to find the proper conditions to grow epitaxial, single-crystal Co/Pd superlattices whose top surface is atomically flat over regions as large as 500 Å. The magnetic properties of these materials are extremely interesting, and initial results already have been reported in some of the publications listed below in Section V.

C-4. Auger and X-Ray Photoelectron Spectroscopy (XPS)

As well as detecting possible sample contamination, Auger and X-ray Photoelectron Spectroscopy (XPS) can be extremely useful for determining the "growth mode" of metals, i.e. whether they form by continuous layer-by-layer growth (Frank-van der Merwe), three-dimensional islands (Volmer-Weber), layer-plus-island (Stranski-Krastanov), or some more complicated mode. Using our MBE machine, we are in the process of making a series of studies of the growth mode of Co on several orientations of single-crystal GaAs at several substrate temperatures. In the near future the results of these growth mode studies will be correlated with the magnetic properties of these films.

C-5. Rutherford BackScattering (RBS)

Rutherford Backscattering Spectroscopy (RBS) as an analysis tool for these materials has proven to be very useful, even though RBS cannot provide direct information with a vertical resolution less than ≈100 Å. This year we succeeded in growing Co/Pd superlattice films of sufficient quality that they exhibited "channeling" of the RBS beam hundreds of Ångstroms into the MBE-grown films. To the best of our knowledge, these are the first metallic superlattices ever to exhibit such channeling. These results are presently being prepared for publication.

D. Brillouin Light Scattering

Inelastic light scattering with a tandem, multi-pass Fabry-Perot interferometer makes it possible to measure phonon and magnon dispersion curves for thin films. This year we used this technique to detect new exchange-coupled magnetic modes in sputtered Co/Pd multilayers. These measurements were done in collaboration with Professor Gernot Güntherodt's group at the University of Aachen, Germany, and have been submitted to the Physical Review. Based on these results, we are presently growing a series of Co/Pd samples by MBE to explore in detail some of the interesting features we detected. We also studied some of the magnetic properties of our Co/Cu materials by polarized neutron scattering. These measurements were made in collaboration with a group at the Rutherford Laboratory in England, and have been submitted to the Physical Review.

E. Mössbauer Spectroscopy

Mössbauer spectroscopy provides a microscopic probe of the local magnetic environment. Several years ago we applied this technique to Fe/Pd and Fe/W superlattices to distinguish between "interfacial" Fe (that which is in contact with, or has partially diffused into, the other component of the superlattice) and "bulk" Fe (surrounded only by other Fe atoms). Although our recent concentration of Co-based magnetic materials means that no Mössbauer studies were conducted this year, this technique may be used again in the future when applicable.

F. Superconductivity Studies

Although our current DOE research is on magnetic ultra-thin films and superlattices, some of our previous DOE contract's work on superconducting materials continues to emerge. This will be the case for the next year, as Valentín García-Vázquez finishes his Ph.D studies. The six manuscripts published/submitted this year reported results on superconductivity are included in the list in Section V below. This work included a Physical Review Rapid Communication reporting results on zinc substitution on the electron superconductor $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$. We also were invited to prepare a review article on deposition techniques used to prepare high T_c thin films. This was published in Modern Physics Letters. Work on tunneling studies to low T_c superconductors also was reported this year.

G. Summary

To summarize this Section on research accomplishments during the current year of DOE funding, considerable progress was made in all three major areas of our program: deposition, characterization, and measurement of physical properties of multilayered materials. Although our results were only briefly described above, this research is described in detail in the 23 publications crediting DOE support which are listed below in Section V.

III. Research to be Conducted Through March 31, 1991

Emphasis during the next six months will continue to be on producing and characterizing high-quality, single-crystal MBE-grown materials for studies of the magnetic properties. Because of the interest our recent results have generated, we will continue to concentrate on Co-based materials during this period. Our collaborations with other groups to study some of the magnetic properties by light scattering and neutron diffraction will continue. Our first *in situ* Surface Magneto-Optic Kerr Effect (SMOKE) measurements should be completed by the end of this six-month period. We also will study the magnetic properties of these materials using our Vibrating Sample Magnetometer (VSM).

IV. Status of Graduate Students Working on this Research

Two students are funded by this DOE grant. The first, Valentín García-Vázquez, started work several years ago on high T_c superconductors under our previous DOE grant, and should receive his Ph.D. degree within a year. The second, Michael Wiedmann, started work in June on magnetism. Since Michael is a first year graduate student, it will be at least four years before he receives his Ph.D. A third student, Chandrakant Barlingay, received a small amount of funding last year, and completed his M.S. thesis in Materials Science this past January. Chandrakant worked with us on high Tc superconductivity, and his research resulted in several publications listed below in Section V.

In addition to the above graduate students, several visiting scientists have been associated with this research. Dr. Luigi Maritato, who recently moved to the Department of Physics at the University of Salerno, Italy from the INFN laboratory in Frascati spent time working on high T_c films in my laboratory this fall, paid by his institution. Several publications crediting DOE sponsorship have resulted from this collaboration. Mr. Masafumi Nakada, a staff scientist at NEC's Central Research Laboratory, returned to Japan in August, after spending a year working in my laboratory on magnetism. He was funded by his company. Another visitor, Prof. Rajka Djajić-Jovanović from the University of Novi Sad, Yugoslavia spent two months a year ago working on superconductivity, paid by the DOE under one of their international programs.

V. PUBLICATIONS, INVITED TALKS, ABSTRACTS, AND SEMINARS CREDITING DOE SUPPORT DURING THE PERIOD JANUARY 1990 - PRESENT)

A. Publications

1. **Zinc Substitution Effects on the Electron Superconductor $Nd_{1.85}Ce_{0.15}CuO_{4-\delta}$**
C. Barlingay, V. García-Vázquez, Charles M. Falco, S. Mazumdar, and S.H. Risbud
Phys. Rev. B - Rapid Communications B 41, 4797 (1990)

2. **Deposition Techniques for High T_c Thin Films**
L. Maritato and Charles M. Falco
Modern Physics Letters B 4, 639 (1990)

3. **dV/dI Double Peak Structures in Superlattice-Based Tunnel Junctions**
L. Maritato, A.M. Cucolo, R. Vaglio, C. Noce, J.L. Makous, and C.M. Falco
in, Science and Technology of Thin Film Superconductors, Robert D. McConnell and Stuart A. Wolf, eds. (Plenum, New York, 1989), p. 495

4. **Superconducting Superlattice Structures**
J. L. Makous, J. A. Leavitt, L. C. McIntyre Jr., L. Maritato, R. Vaglio, A.M. Cucolo, and Charles M. Falco
Proc. of the Materials Research Society - IN PRESS

5. **A Beamline for Layered Synthetic Microstructure Studies**
J. Boudry, C. Riedel, B. Edwards, M. Lagally, R. Redaelli, F. Cerrina, Charles M. Falco, F. Fernandez, J.H. Underwood and M. Hettrick
Nucl. Instr. Methods - IN PRESS

6. **Melt Processing of Bi-Ca-Sr-Cu-O Superconductors**
E.D. Zanotto, J.P. Cronin, B. Dutta, B. Samuels, S. Subramoney, G.L. Smith, G. Dale, T.J. Gudgel, G. Rajendran, E.V. Uhlmann, M. Denesuk, B.D. Fabes, D.R. Uhlmann, V. Garcia, J. Makous, and C.M. Falco
J. Amer. Ceramics Soc. - IN PRESS

7. **Optical Properties of Artificially Structured Metals**
Charles M. Falco, Jon Slaughter, and Brad Engel
J. Noncrystalline Solids - IN PRESS

8. **$YBa_2Cu_3O_{7-x}$ Thin Films Prepared by Multilayer Sputtering**
Valentin García-Vázquez and Charles M. Falco
Proc. of the Materials Research Society - IN PRESS

9. **Structural and Magnetic Properties of Ti/Co Multilayers**
Robert Van Leeuwen, Craig D. England, John R. Dutcher, Charles M. Falco, and Wayne R. Bennett.
J. Appl. Phys. - IN PRESS

10. **Magnetic and Magneto-Optical Properties of Interfaces and Superlattices**
Brad N. Engel and Charles M. Falco
Bull. of the Materials Research Society - IN PRESS

11. **Evidence for Collective Exchange Modes in Co/Pd Multilayers Observed by Brillouin Light Scattering**
B. Hillebrands, J.V. Harzer, R.L. Stamps, G. Güntherodt, C.D. England, and Charles M. Falco
Proc. of the Materials Research Society - IN PRESS

12. **X-ray Characterization of Magnetic Multilayers and Superlattices**
Charles M. Falco, J.M. Slaughter and Brad N. Engel
in, Nanostructured Magnetic Materials, G. Hadjipanyis and G. Prinz, eds. (Plenum, New York, IN PRESS)

13. **Structural and Magnetic Properties of Epitaxial Co/Pd Superlattices**
Brad N. Engel, Craig D. England, Masafumi Nakada, Robert Van Leeuwen, and Charles M. Falco
in, Nanostructured Magnetic Materials, G. Hadjipanyis and G. Prinz, eds. (Plenum, New York, IN PRESS)

14. **MBE Growth of Metal/Semiconductor Interfaces**
J.M. Slaughter, Brad N. Engel, M.H. Wiedmann, Patrick A. Kearney, and Charles M. Falco
in, Nanostructured Magnetic Materials, G. Hadjipanyis and G. Prinz, eds. (Plenum, New York, IN PRESS)

15. **Magnetic Properties of Metallic Multilayers and Superlattices**
Charles M. Falco and Brad N. Engel
Physica B - IN PRESS

16. **Structural, Electrical and Mössbauer Studies of Fe/W Metallic Superlattices**
A. Boufelfel, Roy M. Emrick, and Charles M. Falco
Phys. Rev. B - SUBMITTED

17. **Magnetism of Fe/Pd Superlattices**
Ahmed Boufelfel, Roy M. Emrick and Charles M. Falco
Phys. Rev. B - SUBMITTED

18. **Magnetic Properties of Pd/Co Multilayers**
W.R. Bennett, C.D. England, D.C. Person, and Charles M. Falco
J. Appl. Phys. - SUBMITTED

19. **Experimental Evidence for the Existence of Exchange-Dominated Collective Spin-Wave Excitations in Multilayers**
B. Hillebrands, J.V. Harzer, G. Güntherodt, C.D. England and Charles M. Falco
Phys. Rev. B - Rapid Communications - SUBMITTED

20. **Magnetic Properties of Co/Pd Multilayers Determined by Brillouin Light Scattering and SQUID Magnetometry**
J. V. Harzer, B. Hillebrands, R.L. Stamps, G. Güntherodt, C.D. England and Charles M. Falco
J. Appl. Phys. - SUBMITTED

21. **Polarized Neutron Reflection to Characterize Cobalt-Copper Multilayers**
W. Schwarzacher, W. Allison, J. Penfold, C. Shackleton, C.D. England, W.R. Bennett, J.R. Dutcher, and C.M. Falco
Phys. Rev. B - SUBMITTED

22. **Magnetic Properties of Epitaxial Co/Pd Superlattices**
Brad N. Engel, Craid D. England, Robert Van Leeuwen, Masafumi Nakada, and Charles M. Falco
J. Appl. Phys. - SUBMITTED

23. **Preparation and Structural Characterization of Epitaxial Co/Pd (111) Superlattices**
Craig D. England, Brad N. Engel, and Charles M. Falco
J. Appl. Phys. - SUBMITTED

B. Invited Talks Crediting DOE Support (January 1990-present)

1. **MBE-Growth of Magneto-Optical Materials**
2nd Storage Research Centers Faculty Meeting. IBM San Jose. January 11-12, 1990
Charles M. Falco, Brad Engel, Craig England and Robert Van Leeuwen

2. **High T_c Superconductivity for Microelectronics**
International Society for Hybrid Microelectronics. Phoenix, Arizona. May 10, 1990
Charles M. Falco

3. **Magneto-Optical Properties of MBE-Deposited Superlattices**
3rd Workshop on Magneto-Optical Recording Technology. Carnegie-Mellon University, Pittspurgh. May 15-17, 1990
Charles M. Falco, Brad Engel, Craig England and Robert Van Leeuwen

4. **X-Ray Characterization of Multilayers and Superlattices**
NATO Advanced Study Institute on the Science and Technology of Nanostructured Magnetic Materials. Aghia Pelaghia (Crete), Greece. June 25 - July 7, 1990
Charles M. Falco, J.M. Slaughter and Brad N. Engel

5. **Magnetic Properties of Metallic Superlattices**
19th International Conference on Low Temperature Physics. Brighton, England. August 16-22, 1990
Charles M. Falco and Brad N. Engel

6. **Structural and Magnetic Ordering at Interfaces of Metallic Superlattices**
The 3rd NEC Symposium on Fundamental Approaches to New Material Phases—Ordering at Surfaces and Interfaces. Hakone, Japan. October 7-11, 1990
Charles M. Falco and Brad N. Engel

7. **Physical Properties of MBE-Grown Metallic Superlattices**
Symposium on Synthesis, Characterization and Properties of Microlayered Alloys, at the 178th meeting of the Electrochemical Society. Seattle, Washington. October 14-19, 1990
Charles M. Falco

8. **Studies of High T_c Thin Films**
Workshop on Manifestations of the Electron-Phonon Interaction in High Temperature Superconductors. Oxtepec, Mexico. December 11 - 14, 1990
Charles M. Falco and Valentín García-Vázquez

9. **Effect of the Interfacial Structure on Elastic, Magnetic, and X-Ray Optical Properties of Metallic Superlattices**
Symposium on Structure-Property Relationships for Metal-Metal Interfaces. Materials Research Society. Anaheim. April 29 - May 3, 1991
Charles M. Falco

C. **Abstracts of Contributed Talks Crediting DOE Support (Jan. 1990-present)**

1. **Effect of Zn and Ni Substitution on the Superconducting Properties of $Nd_{1.85}Ce_{0.15}CuO$**
C.K. Barlingay, V. García-Vázquez, Charles M. Falco, S. Mazumdar, and S.H. Risbud
American Physical Society March 1990 Meeting, Anaheim. Mar. 12-16, 1990

2. **Is There a Dopant-Induced Structural Transition in Nd(Ce)CuO?**
V. García-Vázquez, C.K. Barlingay, S.H. Risbud, S. Mazumdar, and Charles M. Falco
American Physical Society March 1990 Meeting, Anaheim. Mar. 12-16, 1990

3. **Structural and Magnetic Properties of Epitaxial Co/Pd Superlattices**
Brad N. Engel, Craig D. England, Robert Van Leeuwen, and Charles M. Falco, Masafumi Nakada
American Physical Society March 1990 Meeting, Anaheim. Mar. 12-16, 1990

4. **Structural and Magnetic Properties of Epitaxial Co/Pd Superlattices**
Brad N. Engel, Craig D. England, Robert Van Leeuwen, and Charles M. Falco and Masafumi Nakada
NATO Advanced Study Institute on the Science and Technology of Nanostructured Magnetic Materials. Crete, Greece. June 25-July 7, 1990

5. **Magnetic Properties of Epitaxial Co/Pd Superlattices**
Brad N. Engel, Craig D. England, Robert Van Leeuwen, Masafumi Nakada, and Charles M. Falco
Conference on Magnetism and Magnetic Materials, San Diego. Oct. 29 - Nov. 2, 1990

6. **Preparation and Structural Characterization of Epitaxial Co/Pd (111) Superlattices**
Craig D. England, Brad N. Engel, and Charles M. Falco
Conference on Magnetism and Magnetic Materials, San Diego. Oct. 29 - Nov. 2, 1990

D. Seminars and Colloquia Resulting from our DOE-Funded Research (1987-present)

1. **Artificial Metallic Superlattices**
Sandia National Laboratory, Albuquerque
March 23, 1990

2. **Artificial Metallic Superlattices**
Physics Department, University of Cincinnati
June 7, 1990

E. Other Recognition of Our DOE-Sponsored Superconductivity Research

Member of the Institute for Defense Analysis' (IDA) Technical Working Group on Superconductivity (TWG-5C). May 1988 - present.

Member, Board of Editors for **Rapid Communications in High Temperature Superconductivity - International Journal of Modern Physics B** and **Modern Physics Letters.** June 1988 - present.

Alexander von Humboldt Foundation Senior Distinguished U.S. Scientist Award, 1989-1990

Co-Organizer of the Materials Physics Topical Group session on "Surface Magnetism" at the March American Physical Society Meeting. Cincinnati, OH. March 18-22, 1991.

VI. CURRENT AND PENDING SUPPORT

Name of Investigator: Charles M. Falco

Source of Support	Project Title	Award	Period Covered	Person-Mo. or % Effort	Location of Research
A.	**Current Support**				
DOE	Artificially Structured Magnetic Materials	$351,868	4/90 - 3/93	6 mo.	U of A
AFOSR	Laboratory for X-Ray Optics	$702,000	10/90 - 9/93	10%	U of A
ONR	Elastic Properties of Metallic Superlattices (Co-PI, G.I. Stegeman)	$276,933	9/90 - 9/92	—	U of A
ODSC	Magneto-Optic Properties of Metallic Multilayers	$ 98,000	7/90 - 6/91	—	U of A
JSOP	X-Ray Mirror Research	$ 70,000	10/90 - 9/91	—	U of A
B.	**Pending Support**				
NSF	U.S./Japan Cooperative Research on Materials	$ 20,158	7/91 - 6/93	—	U of A
NSF	U.S./Latin America Cooperative Research: Symposium on Superconductivity (Mexico)	$ 10,900	10/90 - 1/91	—	U of A

END

DATE FILMED

11 / 07 / 90