

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

March 10, 1965

George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Huntsville, Alabama 35812

Attention: PR-EC

Subject: Monthly Progress Report No. 1
Contract NAS8-20011
Project No. A-837

Dear Sir:

The research program to be carried out under Contract NAS8-20011 "Investigation of Thin Magnetic Films for Memory Applications" has been outlined and task assignments have been made in four distinct phases identified below. Work in the four phases will be conducted simultaneously.

Phase I. This phase includes the preparation and evaluation of a number of 80-20 permalloy films formed in high vacuum ($\sim 10^{-6}$ Torr) by evaporation of the alloy from a quartz crucible by RF-induction heating techniques. The films will be evaluated for their magnetic properties by the hysteresis loop tracer and the Kerr effect apparatus and for thickness and composition by x-ray fluorescence or electron microprobe techniques. The morphology of the films will be studied by electron microscopy. Evidence for domain wall creep will be examined and alterations in the evaporation technique will be made in an effort to inhibit the creep in the films.

Phase II. Studies of domains in thin permalloy films will be made by Lorentz microscopy. Two approaches are planned. A special high resolution holder for the objective position of the specimen in the EMU-3 electron microscope is being designed. In addition a Lorentz attachment similar to that of Cohen (Proceedings of Fifth International Congress for Electron Microscopy, Vol. I, Academic Press, New York (1962) is being built for the intermediate lens position of the same microscope.

Phase III. This phase is devoted to evaporation studies of the deposition of permalloy through a fine slit onto a rotating substrate. Analysis of the material on the substrates by x-ray fluorescence or microprobe techniques is expected to give a time distribution of the material deposited on the substrate in the normal process.

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March 10, 1965

Phase IV. This phase includes the literature search in direct support of the above technical phases and will provide the material for an appropriate state-of-the-art survey on magnetic films for computer memories. Various sources of information are being utilized including RSIC.

The initial effort during the past month has been devoted to refinements in the magnetic test apparatus and to the detailed design of other equipment required for the work. All of the phases are currently proceeding as planned and no delays in the program are anticipated.

No trips or visits were made under the contract during the last month.

Respectfully submitted,

John A. Copeland
Project Director

JAC:brj

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

April 12, 1965

George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Huntsville, Alabama

Attention: PR-EC

Subject: Monthly Progress Report No. 2
Contract NAS8-20011
Project No. A-837

Dear Sir:

Phase I. During the past month project efforts have been mainly devoted to Phase I described in the last monthly progress report. A jig and evaporator for deposition of the permalloy films have been designed by M. D. Carithers and are presently being built. C. K. Kuo has added the coils and pulse drivers to our Kerr apparatus which will be used for domain-wall motion studies.

Phase II. J. L. Brown is working on the design of the two Lorentz attachments for the EMU-3 electron microscope, a high resolution holder to go in the normal specimen position of the microscope and a Cohen-type attachment which is placed in the intermediate lens position. Some parts for the high resolution holder are already being machined.

Phase III. The rotating substrate experiment by J. O. Darnell is scheduled during the preparation of the films for Phase I so that analysis of the deposition rates can be made under the conditions of preparation of the permalloy films.

Phase IV. The literature search under the supervision of R. L. Bullock and B. L. Johnson is now operating on a "current" basis. (The first phase covered a review of supporting documents from 1957 to 1964.) Documents ordered from NASA and DDC are arriving regularly and are being entered into the system. In addition to supplying the input for the ultimate state-of-the-art survey, the descriptor-based system is available for interrogation by project team members and NASA's technical representatives.

April 12, 1965

Phase V. This phase which was added to the project during the last month will involve feasibility studies of the preparation of permalloy films by chemical vapor deposition and subsequent study of their magnetic properties. Dr. J. D. Fleming of the Georgia Tech School of Chemical Engineering is directing this phase of the work. The necessary apparatus for depositing film on our normal substrates is being set up now and the first films are expected during the next month.

Mr. G. A. Bailey of the Astrionics Lab of MSFC visited us on April 8 and 9 to discuss the progress on the program. No other trips or visits were made under the contract during the last month.

Respectfully submitted,

John A. Copeland
Project Director

JAC:brj

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

May 13, 1965

George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Huntsville, Alabama

Attention: PR-EC

Subject: Monthly Progress Report No. 3
Contract NAS8-20011
Project No. A-837

Dear Sir:

Phase I. The jig and evaporator for deposition of permalloy films designed by M. D. Carithers has been completed. Photographs of the jig and substrate cleaning racks are enclosed. The final plans for this jig will be made available to MSFC as soon as possible. Production and evaluation should begin during the next monthly period.

Phase II. J. L. Brown has completed the high resolution Lorentz holder which goes in the normal specimen position of the EMU-3 electron microscope. Work on the Cohen-type attachment is continuing.

Phase III. The rotating substrate experiment by J. O. Darnell is scheduled during the preparation of films for Phase I so that analysis of the deposition rates can be made under the conditions of preparation of the permalloy films.

Phase IV. Four specific requests from project personnel were processed by the information system during this period. The request on "Lorentz Microscopy" provided documents that were quite helpful to J. L. Brown in constructing the Lorentz holder reported in Phase II. In addition to answering specific requests, the system will provide source documents to be analyzed and evaluated in preparing the state-of-the-art survey on problems associated with "Domain-wall Creep." As soon as the detailed subject of the survey is determined, pertinent documents will be retrieved from the system for survey.

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May 13, 1965

A preliminary examination of documents available or abstracted in International Aerospace Abstracts indicates that some information can be obtained on memory systems in computers for satellite applications but that a direct request for details through the appropriate project officers at Goddard may be desirable.

Phase V. The furnace for deposition of iron, nickel, and permalloy film by chemical vapor deposition has been completed. Dr. J. D. Fleming reports that preparation of the first films by this technique is eminent.

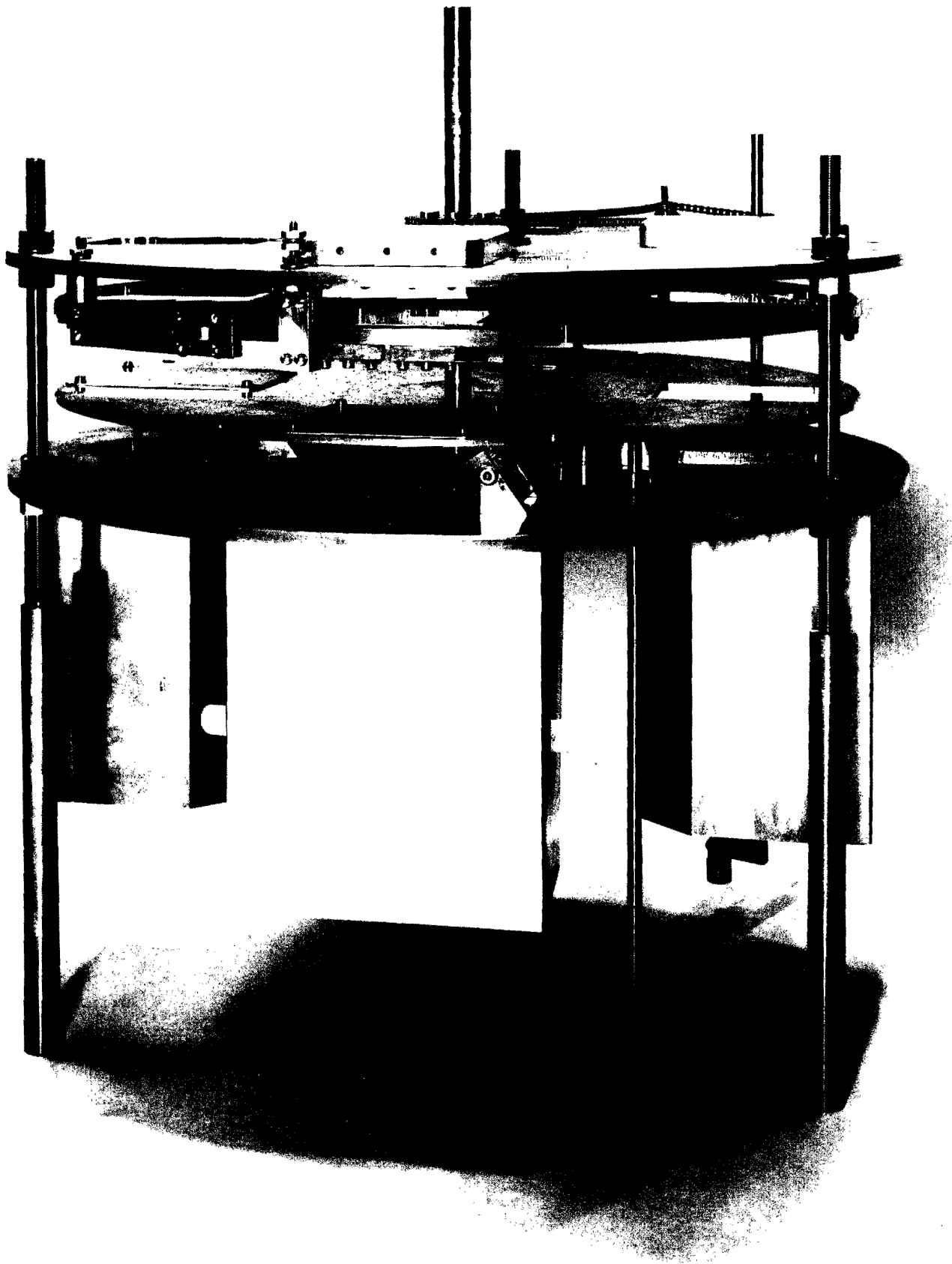
Dr. E. J. Scheibner and J. A. Copeland attended the International Conference on Magnetics in Washington, D. C. April 21 through 24. Several discussions with Mr. G. A. Bailey of MSFC took place during the conference. On April 27 through 30, C. K. Kuo visited MSFC to discuss magnetic measurements with Mr. G. A. Bailey and Mr. Gayle Berryman. No other trips were made under the contract during the last monthly period.

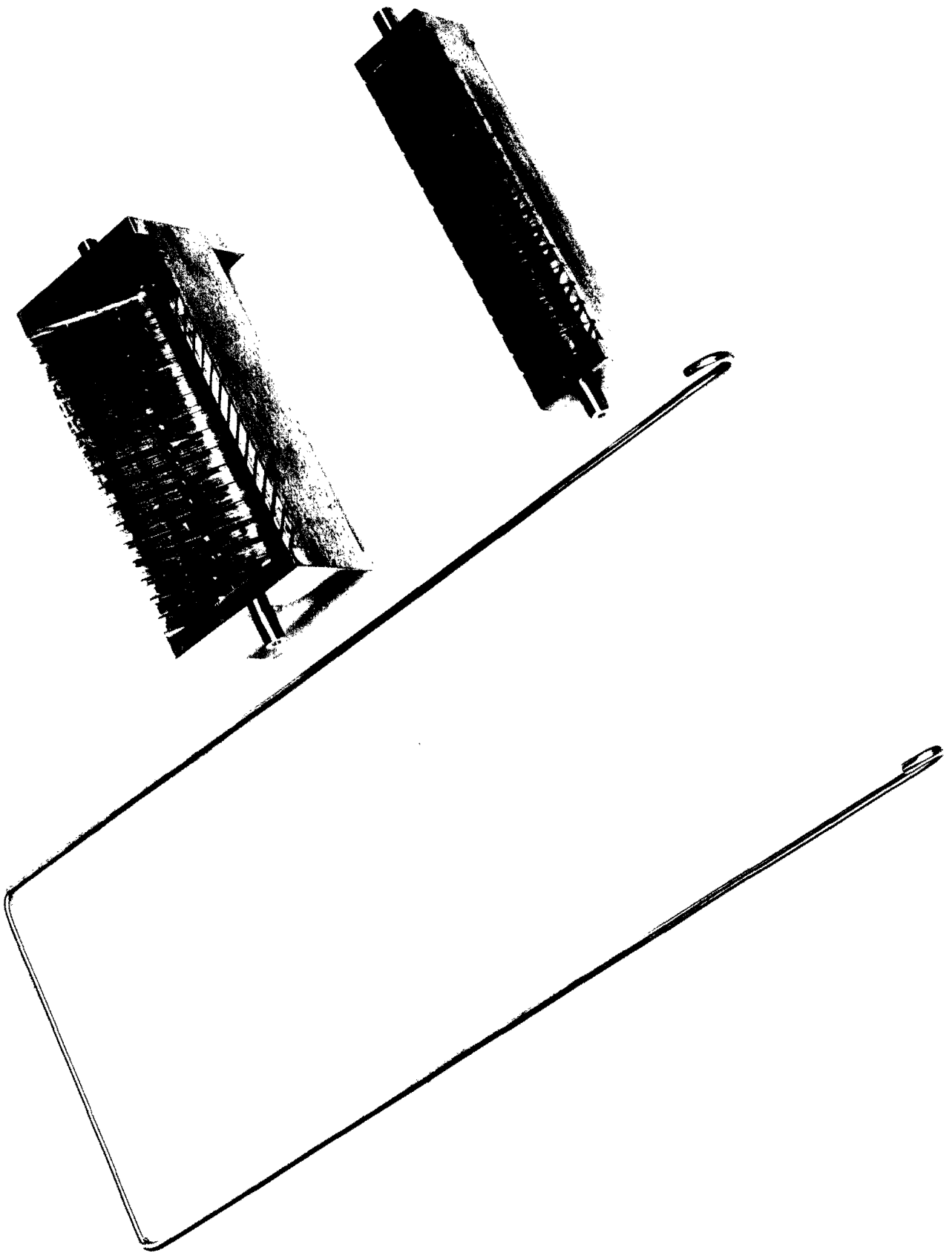
Respectfully submitted,

John A. Copeland
Project Director

JAC:brj

Enclosures





GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

June 10, 1965

George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Huntsville, Alabama 35812

Attention: PR-EC

Subject: Monthly Progress Report No. 4
Contract NAS8-20011
Project No. A-837

Dear Sir:

Phase I. Production of permalloy films by vacuum evaporation has begun. A substrate cleaning procedure consisting of soaking in hot chromic acid and rinsing with distilled and deionized water has been selected. The first films produced were in the thickness range 500 Å to 1000 Å with coercive forces of about 2.3 oe. Measurements of magnetization, anisotropy, thickness, composition, creep threshold, resistivity, and temperature coefficient of resistivity are being made.

Phase II. J. L. Brown reports that the modified specimen holder for Lorenz microscopy has been tested in the microscope. The design is satisfactory from the standpoint of applying the desired magnetic field at the specimen and compensating for the resulting beam deflection. There are some electrostatic charging effects on the surface of the plastic coil forms. Proper shielding should eliminate this.

Phase III. The rotating substrate experiment by J. O. Darnell is scheduled during the latter part of the preparation of films for Phase I which should be near the end of the next monthly period.

Phase IV. The literature search is continuing on a current basis in support of the other phases. Papers related to "Domain-wall Creep" are being collected and evaluated.

Phase V. Iron, nickel, and permalloy films have been produced by chemical vapor deposition. Presently work is being done to prevent oxidation because of back-flow and to prevent irregular gas delivery due to condensation in the gas lines. Harold Peacock is working on this phase under Dr. J. D. Fleming.

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June 10, 1965

Mr. G. A. Bailey of the Astrionics Laboratory of MSFC visited us June 7 through June 11 to discuss the program. No other trips or visits were made under the contract during the last month.

Respectfully submitted,

John A. Copeland
Project Director

JAC:brj

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

July 13, 1965

George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Huntsville, Alabama

Attention: PR-EC

Subject: Monthly Progress Report No. 5
Contract NAS8-20011
Project No. A-837

Dear Sir:

Phase I. Over one hundred permalloy films have been prepared and initial measurements of the magnetic properties have been made by M. D. Carithers and C. K. Kuo. Films which were deposited in successive depositions with the film remaining in vacuum between depositions showed a slight improvement in some characteristics; however, films prepared in the same way except that oxygen gas was admitted to the system for a few minutes between depositions show a drastic change in magnetic properties. When hysteresis loops are observed with the drive field along either of two perpendicular axis the loop is square with a H_c of about 10 oe. In between these directions the loop closes. B. R. Livesay is preparing to make torque measurements in the plane of these films to see if any biaxial anisotropy is present.

Phase II. Excellent Lorentz pictures have been obtained by J. L. Brown by using the high resolution holder which places the film in the normal specimen position in the EMU-3 microscope. Films have been deposited onto electron microscope grids at the same time that films were made for Phase I.

Phase III. The decision was made to postpone the rotating substrate experiment in order to make more films for use in Phases I and II to obtain more data on the effect of oxygen on the apparent biaxial behavior. Since a number of films for Phase I are being made during evacuation cycle from the same melt, we will obtain much of the desired information from these films.

Phase IV. The literature search is continuing on a current basis. Papers related to domain walls in thin films are being collected and evaluated.

July 13, 1965

Phase V. The problem of oxidation in the films prepared by chemical vapor deposition seems to have been solved by using carbon monoxide rather than argon as the carrier gas for the $\text{Ni}(\text{CO})_4$. Dr. J. D. Fleming reports that bright films of nickel have been obtained. Plating with $\text{Fe}(\text{CO})_5$ and mixtures of $\text{Ni}(\text{CO})_4$ and $\text{Fe}(\text{CO})_5$ is now in progress.

Dr. E. J. Scheibner and Dr. J. A. Copeland visited MSFC to discuss the progress of research under this project and the placement of emphasis during the final period.

Respectfully submitted,

John A. Copeland
Project Director

JAC:brj

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA, GEORGIA 30332

August 11, 1965

George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Huntsville, Alabama 35812

Attention: PR-EC

Subject: Monthly Progress Report No. 6
Contract NAS8-20011
Project No. A-837

Dear Sir:

All experimental studies have been completed. The data collected is being evaluated and will be presented in the final report. Sections of the final report are being written by J. L. Brown, M. D. Carithers, Dr. J. A. Copeland, Dr. J. D. Fleming, B. R. Livesay, C. K. Kuo, and Dr. E. J. Scheibner.

Dr. E. J. Scheibner and Dr. J. A. Copeland visited Goddard Space Flight Center to confer with Mr. G. A. Bailey of MSFC and Dr. R. W. Rochelle and others of GSFC on uses of thin magnetic film memory systems in space probe vehicles. Dr. J. A. Copeland and C. K. Kuo visited MSFC at different times during the last month to discuss experimental techniques and results with Mr. G. A. Bailey and Mr. G. Berryman.

Respectfully submitted,

John A. Copeland
Project Director

JAC:brj

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FINAL REPORT

PROJECT A-837

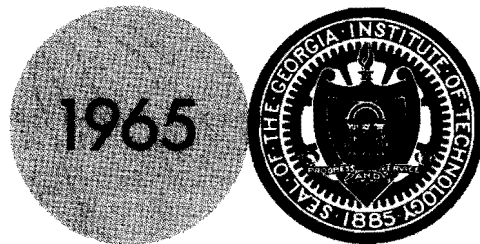
FABRICATION AND EVALUATION OF
PERMALLOY FILMS FOR MAGNETIC MEMORIES

By J. A. Copeland

Prepared for
George C. Marshall Space Flight Center
National Aeronautical and Space Administration
Huntsville, Alabama

Contract NAS8-20011

1 September 1965



Engineering Experiment Station
GEORGIA INSTITUTE OF TECHNOLOGY
Atlanta, Georgia

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ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

FINAL REPORT

FABRICATION AND EVALUATION OF
PERMALLOY FILMS FOR MAGNETIC MEMORIES

by

J. A. Copeland

1 September 1965

Contract NAS8-20011

Georgia Tech Project No. A-837

George C. Marshall Space Flight Center
National Aeronautical and Space Administration
Huntsville, Alabama

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I. INTRODUCTION

Studies of the properties of thin magnetic films have contributed a great deal to the general understanding of magnetic materials in the last ten years. Interest in permalloy films by electronic engineers has been high because of their potential as memory elements in high-speed computers. Although much of the basic physics governing the behavior of thin magnetic films is understood, there are still some phenomena about which more knowledge is needed. The two largest problems in the application of thin permalloy films are the variation in local anisotropy properties (dispersion) and the slow movement of domain walls under influence of relatively small stray fields (creep).

The work described in this report has been conducted under the sponsorship of Marshall Space Flight Center, National Aeronautical and Space Administration under Contract NAS8-20011. The purpose of this research program is to investigate the properties of thin magnetic films which have potential use in digital computer memories and to conduct a state-of-the-art survey with respect to magnetic film computer memory fabrication methods.

The main experimental study was on the properties of permalloy films made by vacuum deposition and the effects of oxidation on them. The details of fabrication are discussed in Chapter II, Section A, the measuring techniques are discussed in Chapter III, and the general results are discussed in Chapter IV.

A preliminary study of the process of making permalloy films by chemical vapor deposition using nickel carbonyl and iron carbonyl was made and is discussed in Chapter II, Section B.

This report has been organized to give the reader a general impression of the fabrication and measuring techniques currently associated with thin magnetic films which are being investigated for potential application in thin film computers. Most of the measuring techniques were used

as part of the experimental study and results of the study are used as examples of the type of information that can be gained. An extensive literature survey was made during the course of the project and appropriate references are included herein. The report therefore constitutes a comprehensive state-of-the-art survey as well as a report on the experimental measurements made. A report on State-of-the-Art on the Effects Gas Occlusions and Strains on the Magnetic Properties of Permalloy Thin Films provided by RSIC and published bibliographies^{*} on thin magnetic films should be used to supplement this report.

*See Reference Section for listing of bibliographies.

II. FABRICATION TECHNIQUES

A. VACUUM DEPOSITION PROCEDURES AND APPARATUS

Of all the possible methods for fabricating thin films the one used primarily in the study was of deposition by evaporation in high vacuum. In this section the design of various types of apparatus and procedures used in the vacuum deposition method are described in detail both for purposes of documentation and so that duplication by others can be readily accomplished. Comparable results on similar type films are often not obtained in separate laboratories because of subtle differences in fabrication methods which are not obvious in the published literature.

A total of one hundred ninety-four permalloy film specimens were fabricated for the various studies. One hundred and twenty specimens were prepared on Corning type 7059 glass substrates and fourteen specimens were fabricated on electron microscope grids for related studies and analysis. With these, sixty specimens were fabricated on glass substrates to determine film thickness and resistivity.

The principal apparatus employed in the fabrication of these specimens included substrate cleaning equipment, a high vacuum system and substrate changer, a high frequency power generator, and thickness measurement apparatus. Other small accessories necessary to establishing process control were utilized.

Substrate Selection

During this initial phase, Corning type 7059 glass was selected as the primary substrate material for studying the magnetic properties of permalloy films. The surface of this glass is quite wavy; however, the smoothness is equal to or better than one microinch, according to the manufacturer. These were obtained in dimensions of 0.7 inch x 0.7 inch x 0.032 inch. To establish process control, monitor slides were cut from non-corrosive type microscope slides to dimensions 3/8 inch by 1-1/2 inch.

The latter were employed to monitor film resistance during deposition, to provide film resistivity data, and as film thickness monitors. Conventional copper grids overcoated with carbon films were utilized for analytical studies with the electron microscope.

Substrate Cleaning Apparatus

The substrate cleaning facilities consist of conventional pyrex glass and polyethylene containers, blow bottles, hot plates, a filtered demineralized water rinse or bath, a vapor degreaser, and a rack for supporting substrates during cleaning.

The filtered demineralized water rinse is depicted in Fig. 1. It provided a temporary and useful water rinse facility for the initiation of this work. The throw-away demineralizing cartridge employed was the Universal Model manufactured by the Illinois Water Treatment Company. The filter was fabricated by stuffing a filtering grade of glass wool into a 5/8 inch diameter x 4 inch long polyethylene tube (Calcium Chloride drying tube). The covered polyethylene dish used for the water bath is 3 inches wide x 9 inches long x 3 inches deep. A water flow rate of approximately 350 cc per minute was maintained during rinsing.

A vapor degreasing chamber constructed from stainless steel was employed during the final stage of substrate cleaning. The top of the container was closed with an aluminum plate. The container was partially filled with the liquid trichloroethylene. Specimens were supported on a rack above the liquid. When the liquid was heated to the boiling point by an electric hot plate, it vaporized and condensed on the substrates. A continuous washing action occurred as the condensed droplets were returned by gravity to the liquid below. The degreaser was very useful in obtaining smear free surfaces.

A special rack was constructed from type 304 stainless steel to support substrates during cleaning. Fig. 2 illustrates the general construction of the rack. It holds sixteen substrates for batch cleaning. This particular rack design was developed by M. D. Carithers for the

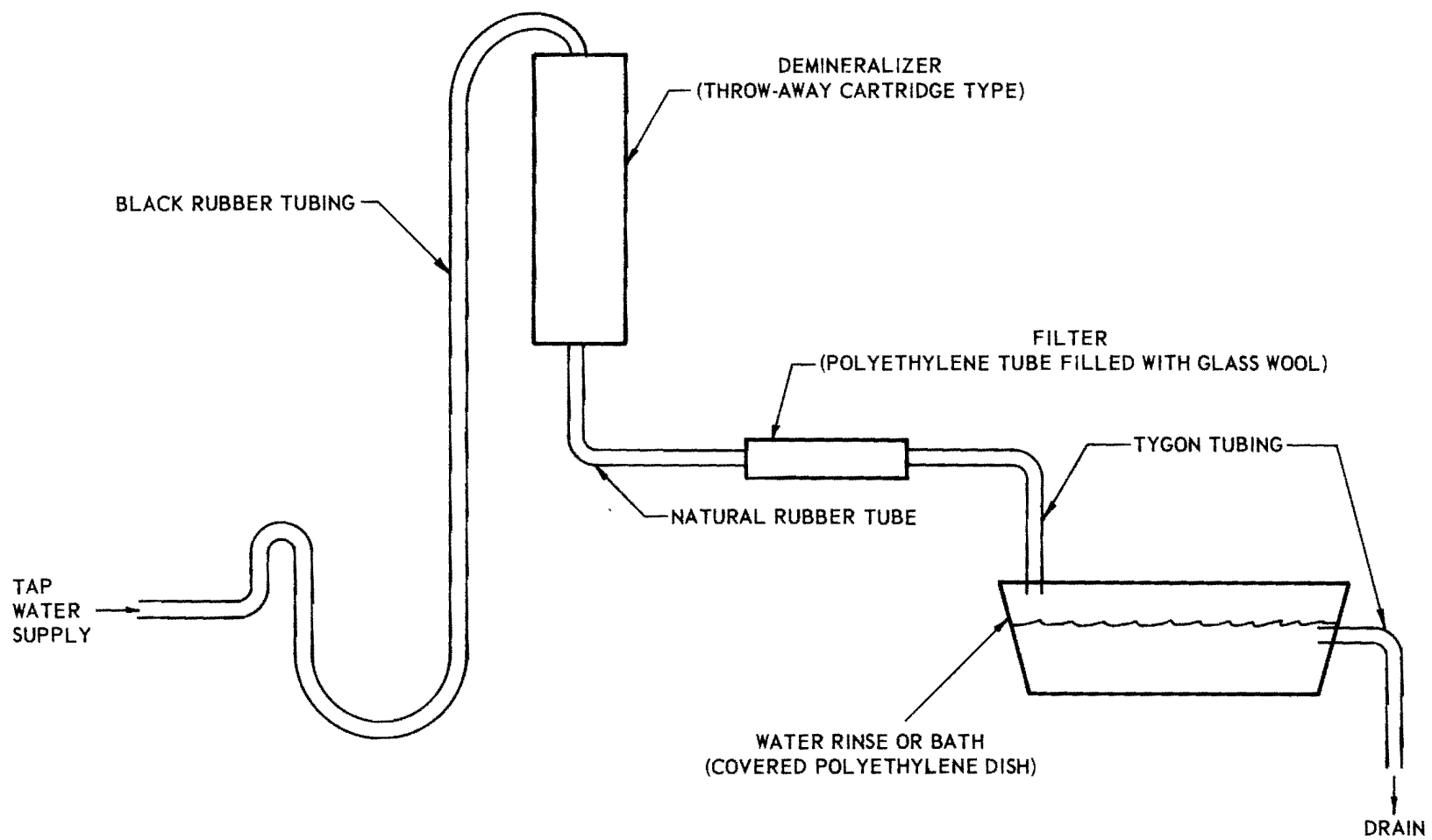


Figure 1. Demineralized Water Rinse for Substrate Cleaning.

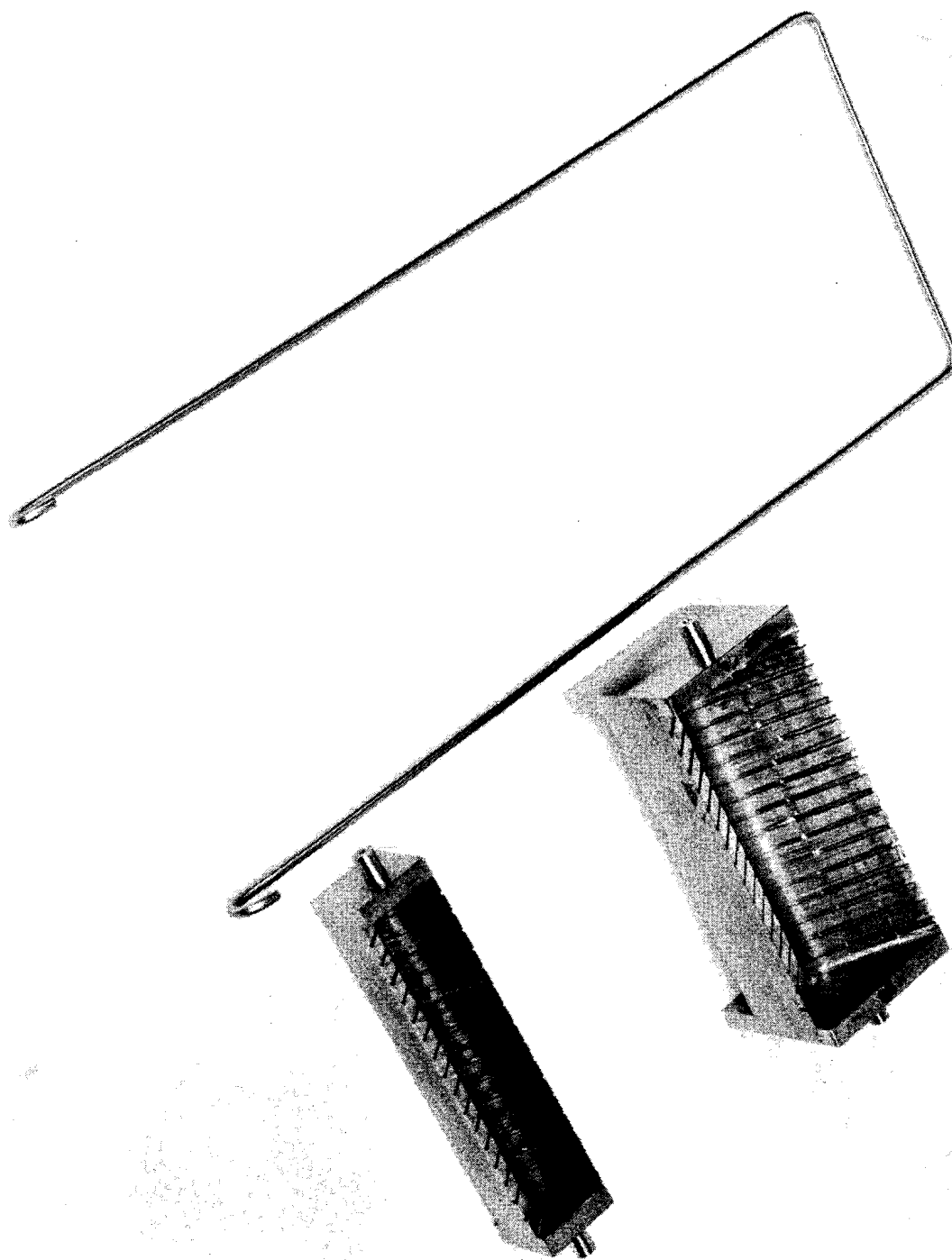


Figure 2. Substrate Cleaning Racks.

batch cleaning of rectangular substrates during his previous employment with the Collins Radio Company in Dallas, Texas. The rack is milled from a solid piece of material to eliminate deep holes and screw threads that tend to hold solution between successive baths or rinses. It is cleaned with the substrates, and the substrates are supported so as to provide for adequate drainage of liquids from the substrate corners and edges. The latter feature eliminates the gross formation of so called water marks or smears that occur if droplets of liquid collect along the edges or corners during final drying of a substrate. The hook type handle is constructed from a stainless steel rod and is used to transport the rack.

Vacuum Deposition Apparatus and Techniques

The basic vacuum deposition apparatus consists of a high vacuum system, a substrate changer and substrate masks, Helmholtz coils, and a high frequency power generator. Accessory items for the measurement of various parameters during deposition are discussed subsequently. The equipment is illustrated in Figs. 3, 4, 5, and 6.

The vacuum system is a standard Veeco model of the VE-400 series. It consists of a forepump with a pumping speed of 5 cubic feet per minute, an oil diffusion pump 4 inches in diameter, a water baffle, a liquid nitrogen cold trap, appropriate valves, gauges, an 18 inch glass bell jar, and power supplies. All pressure measurements related to this work were made with the standard ion gauge supplied with the system. The gauge tube is located near the cold trap and in the basic plumbing immediately between the diffusion pump and baseplate. Thus, the pressures recorded are probably lower than the actual bell jar pressures. This difference, however, is expected to be less than one order of magnitude, especially during evaporation when a Meissner cold trap inside the bell jar was operated.

A stainless steel feedthrough collar was added to the system to provide for extra feedthroughs for electrical apparatus and rotary shafting to operate the substrate changer discussed subsequently. Also, a Meissner cold trap was constructed and located in the bell jar. One-half inch

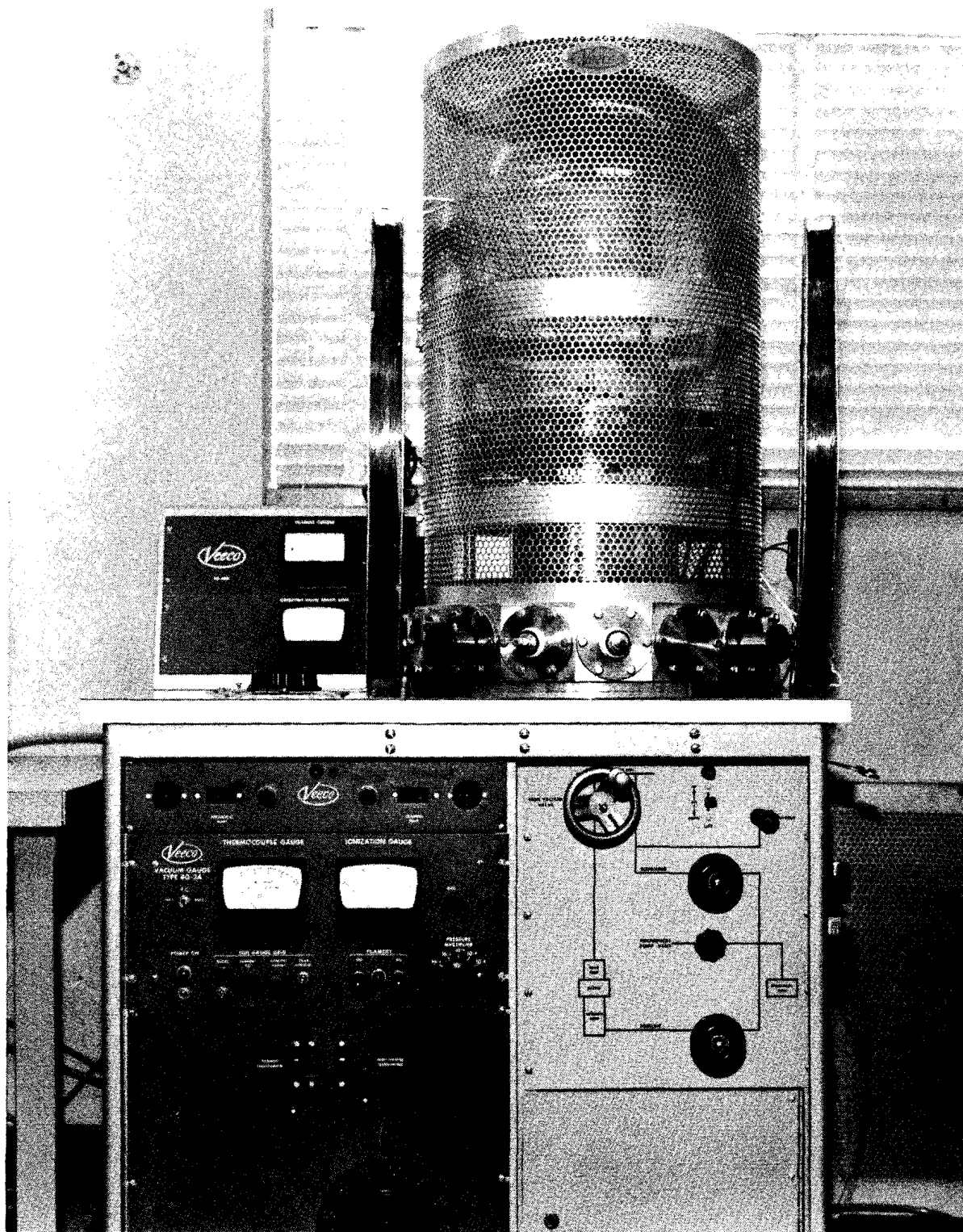


Figure 3. High Vacuum System.

diameter copper tubing was coiled to form a helix of 6 turns and approximately 6 inches in diameter and 6 inches in length. Liquid nitrogen was passed through the tubing by "Monel" alloy feedthroughs mounted in the stainless steel collar. The Meissner trap was operated immediately before and during the deposition of films. This resulted in lower pressures inside the bell jar during the critical period of film deposition.

The development of a substrate changer for this and other thin film research was funded by the Engineering Experiment Station of Georgia Tech. Because design and construction began with the initiation of this contract, primary attention was given to designing an apparatus suitable for the deposition of magnetic films. The apparatus is illustrated in Fig. 4. Non-magnetic materials free of low vapor pressure constituents, particularly zinc, were selected for construction of the changer. Primarily, types 303 and 304 stainless steel were employed. A zinc free bronze alloy impregnated with about 1-1/2 percent graphite was selected for necessary bearings and thrust washers. A few accessory items were fabricated from aluminum, copper, and beryllium copper. Mica, boron nitride, and alumina were utilized to meet electrical insulation requirements. Considerable attention was devoted to providing adequate pump-out features such as grooves or channels on mating surfaces. Screws were center drilled for screw holes that bottomed-out. Where possible bottomless holes were utilized for screws. Three legs support the apparatus. Top and bottom support plates are secured to the legs by nuts and are adjustable in the vertical direction. The substrate carrier plate is fixed to a bronze bearing fitted and supported in the center of the top support plate. A pitch chain sprocket is secured to the top of the bearing to provide rotation of the substrate carrier plate about a centrally located axle. The central axle extends through the bottom plate and is supported by a precision collar that rides on top of the bottom support plate; the substrate carrier bearing locates the axle centrally at the top end. The shutter plate is fixed to a bronze bearing that rotates freely about the center axle. A pitch chain sprocket secured to the bearing permits turning of the shutter. Miniature stainless steel pitch chains,

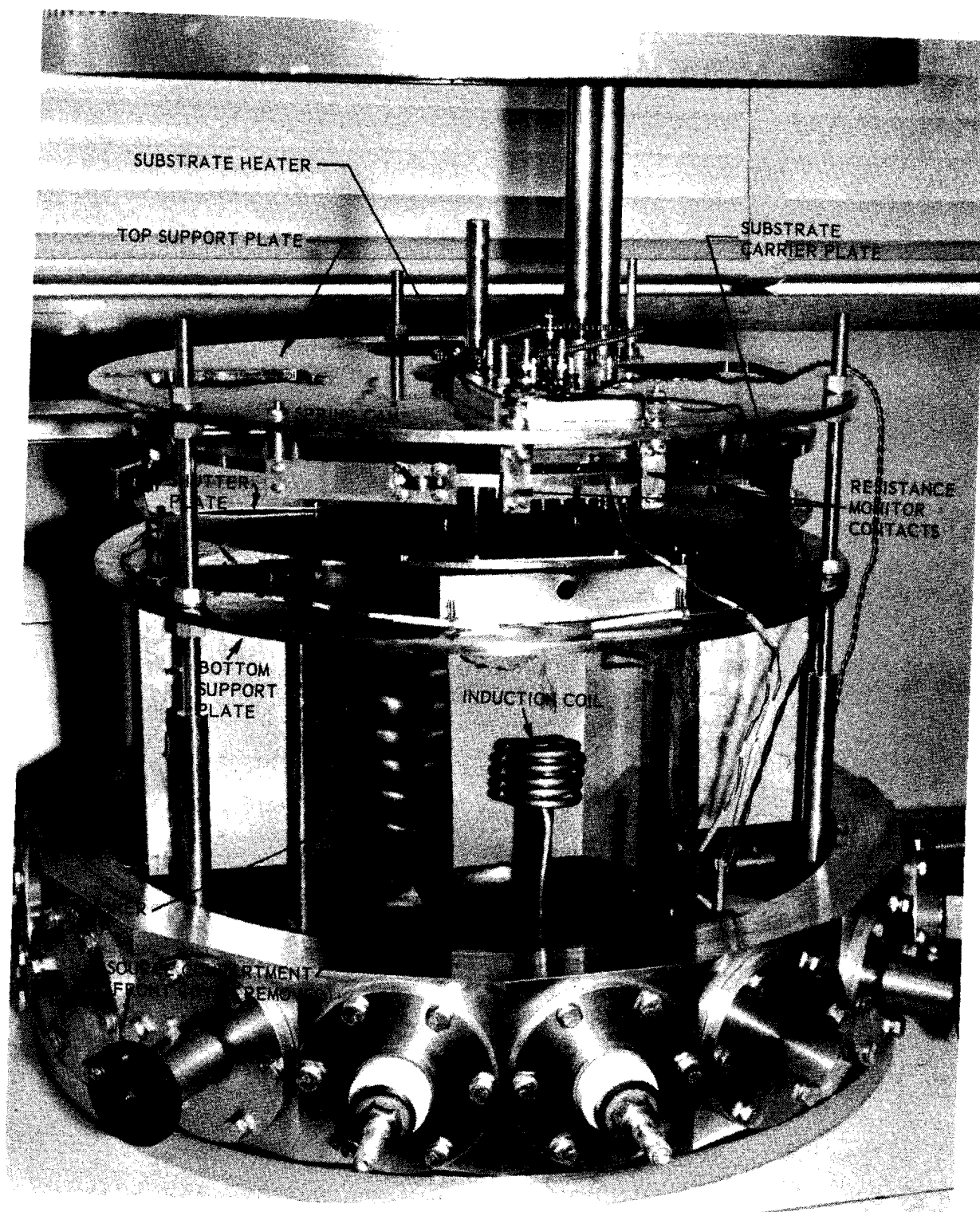


Figure 4. Substrate Changer for Deposition of Magnetic Films.

universal joints, gears, and shafting are connected to rotary feedthroughs installed in the stainless steel collar to provide for independent rotation of the substrate carrier and shutter plates from outside the bell jar. Adjustable spring cams ride on the perimeters of the shutter and substrate carrier plates. The cams engage with notches in the respective plates to obtain independent registration of the plates with respect to each other and the various source compartments. Independent substrate holders are positioned in the substrate carrier plate. Four positions will accommodate substrates with a maximum size of approximately 2-1/2 by 3-1/8 inches. Capacity can be doubled to eight such positions. Holes in the top support plate permit access to the substrate carrier plate.

The substrate holder served also as the mask for studies in this report; to meet future requirements, the changer was designed with sufficient freedom to allow for the addition of a mask changer plate between the shutter and substrate carrier plate. Four ports or vapor windows are located ninety degrees apart in the bottom plate. Evaporation filaments or sources are located below the windows. The stainless steel forms secured to the bottom side of the bottom plate are source enclosures which provide shielding of the sources from each other. Also, the shields prevent unwarranted vapor condensation on the bell jar and changer. Each source compartment has a quick-release front cover plate for ready access to the interior. An adjustable frame for holding a 1 inch x 1-1/2 inch microscope slide is mounted on top of the bottom plate at each of the vapor windows to serve as a front surface mirror for viewing each source from the exterior of the bell jar during evaporation.

Radiant substrate heater assemblies and a resistance monitor are the major accessories incorporated into the changer. The heater was designed to rest in any of the four holes in the top plate directly above a substrate or substrates to be coated and can be readily transferred from one position to another or removed for access to the substrate carrier plate. Figure 4 shows the heater installed in the changer. Major construction details are illustrated in Fig. 5. The graphite cloth element provided uniform

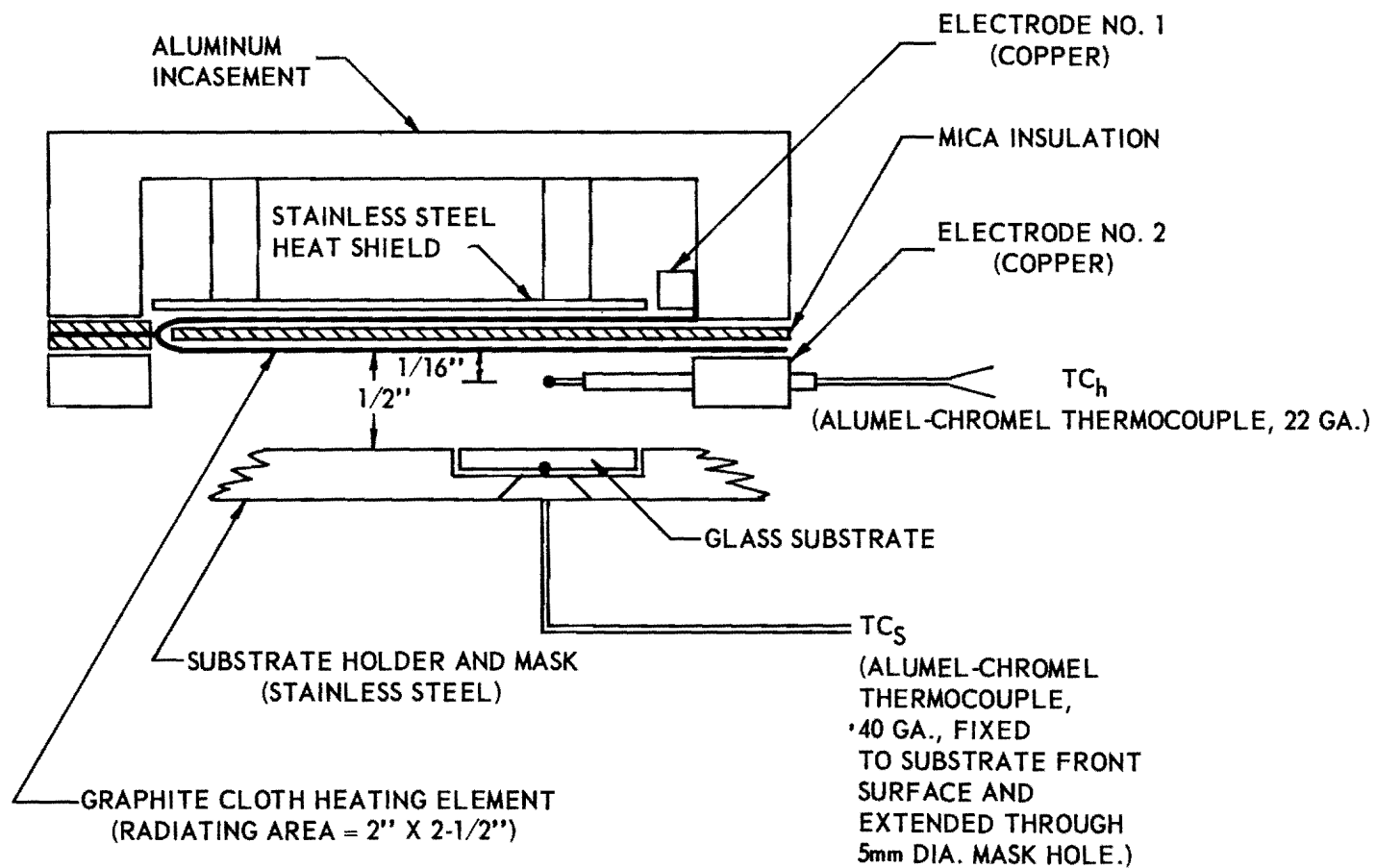


Figure 5. Substrate Heater Assembly Showing Relative Positions of Substrate and Thermocouples for Determination of Substrate Temperature.

heating over the entire substrate holder and was very efficient. For example, the cloth barely reached the color temperature range in obtaining substrate temperatures of 500°C . This indicated that the cloth was approximately 650°C , thus, operating at a temperature quite close to that of the substrate (the method of establishing substrate temperature is discussed subsequently). For the deposition of the magnetic films of this program a non-inductive element was desirable; thus, the graphite cloth was folded to form two closely spaced layers in series. The layers were insulated by a thin sheet of mica. The power supply leads to the heater were twisted to eliminate extraneous magnetic fields. The heater also operated quite satisfactorily with a single layer of graphite cloth; however, a greater current was required for the single layer element to obtain a given substrate temperature. The resistance monitor consists of a set of stationary spring contacts mounted in the top support plate that engage with a set of contacts fixed to the substrate carrier plate at each of the four substrate positions, see Fig. 4. By connecting leads from the contacts on the substrate carrier plate to pre-deposited terminations on a monitor slide in the substrate holder, the resistance of conductive films can be measured during the deposition process. This feature will permit a fair degree of process control for the deposition of conductive films and a given set of deposition parameters.

The overall performance of the substrate changer has been quite satisfactory for this program. Initially, major difficulties were encountered with bevel gears employed for the transmission of mechanical force to rotate the substrate changer plate and shutter plate. Also, set screws that secured gears and universal joints to shafts were troublesome. Replacement of the bevel gears with worm gears and the set screws with tapered pins eliminated these troubles. The heater can be simplified by constructing the case from lava or ceramic instead of metal. Also, a considerable improvement can be realized by modifying the evaporation source enclosures to serve as Meissner traps as well as vapor shields.

Four substrate holders were fabricated from type 304 stainless steel. These were machined to provide for the required masking. Each holder provides for the masking of four 0.7 inch x 0.7 inch x 0.032 inch substrates and two of the 3/8 inch x 1-1/2 inch substrates. One of the latter was used to obtain a relative thickness measurement for the complete set of substrates, and the other was employed for resistance monitoring during deposition. Fig. 6 shows the general outline of the substrate holder and the position of the substrates relative to each other and the direction of the applied magnetic field. The masking provided for the deposition of a circular pattern of five millimeters in diameter on the regular specimens for magnetic measurements. A 1/16 inch wide slit defined the deposit pattern on the thickness monitor slide, and a similar slit with "dumbbell" shaped ends at the position of the pre-deposited terminals on the resistance monitor slide defined a resistor pattern equal to 5/8 inch or ten squares in length. When electron microscope grids were coated, they were positioned over the center of the thickness monitor slit and beneath the thickness monitor slide.

Similar masks were machined to form terminals on the resistance monitor slides by evaporation techniques.

A high frequency induction heating unit, model T-2-1/2-1, manufactured by Lepel High Frequency Laboratories, Incorporated was used to evaporate permalloy from a quartz crucible to deposit the permalloy film specimens of this report. The generator operates at a nominal frequency of 450 Kilocycles per second and supplies a maximum power of 2-1/2 Kilowatts with full coupling to the load.

Helmholtz coils were fixed to the bell jar to provide a uniform magnetic field during the deposition of the permalloy films, see Fig. 3. The coils were formed by winding 20 gauge enameled copper wire on aluminum forms approximately 26 inches in diameter. The coils were spaced approximately 20 inches apart with the substrates positioned axially and midway between the coils during film deposition. The typical magnetic field intensity at the substrate was 27 oersteds. For this work, the applied

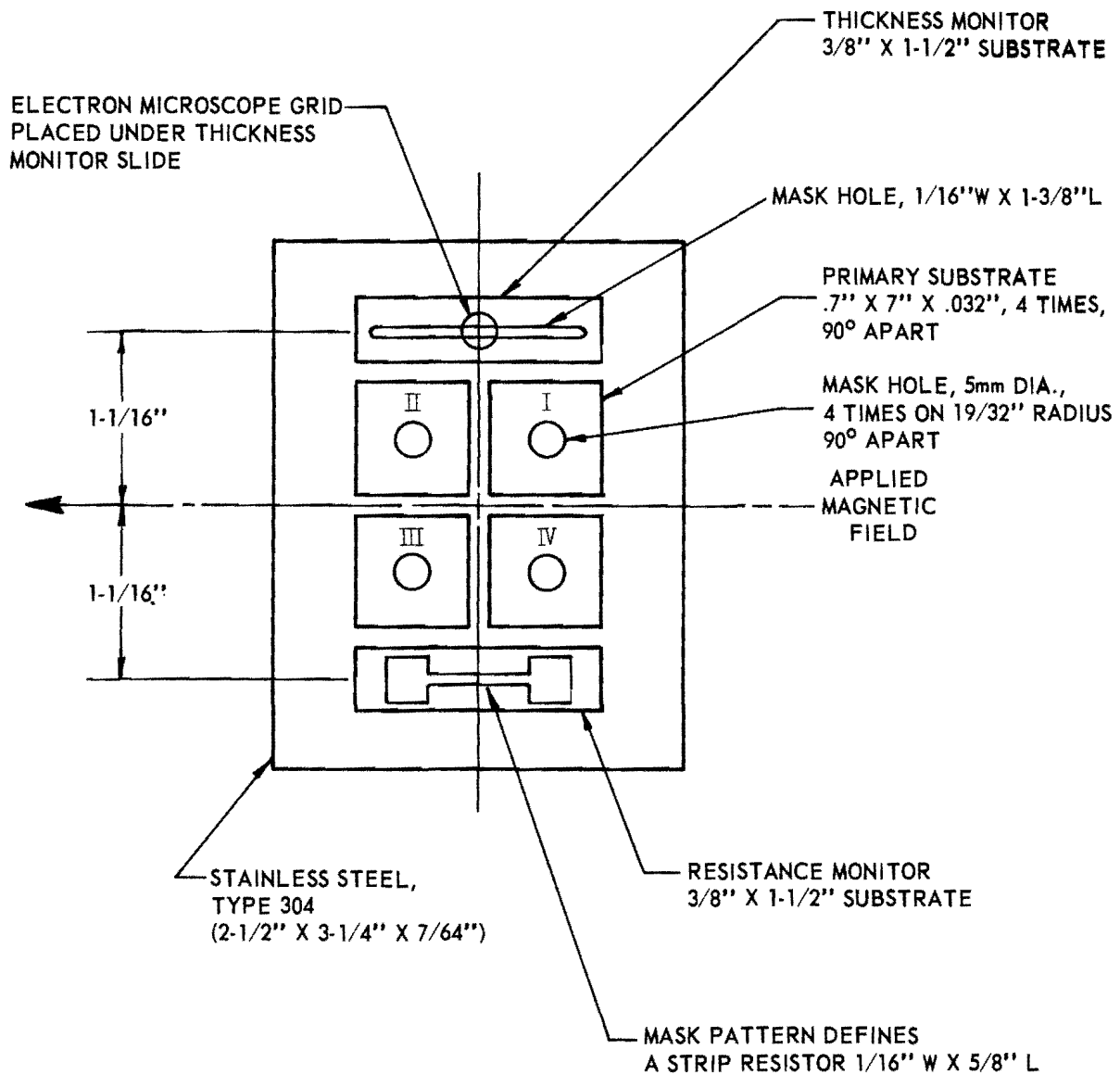


Figure 6. Substrate Holder Showing Relative Positions of Substrates and Direction of the Applied Magnetic Field.

magnetic field was directed approximately 90 degrees from that of the earth's magnetic field. A more ideal situation can be had by aligning the Helmholtz field with the earth's field.

Film Thickness Measurement Apparatus

A constant deviation spectrometer, Hilger and Watts model D 186, was used in conjunction with an interferometer to measure film thickness. The interferometer is equipped with a white light source and operates on the principle of multiple beam interferometry¹ to produce fringes of equal chromatic order. The interferometer was constructed under a previous project funded by the Engineering Experiment Station of Georgia Tech. Design was based on that described by Scott, McLauchlan and Sennett.²

Other Fabrication Apparatus

A stereomicroscope with a magnification range from 0.7X to 60X, an optical pyrometer, wheatstone resistance bridge, thermocouple potentiometers, and multimeters were available for routine measurements and examination of specimens during fabrication.

Substrate Cleaning

Five cleaning procedures were examined for possible application in this work. The procedures are described and the techniques of examination and the procedure selected for the preparation of substrates in this report are discussed below.

The following cleaning methods are similar in that the most significant variation was that of the cleaning agent and whether or not scrubbing action was used in its application. Only reagent grade chemicals were employed and use of the cleaning apparatus was restricted to cleaning substrates and substrate holders.

Cleaning Method 1: Since the glass selected for the substrates is a CaCO_3 type glass, Corning type 7059, a cleaning technique using CaCO_3 was investigated. A one-liter beaker of demineralized water was heated to the boiling point. To this, CaCO_3 powder was added to form a saturated

solution. Upon cooling, the CaCO_3 precipitated. After siphoning away excess liquid, the precipitate was dried. A dried layer of CaCO_3 approximately 1/2 inch in depth was formed on the bottom of the beaker. The top 1/4 inch was removed and mixed with filtered demineralized water to form a slurry of CaCO_3 . The substrates were cleaned in a stepwise fashion as follows:

1. Submerge substrate cleaning rack of Fig. 2 in a beaker of demineralized water.
2. With finger cots on fingers, use a 400 mesh nylon cloth between finger tips to scrub substrate surfaces with the CaCO_3 slurry,
3. Rinse away gross CaCO_3 with water from the drain tube of the demineralized water rinse of Fig. 1,
4. Place substrates in cleaning rack under water,
5. Remove racked substrates from demineralized water and place in flowing demineralized water rinse or bath of Fig. 1 and allow to rinse for a minimum of 10 minutes,
6. Remove racked specimens from water rinse and rinse with methanol from a blow bottle,
7. Successively dip racked substrates in two baths of methanol,
8. Rinse racked substrates with methanol from blow bottle,
9. Place racked substrates in trichloroethylene degreaser for a minimum of 10 minutes or until ready to place in vacuum deposition apparatus,
10. Slowly remove substrates from degreaser,
11. Use tweezers to remove substrates from cleaning rack and position in holders in the vacuum deposition apparatus.

Cleaning Method 2: A slurry was formed by mixing the detergent Alconox with water and the substrates were cleaned as in Method 1 with the Alconox slurry substituted for the CaCO_3 slurry. In step 4 hot water was used for Method 2.

Cleaning Method 3: The stepwise procedure was as follows:

1. Follow steps 1 through 5 of Method 2,
2. Place racked substrates in a fresh hot chromic acid bath, 80 to 100°C, for 5 minutes (chromic acid formed by saturating concentrated sulfuric acid with chromium trioxide at room temperature,
3. Remove from the chromic acid and rinse away gross acid with flowing demineralized water from drain tube of Fig. 1,
4. Place racked specimens in flowing water rinse of Fig. 1 for a minimum of 10 minutes,
5. Continue with steps 6 through 11 of Method 1.

Cleaning Method 4:

1. Scribe code numbers on back of substrates and arrange in deposition order in substrate cleaning rack,
2. Place racked substrates in fresh hot chromic acid 80 to 100°C for five minutes (keep chromic acid dish covered to minimize oxidation at elevated temperatures),
3. Follow steps 3 through 5 of Method 3.

Cleaning Method 5: Specimens cleaned by each of the previous four methods were selected for flaming. These were fire polished in the oxidizing region of the flame of a Meeker burner.

In the performance of the above cleaning procedures, the substrates were not allowed to dry between successive baths. The methanol rinses are used primarily to remove water from the substrates and rack before degreasing since water and trichloroethylene do not mix very well. The trichloroethylene degreaser is used primarily as a storage point immediately before film deposition and as a technique of drying the substrates to obtain a streak free surface rather than for any unique cleaning or degreasing property of the trichloroethylene vapor.

Selection of Cleaning Method

Method 4 of the preceding cleaning procedures was selected as the most appropriate for this work and was employed for all of the permalloy films of this study. The selection was based upon a comparative examination of substrate surfaces with a stereomicroscope, time and ease of performance, ease of process control, and past experience.

Immediately after cleaning, the substrate surfaces were examined with a stereomicroscope at magnifications ranging from 3X to 30X. To aid in the detection of surface particles, the substrates were placed upon a clean piece of blue cobalt glass. A low intensity microscope lamp was held so that the light intercepted the surface at a small angle while the surface was viewed from the normal direction with the microscope. This technique permits the detection of particles and surface defects that do not ordinarily appear with normal and full illumination. From a standpoint of particle count, Method 1 was inferior to Methods 2, 3, and 4. Relatively, the surfaces of substrates cleaned by Method 1 were heavily populated with "silica gel like" particles (so named because of their inertness to strong acids and flaming). The surfaces of substrates cleaned by Methods 2, 3, and 4 appeared about the same.

After the microscopic examination, substrates cleaned by Methods 1, 2, 3, and 4 were selected for fire polishing as discussed in Method 5 above. No significant reduction in particle population was detected. However, this technique can be used to some possible advantage in smoothing out surface defects such as surface scratches on soft glass. Aside from the extra time and handling required, the main disadvantage was technique control. The substrates were held with tweezers during the flaming and the results depended considerably on artful execution by different personnel. Thus, fire polishing was eliminated from further consideration for the program.

At this point, the choice of cleaning techniques lay between Methods 2 and 4. Alconox is an ionic detergent but quite satisfactory results have been obtained with its use in cleaning substrates in this laboratory,

especially, when adherence of subsequently deposited films was used as a judgment criterion. Likewise, chromic acid has been used successfully with similar results. In certain instances the two cleaners have been used successively, as was done in Method 2, to obtain superior adherence properties of subsequently deposited films. However, where glass substrates are vacuum baked as a preliminary step to film deposition, there is no apparent need of combining the two cleaning agents as in Method 2. Alco-nox is a proprietary product; on the otherhand, chromic acid can be prepared from reagent or better grade chemicals of known composition to provide a greater degree of experimental control. Consideration was given to the inability to completely remove chromium ions from the glass surface by cleaning Method 4 and their possible effects on the magnetic properties of subsequently deposited films. Since most of the films in this program will be deposited in thicknesses greater than $1000 \text{ \AA}$, it was felt that the chromium ion impurities are sufficiently limited to be insignificant. Also, in future work, it is expected that films of SiO_2 will be deposited on substrate surfaces immediately before magnetic film deposition to promote surface smoothness and film adherence and to provide a more consistent surface chemistry. Thus the effects of chromium ions left during the initial cleaning on the magnetic film will be further reduced or, essentially, eliminated (SiO_2 layers were not employed on any of the specimens included in this report).

After the above considerations, substrate cleaning Method 4 was considered as the most desirable for the initiation of this work. During the course of vacuum deposition, further cleaning was obtained by vacuum baking of the substrates immediately before film deposition.

General Fabrication Procedure

Details of the procedures and preliminary precautions followed to fabricate specimens by vacuum techniques are discussed.

Preparation of Vacuum Deposition Apparatus: The oil diffusion pump was disassembled and cleaned by scrubbing and rinsing with

trichloroethylene and acetone. After drying with hot air, it was filled with new oil, Dow Corning type 704. The interior surfaces of the vacuum chamber were cleaned with acetone and paper towels.

When the substrate changer was initially received it was contaminated with cutting oils and required thorough cleaning. Stainless steel and ceramic parts were cleaned with chromic acid; copper and bronze parts were cleaned in dilute nitric acid; and, after first scrubbing with 'Brillo' pads, aluminum parts were rinsed with Alconox solution (about 1 teaspoonful of Alconox powder dissolved in 1 liter of hot water). The acids and detergents were removed by thoroughly rinsing the parts successively in flowing de-ionized water and methanol. After the parts had dried, the changer was assembled and installed in the vacuum plant. The cleaned parts were handled with finger cots and nylon gloves. Once preparation of the apparatus was completed, the system was evacuated. The vacuum pumps were operated continuously, and the bell jar was maintained in an evacuated condition except for routine openings during the fabrication of specimens and for repairs or adjustments.

During subsequent evaporations, film deposits were formed on exposed areas of the changer. These were allowed to grow in thickness until they could be peeled off with a portable hand vacuum cleaner. The more persistent deposits were removed by scrubbing with paper towels saturated with acetone.

Before the fabrication of each series of permalloy specimens, permalloy wire was added, if required, to a quartz crucible and melted in high vacuum by induction heating. The crucible was 7/18 inch in diameter and 1/2 inch deep and was supported by a boron nitride rod at a distance of 6-1/2 inches below the substrates. A sufficient quantity of permalloy was melted to fill the crucible to a depth of about 1/4 inch. The high frequency generator was adjusted to obtain an evaporant temperature of approximately 1380°C after heating for a period of one minute. Evaporant temperature was measured with an optical pyrometer as indicated in Fig. 7.

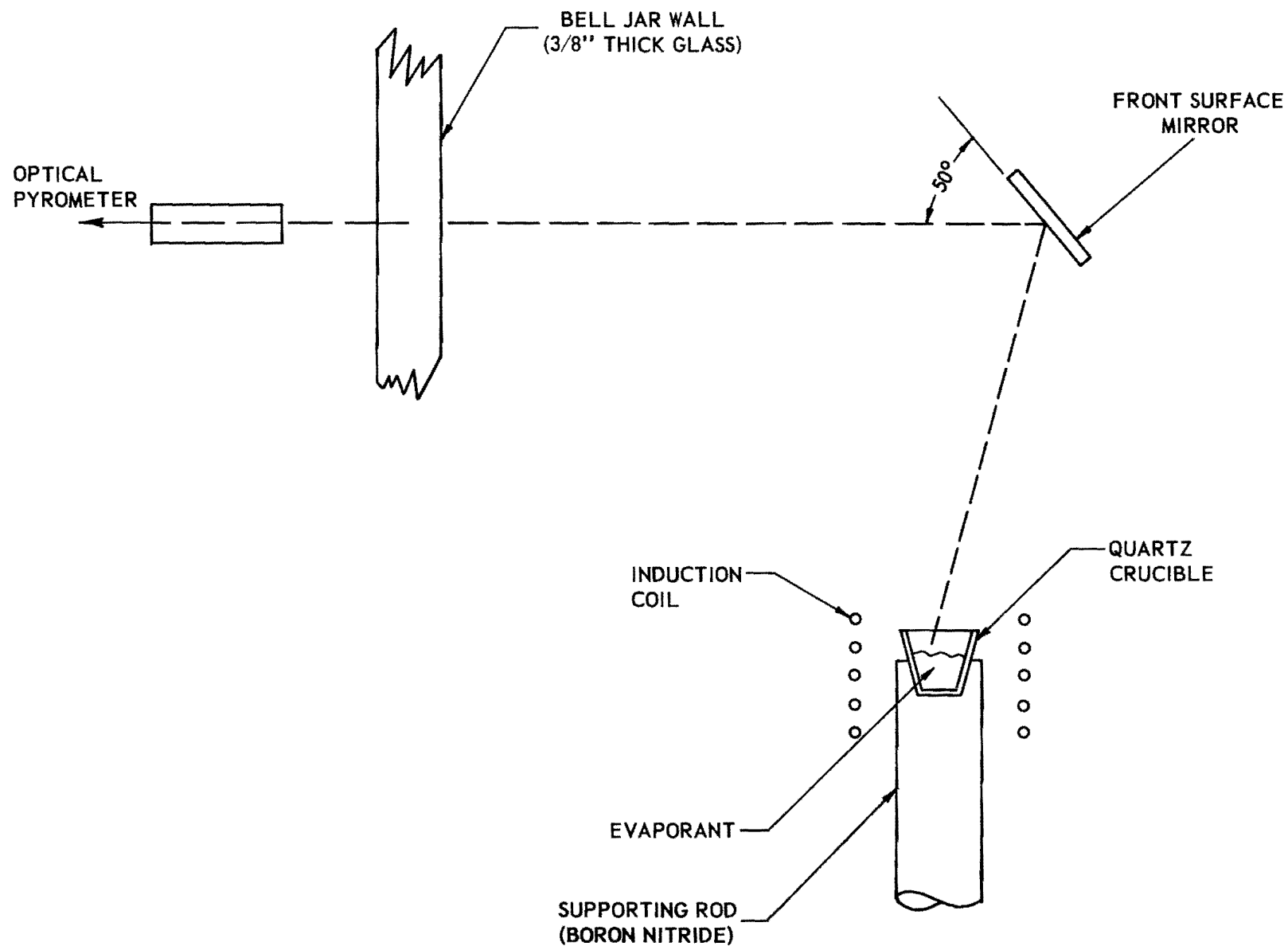


Figure 7. Arrangement for Measuring Evaporant Temperature.

Preparation of Substrates: Substrates for the resistance and thickness monitors were cut from soft glass microscope slides; whereas, Corning type 7059 glass substrates were employed for the primary permalloy film specimens.

Resistance monitor slides were prepared a few days in advance of permalloy film depositions. The substrates were cleaned by Method 4. Subsequently, terminals were formed on the slides by evaporating a dual layered film of gold over chromium. Separate source compartments of the substrate changer were used to deposit the gold and chromium. The substrates were vacuum baked to 350°C and the chromium was deposited to a thickness of about $500 \text{ \AA}$. Immediately, the substrates were rotated to the gold position and gold was deposited over the chromium to a thickness of about $2500 \text{ \AA}$. The terminated slides were stored in a desiccator until they were used during subsequent permalloy film deposition.

Initially, the four substrate holders or masks were cleaned by Method 4, wrapped in aluminum foil, and stored in a desiccator; thereafter, they were wrapped in aluminum foil and stored in the desiccator between successive depositions.

Immediately before each permalloy deposition, the substrate masks were installed in the changer apparatus. In the meantime, the thickness monitor substrates and the Corning glass or primary substrates were cleaned by Method 4 as two separate batches. Also while these were being cleaned, gold wire leads were bonded to four of the pre-terminated resistance monitor slides by thermal compression techniques and installed in the substrate masks. The thickness monitor and primary substrates were transferred directly from the vapor degreaser to the deposition masks immediately before evacuation. The arrangement of the substrates in each of the four masks is shown in Fig. 6.

General Evaporation Procedure: After the substrates were installed in the deposition apparatus, the following general procedure was followed as a check list to deposit the permalloy films:

1. Fill LN_2 trap with LN_2 at beginning of an evaporation series and add as may be required.
2. Evacuate to about 2 to 5×10^{-7} Torr,
3. Turn on substrate heater and set for desired temperature,
4. After heating substrates for 25 minutes, start LN_2 through Meissner trap,
5. After 28 minutes of substrate heating, turn on Helmholtz coils and adjust for maximum current and record coil current (should be about 3.8 to 4.25 amp.),
6. After 30 minutes of substrate heating, record substrate temperature and high vacuum pressure,
7. With shutter closed, turn on high frequency generator and heat evaporant for one minute, check evaporant temperature with optical pyrometer, and make necessary power adjustments for obtaining an evaporant temperature of approximately 1380°C for a one minute heating period,
8. Open shutter and evaporate permalloy for the preselected time, record pressure during last 15 seconds of evaporation, and record monitor resistance at the end of the evaporation,
9. Close the LN_2 valve to the Meissner trap,
10. Turn substrate heater off and leave Helmholtz coils on until $\text{TC}_h \leq 100^\circ\text{C}$, unless specified otherwise,
11. Switch off Helmholtz coils,
12. Rotate next group of substrates into deposition position and proceed as above until all four sets are completed,
13. After permalloy is deposited on the final set of substrates, cool until TC_h indicates a temperature of 50 to 75°C ,
14. Open vacuum chamber, remove specimens and store in a desiccator.

Substrate temperature was indicated by a 22 gauge alumel-chromel thermocouple, TC_h , positioned between the graphite cloth heating element

of the substrate heater and the substrate holder. TC_h was calibrated against a second alumel-chromel thermocouple, TC_s , fixed to the front surface of a substrate in position III of the holder of Fig. 6. The latter thermocouple was fabricated from 40 gauge wires. A loop of nichrome wire was heated by resistance heating and employed to embed the thermocouple in the substrate surface. Fig. 5 illustrates the arrangement of the thermocouples for calibration of TC_h . The temperatures indicated by both thermocouples were measured versus time for one hour and one-half hour heating periods and various variac settings of the heater power supply. Data obtained for one hour heating periods are plotted in Fig. 8. The thermocouple fixed to the substrate, TC_s , indicated higher temperatures than TC_h for final temperatures below 425°C and lower values for final temperatures above 425°C . Heater currents at the end of the heating periods ranged from 5 amperes for a substrate temperature of 200°C to 14.1 amperes for a substrate temperature of 500°C . These data were used to interpolate substrate temperatures from measurements with TC_h during the fabrication of the permalloy films.

The resistance of each resistance monitor was measured with an ohmmeter during deposition. After removal from the vacuum chamber, the resistance of each monitor was measured at room temperature with a wheatstone bridge. Data tables reflect the latter values. When sufficient film resistivity data has been obtained for these films, the resistance monitor can be employed to control the thickness of film deposits.

Pressure measurements were discussed previously in the description of the vacuum apparatus.

Fabrication of Experimental Specimens

One hundred ninety-four permalloy film specimens were fabricated for this study. These were grouped and deposited in thirty separate evaporation. Included in each group were four Corning type 7059 glass substrates, a thickness monitor, and a resistance monitor. In addition, an electron microscope grid was included in fourteen of the deposition groups. Fabrication data are given in Tables 1 and 2 of the Appendix. Detailed data for all

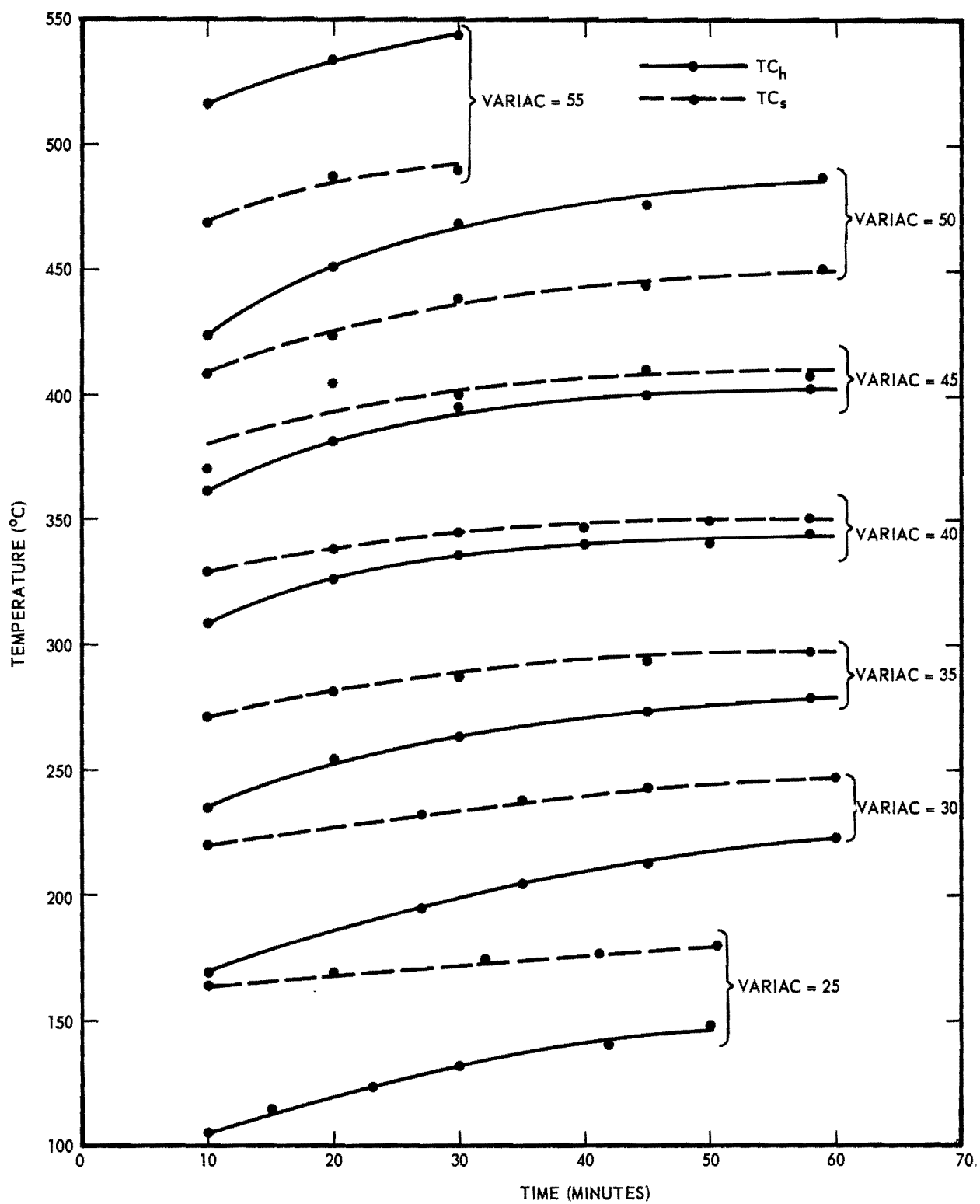


Figure 8. Substrate Temperature vs. Time and Variac Setting.

films are listed in Table 1. In Table 2, the specimens are grouped according to deposition group and order of deposition to illustrate more clearly the significant fabrication variables. Films were deposited simultaneously on all of the substrates in a particular group, for example, specimens 1 through 4. Specimens numbered 1 through 120 were fabricated on Corning type 7059 glass substrates. Those numbered E-1 through E-14 were electron microscope grids. Except for the electron microscope grids, all of the substrates were cleaned by Method 4 before vacuum processing. Additional cleaning was obtained by baking the substrates in high vacuum immediately before film deposition.

Permalloy wire with a composition of 80.55 percent nickel and 19.45 percent iron was evaporated from a quartz crucible by induction heating techniques to deposit the films. The general evaporation procedure previously discussed was varied according to the experimental variables introduced. Predominantly, the films were deposited in high vacuum at pressures in the 10^{-5} Torr range at an average rate of approximately 400 Å per minute. The deposition rate varied slightly from the average value depending on the total evaporation time and variations in evaporant temperature. Substrate temperatures were varied from room temperature to 400°C. Helmholtz coils were used to apply a uniform magnetic field during deposition and subsequent cooling of most of the films. One group of films (specimens E-14 and 117 thru 120) was deposited in a residual oxygen environment to form films composed of Ni-Fe oxides. Also, films were deposited in single and multi-layered structures to thicknesses ranging from 230 to 2500 Å. Multi-layered structures were formed on a number of specimens by interrupting the evaporation at high vacuum for a 5 minute period; other layered structures were formed by admitting dry O₂ into the vacuum chamber during an interrupted evaporation cycle. Film structures consisting of up to four permalloy layers separated by thin Ni-Fe oxide films were fabricated by each technique.

The condition of the permalloy melt and evaporant is implied in the second column of Table 2, headed Ni-Fe EVAPORANT. "New Melt" indicates

that the quartz crucible was filled with new permalloy wire before the evaporation; otherwise, the same melt was used to deposit films on previous groups as indicated.

The total thickness of the film on the thickness monitor coated with each group of specimens is given in column 10 of Table 2. These values are an accurate indication of the film thicknesses on corresponding microscope grids; however, films on the Corning glass substrates were from 5 to 20 percent thicker than the value indicated for the corresponding monitor films.

Several specimens were fabricated as layered film structures. The number of permalloy layers in the structure is denoted in column 9 of Table 2. The layers were formed by depositing a permalloy film and oxidizing the deposit for the specified time in a high vacuum environment at pressures of $(1 - 10) \times 10^{-6}$ Torr or admitting dry oxygen into the system and oxidizing in a partial pressure of oxygen. The deposition-oxidation cycle was repeated an appropriate number of times to form multi-layered permalloy structures. The temperature of the substrates were maintained at the deposition temperature during the oxidation. The word "NONE" indicates that an oxidation cycle was not introduced; however, even these films underwent the normal surface oxidation that could not be prevented under the fabrication conditions. Those films oxidized in dry O_2 were exposed to the O_2 for approximately 20 minutes. The O_2 was admitted to the system until the pressure increased to 1 Torr. After oxidizing for 10 minutes at 1 Torr, the oxygen bleeder valve was closed. An additional 10 minute period was required to evacuate the system to 5×10^{-7} Torr.

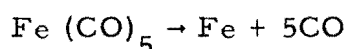
The last six columns of Table 2 indicate if a magnetic field was applied during the fabrication phases of deposition, oxidation, and/or post-deposition cooling. The magnetic intensity of the applied field was 27 ± 2 oe, and it was directed approximately 90 degrees from that of the earth's magnetic field.

M. D. Carithers

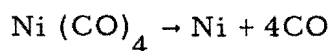
B. CHEMICAL VAPOR DEPOSITION

Deposition of magnetic films by chemical vapor plating appears to offer several potential advantages. The compounds which are the sources of the metals have high vapor pressures in contrast to the low vapor pressures of most contaminants likely to be present. Distillation of the compound thus affords high purity with little effort. Since the process involves several independent variables, control of the composition and crystalline nature of the film should be facilitated. Since the film is deposited under conditions close to chemical equilibrium, an active mass transport mechanism is operative in contrast to the situation with evaporation and sputtering. As a result, near-equilibrium alloy structures may be expected and post-deposition annealing to promote homogeneity may be simplified. The chemical vapor plating process is amenable to continuous operation thus lending itself to efficient production applications.

These advantages appeared sufficiently promising to warrant a brief feasibility study of chemical vapor deposition of iron and nickel. For this preliminary study, deposition by pyrolysis of the carbonyls was selected since the required compounds are commercially available. Pyrolysis occurs according to the equations



and



The feasibility study was intended to evaluate the possibility of depositing thin films of iron and nickel on suitable glass substrates and to outline the engineering problems associated with this process under conditions suitable for large scale production. In keeping with this objective, the plating pressures were restricted to the atmospheric range and the necessary environmental conditions were obtained by flushing rather than

operation under reduced pressure. No attempts were made to determine optimum plating conditions, to evaluate the deposition kinetics, or to control the composition of the films beyond eliminating gross oxidation.

Experimental Work

A furnace was constructed of the form shown schematically in Fig. 9. The base plate was machined from fused silica foam. The heating coil was wound from Kanthal wire and placed in a circular hollow covered by a metal plate upon which the substrate rested. The metal plate was drilled radially and an insulated thermocouple was placed at the center point to permit temperature monitoring. The plating gas stream was conducted over the substrate through a coarse fritted glass filter which served as a diffuser. The system was contained within a Pyrex pipe joint from which the gases escaped through a sand seal.

In order to determine the temperature uniformity on the heater plate, nine thermocouples were spot-welded to the heater plate in the positions indicated in Fig. 10. The temperatures were found to vary within $\pm 10^{\circ}\text{C}$. In order to improve the temperature uniformity, grains of fused silica were placed on the heater coil at positions beneath the thermocouples giving the higher temperature readings. By trial and error relocation of these grains, the temperature variation was decreased to $\pm 1.5^{\circ}\text{C}$ as shown by the readings tabulated in Fig. 10.

Plating with $\text{Ni}(\text{CO})_4$: Following construction of the furnace, plating was begun with $\text{Ni}(\text{CO})_4$ using argon as a carrier gas. The $\text{Ni}(\text{CO})_4$ was introduced by gentle heating of a containment cylinder and the flow rates were monitored with rotameters. Condensation of the carbonyl occurred in the transfer lines but this problem was eliminated by wrapping the lines with electrical heating tape. Adherent coatings of nickel were obtained at temperatures ranging from about 160 to 230°C . With a few exceptions, however, the films were obviously oxidized.

Attempts were made to eliminate oxidation by cold trapping and scavenging the argon. Since these efforts were unsuccessful, the oxidation

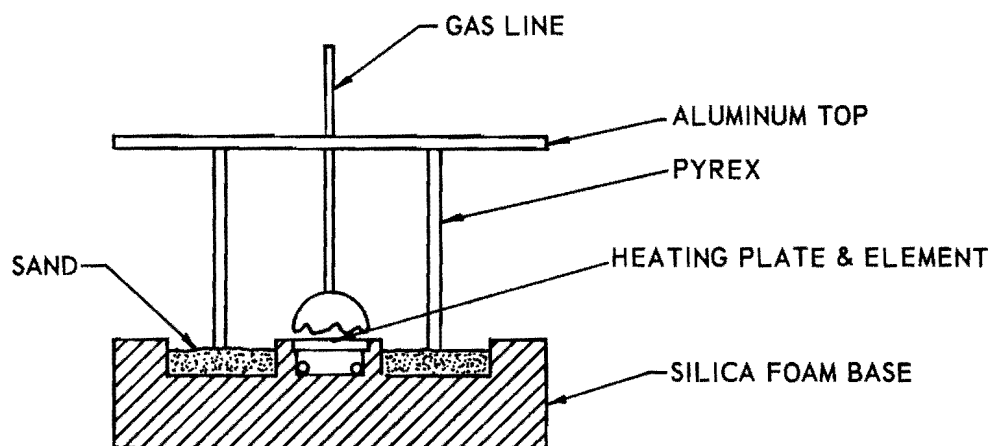
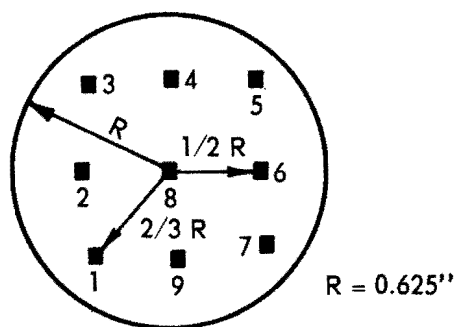


Figure 9. Chemical Vapor Deposition Furnace.



THERMO. NO.	1	2	3	4	5	6	7	8	9
TEMP. °C	188	187	186.8	188	188	189.8	187.5	189.3	188.5

Figure 10. Thermocouple Arrangement in Furnace.

was attributed to inadequate blanketing of the substrate by the flowing gas. This problem was not solved by surrounding the substrate with a secondary flow baffle although some improvement was noted.

In order to augment the protection afforded by gas blanketing with improved protection by a reducing atmosphere, carbon monoxide was substituted for argon as the carrier gas. While only a few runs with carbon monoxide have been made, it appears that this has strongly decreased and possibly eliminated the problem with oxidation. In two runs, bright films were obtained. In a third run, the film showed the effects of oxidation but the system was only briefly flushed with carrier gas prior to the deposition.

The nickel films deposited appeared uniform insofar as light transmission is concerned. The films were adherent and showed no macroscopic flaws although the plating surface was darkened, indicating that homogeneous gas phase nucleation of the nickel may have occurred. This would be undesirable since the resulting finely divided nickel would cause catalytic disproportionation of carbon monoxide and increase the carbon content of the film. This problem should be eliminated by a lower carbonyl concentration in the carrier gas and by the use of a water cooled injection nozzle.

Plating with $\text{Fe}(\text{CO})_5$: Since the vapor pressure of $\text{Fe}(\text{CO})_5$ is lower than that of $\text{Ni}(\text{CO})_4$, condensation of the carbonyl in the transfer lines has proved more troublesome than with the nickel plating. The first films deposited showed little oxidation but the deposits were spotty, indicating carbonyl starvation. This would be likely to result from condensation in the transfer lines. Following these results, the baffle plate in the gas distributor was removed, the heating arrangement and insulation of the lines carrying the $\text{Fe}(\text{CO})_5$ was modified, and the carbon monoxide carrier gas was preheated. Condensation of the $\text{Fe}(\text{CO})_5$ was, thereby, avoided and uniform films were obtained. These films, though uniform, were quite brittle. It was concluded that the brittleness was due to the presence of carbon in the films. Returning to the use of argon, instead of carbon monoxide, as the carrier gas alleviated this condition, and satisfactory iron films were obtained.

Proposed Future Work

The preliminary study indicates that chemical vapor deposition of iron-nickel films by deposition from the carbonyls should present few major problems. As a result, it appears that a more detailed, systematic study is warranted. This study should include investigation of the following factors:

1. Kinetics of deposition of iron and nickel from mixed carbonyl vapors as influenced by temperature, pressure, concentration, and the nature of the carrier gas.
2. Physical and chemical uniformity of the films.
3. Characterization of the films as to microstructure and pertinent physical properties.

W. J. Corbett
J. D. Fleming

C. ELECTROCHEMICAL DEPOSITION

The process of making alloy films of iron, nickel, and cobalt is made easier by the fact that the standard potentials of the three metals are within the range -0.25 to -0.44 volts.³ The characteristics of the plated films are very similar to films of the same composition made by vacuum deposition, including a uniaxial magnetic anisotropy induced by a magnetic field applied during the formation of the film.^{4, 5} Phosphorous may be added to the film by using hypophosphite as an anion in the plating bath. A small amount of phosphorous may be used to reduce the anisotropy constant, H_k , to the order of 1 oe.⁶

Thin magnetic films have been plated onto flat metallic substrates, metal coated glass substrates, and onto copper and beryllium-copper wires.⁷ Usually the substrates are smoothed by electro-etching. In the case of 5 mil wires, as much as 1 mil may be etched off and replated before deposition of the magnetic film.

The magnetostriction constant is less sensitive to the nickel to iron composition ratio in electrodeposited permalloy films than it is in vacuum deposited films, which in turn are much less sensitive than bulk samples.⁸ Drastic aging effects in electrodeposited films have been reported,⁹ but it seems that recently these effects have been much reduced by proper over-coating.

No experimental studies of electrodeposited films were made as part of this project, but the process is mentioned because of the increasing interest in electroplated thin film computer elements.

J. A. Copeland

III. MEASURING TECHNIQUES

A. HYSTERESIS LOOP

The hysteresis, or M-H loop tracer^{10, 11, 12} has been one of the effective tools for studying magnetic thin films. The properties of magnetic thin films, such as coercive force, anisotropy field, magnetization, susceptibility, Curie point, dispersion, remanence and anisotropy characteristics, etc., can be evaluated with the hysteresis loop tracer.

The hysteresis loop tracer used for taking the data in this report operates at 60 cps. With its high sensitivity, it allows measurements to be made on permalloy films 5 mm in diameter and less than one hundred angstroms in thickness. Both the specimen holder and pickup coil holder are made so that they can be rotated 360°. The angular resolution of the specimen holder is better than $\pm 0.2^\circ$.

General Description

A magnetic thin film is placed at the center of a pair of Helmholtz coils which produce an alternating magnetic field strong enough to saturate the film. A voltage is induced in the pickup coil, which is placed near the film, by the flux change due to the magnetization change of the film. The voltage, often being electronically integrated, produces a signal proportional to the magnetization, M. This signal is amplified and displayed on the vertical axis of a cathode-ray tube, as a function of the driving field which is displayed on the horizontal axis to get the hysteresis loop of the film. A schematic diagram of the loop tracer is shown in Fig. 11.

Field Coils and Pickup Coils

The Helmholtz coils used to produce alternating fields have 1000 turns and produce a field of 37.5 oe/amp. A current sampling resistor, 0.1 Ω and ± 0.1 percent in accuracy, is connected in series with the Helmholtz coils to measure the field intensity, H.

The pickup coil is made up of two D-shaped coils with their straight edges bound together. The two halves of the pickup coils are wound so that the voltages in the two halves induced by the magnetization change of the film are adding at the output terminals of the pickup coils. The number of turns of the pickup coil is determined by evaluating such factors as the necessary sensitivity of the M-H loop tracer, stability of the system, noise, and the practical size of the coils. The pickup coil being used in these measurements has 50 turns in each half.

A set of 9 inch cube coils (3 orthogonal pairs of square coils) driven by battery power are used to cancel the earth field at the location of the film. The earth's field at the film can be nulled to ± 0.02 oe.

Amplifier and Integrator

The amplifier used to amplify the voltage from the pickup coils is a Millivac VS-67-A, which has a noise level equivalent to about $1 \mu v$ at the input terminals with a 8 ohm pickup coil connected to the input and the bandwidth of the amplifier set at 180 Kc. The gain of the amplifier is adjustable in steps to 10, 20, 40, 60 or 80 db and the bandpass is adjustable both at the top and bottom of the band.

A schematic diagram of the Miller integrator is shown in Fig. 12. It is built in a separate small box with switches to select integration constants and to select direct transmission or integration. The gain at 60 cps is about 0 db.

Stray Signal Compensation

A pair of compensation coils are connected in series with the pickup coils to compensate for part of the extraneous 60 cps signals which come from the pickup coils or any part of the system.

The compensation coils are so built that they can be rotated 360° and moved up and down through the axis of the Helmholtz coils to produce the right amount of voltage to reduce the extraneous signals to the lowest magnitude.

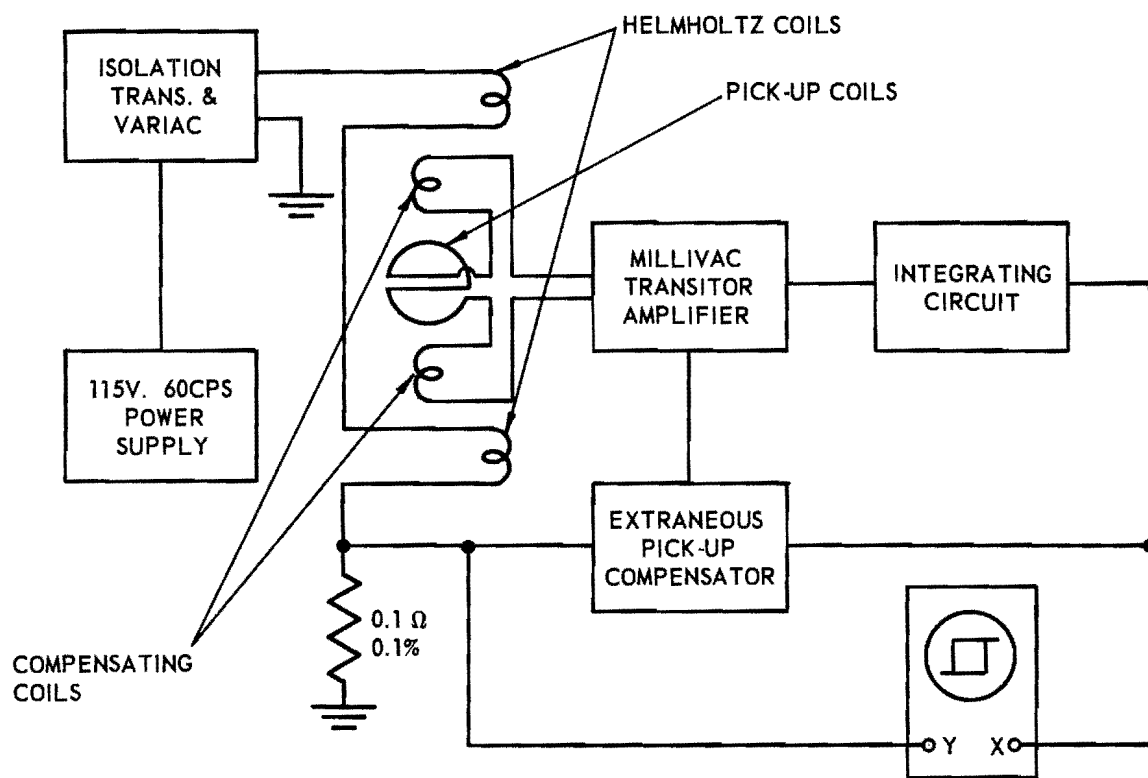


Figure 11. Block Diagram of Hysteresis Loop Tracer.

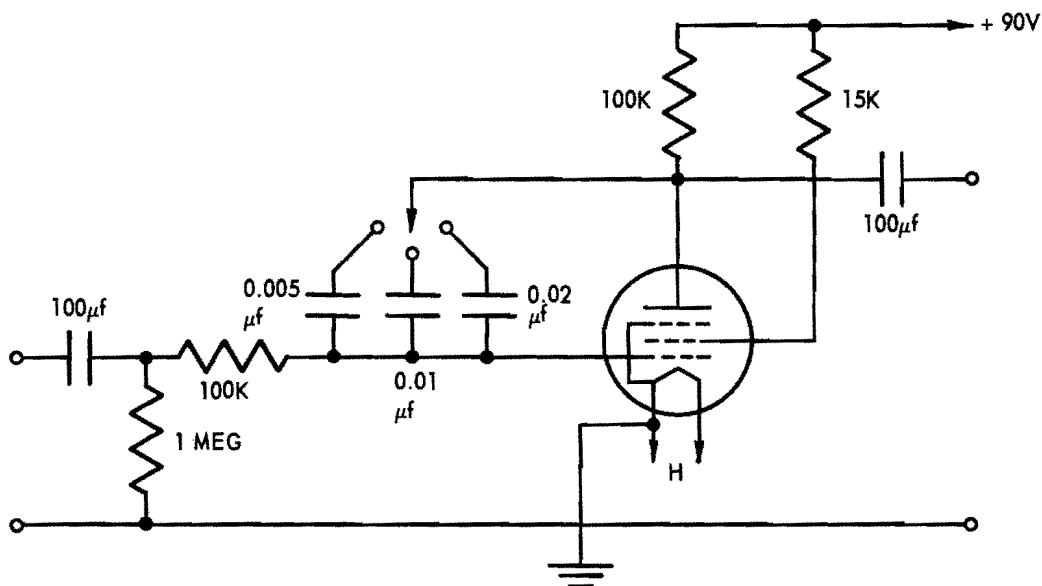


Figure 12. Schematic Diagram of Integrator Circuit.

The induced 60 cps voltage in the compensation coils is variable in magnitude but varies little in phase. It will cancel out either those in-phase or 180° out-of-phase components of the extraneous 60 cps signals. The remaining 90° out-of-phase stray signal component which is only a very small portion of the total stray signal, is nulled by a voltage produced by the compensator circuit. The compensator circuit, which is shown in Fig. 13, produces a voltage adjustable in both magnitude and phase.

Calibration and Measurements

The H axis is calibrated by passing a known direct current through the Helmholtz coils and measuring the field at the center with a Hall-effect probe. The value of the magnetization flux φ_m of a film corresponding to a certain distance, in volts, on the vertical axis of the oscilloscope, can be determined for all films of a certain diameter by calibrating with a fairly thick film for which the magnetization flux φ can be obtained from thickness and torque measurements of the magnetization.

Once it is calibrated, the accuracy of the H axis measurement depends only on the accuracy of equipment and of the reading processes. Since the calibration of the φ axis (magnetization flux) is effected by factors such as the geometry of the specimen and the relative position of the specimen to the pickup coils, a reduced absolute accuracy will be unavoidable; however, this will not effect measurements of M/M_s .

The hysteresis curve is a plot of the magnetization component parallel to the driving field versus the magnitude of the driving field. Some of the important parameters which are taken from the hysteresis curve are the coercive force, H_c , the saturation field, H_s , the remanent magnetization, M_r , and the saturation magnetization, M_s . These points are shown in Fig. 14.

If the drive field is reduced to a small fraction of H_c , the hysteresis loop becomes almost a straight line. The slope of this line is the initial susceptibility, χ , and if this line is extrapolated until it intercepts the line $M = M_s$, the value of H corresponding to the intercept is $H_i = M_s/\chi$. The value of the anisotropy constant, K, of a uniaxial permalloy film is given

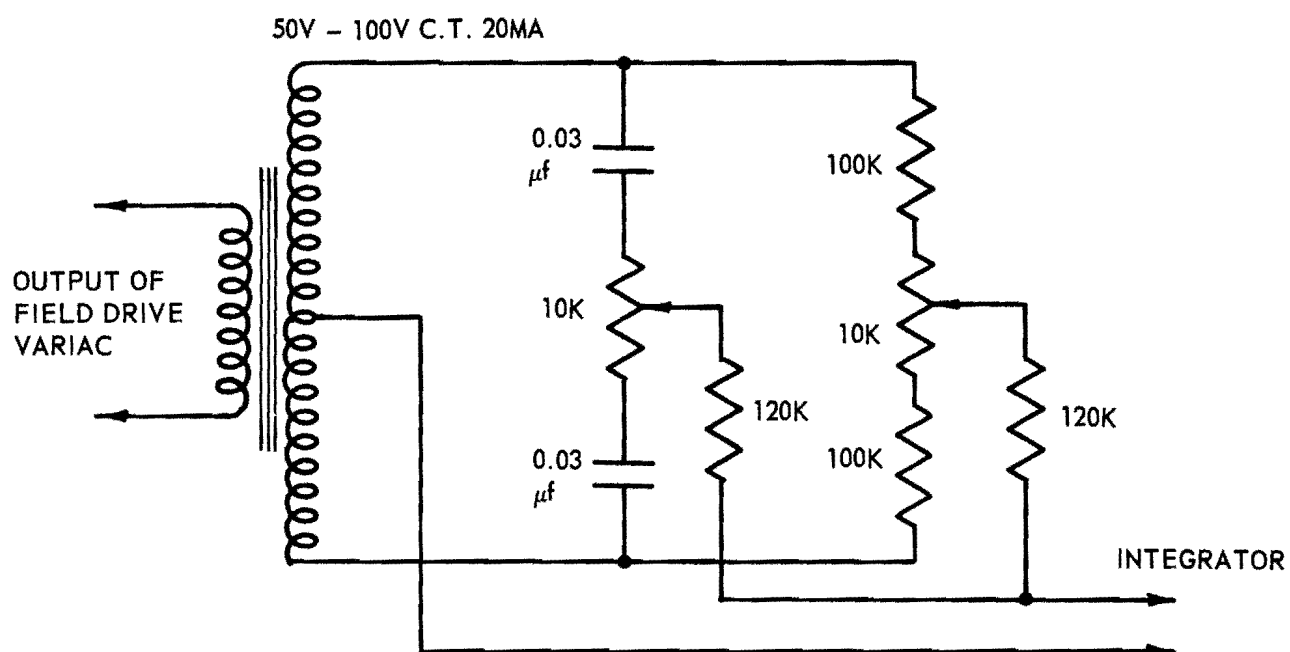


Figure 13. Schematic Diagram of Compensator Circuit.

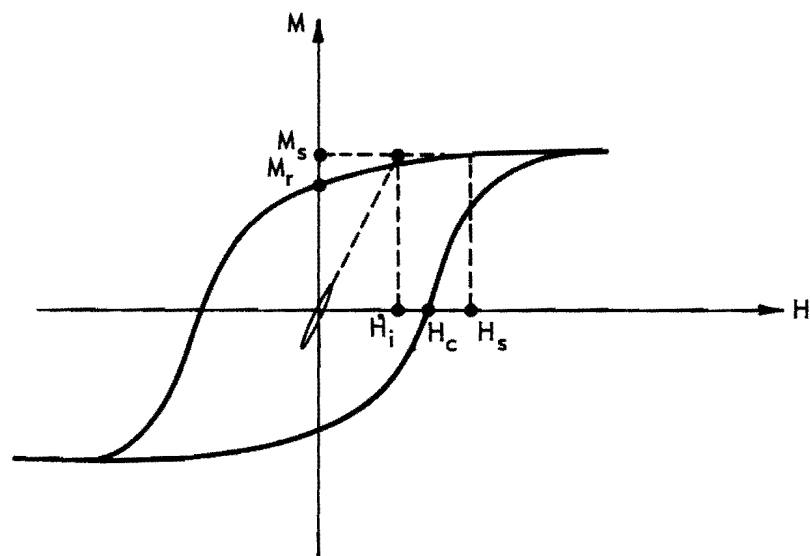


Figure 14. Large and Small Drive Hysteresis Loops.

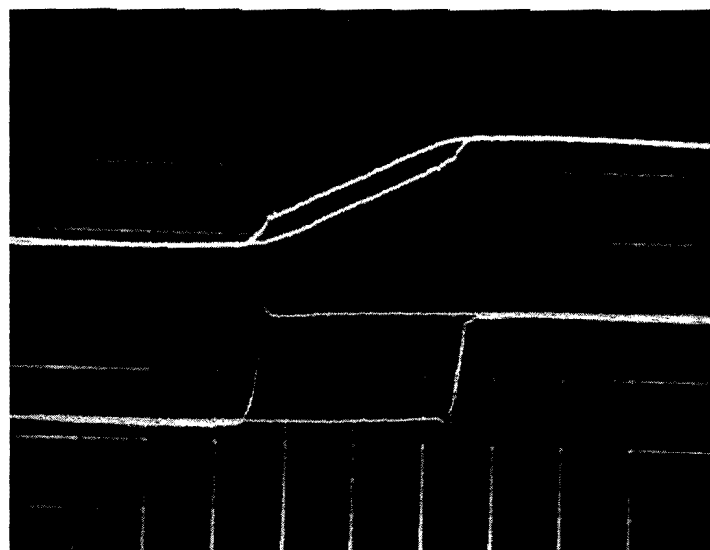
by $K = H_k M_s / 2$ where H_k is the value of H_i when the drive field is parallel to the hard axis. Since most thin permalloy films are anisotropic, the quantities described above depend on the orientation of the film.

Anisotropy Dispersion

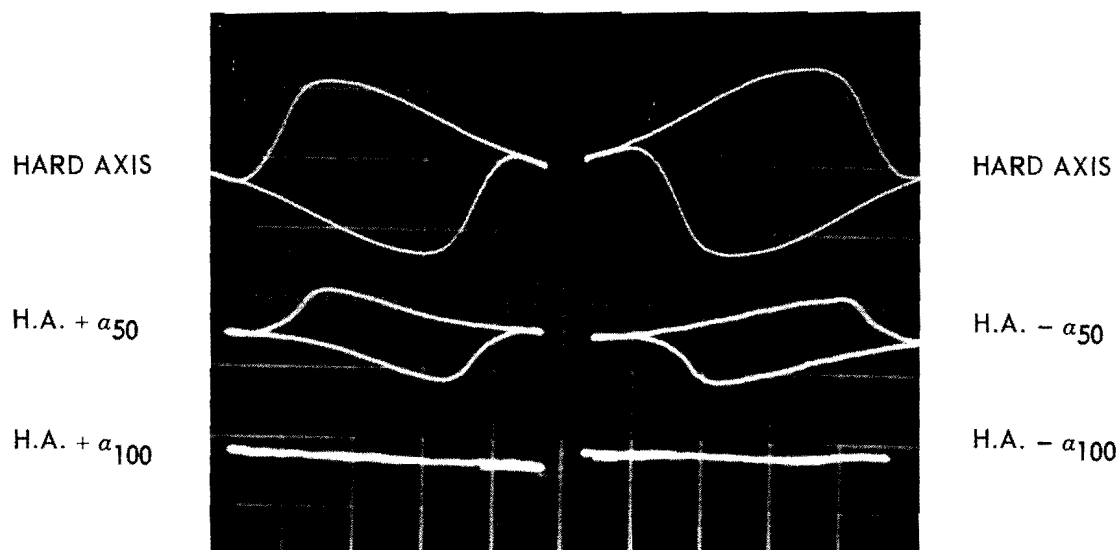
When an alternating drive field is applied along a direction that is almost parallel to the hard axis of a uniaxial permalloy film the magnetization will rotate in the plane of the film either clockwise or counterclockwise depending on whether the hard axis is slightly clockwise or counterclockwise of the drive field direction. The magnetization component parallel to the drive field is independent of the direction of rotation; however, the magnetization component perpendicular to the drive field, M_t , direction changes the sign (+ to -) when the direction of rotation of the magnetization changes. This transverse component can be measured by rotating the figure-eight pickup coil of the loop tracer by 90° . Examples of the type of display of M_t versus H seen on the loop-tracer oscilloscope are shown in Fig. 15.

The local direction of the hard axis of a permalloy film varies from one small region to another within the film. When the film is set so that the average hard axis is parallel to the drive field, half of the film has a local hard-axis slightly clockwise from the drive field and the local magnetization rotates clockwise while in the other half of the film the local magnetization rotates counterclockwise. In this case there is no average transverse flux and no signal from the pickup coil. If the film is rotated the signal from the pickup will increase and reach its peak when the film has been rotated enough that all of the local hard axes are either clockwise or counterclockwise of the drive field direction. If this angle is α_{100} , the local hard axes are dispersed over directions within α_{100} of the average hard axis.

Since the distribution of the local hard axes is usually Gaussian,¹³ a better parameter for specifying the amount of angular dispersion within a given film is the angle, α_F , which contains some fraction, F , of the hard axes of the film. The amplitude of the signal when the film is rotated by α_F



A. HARD - AXIS AND
EASY- AXIS HYSTERESIS LOOPS



B. TRANSVERSE LOOPS

Figure 15. Oscillographs of Dispersion Loops.

from the average hard-axis will be just F times the maximum amplitude since the fraction of the film rotating one way will be $(0.5 + F/2)$ and the fraction rotating the opposite way will be $(0.5 - F/2)$. The parameter used in this report's data is for $F = 0.5$.

A three dimensional plot or contour plot is needed to show the distribution of the local anisotropy in magnitude as well as direction. Torok has devised a technique for obtaining such plots.¹⁴

C. K. Kuo

B. KERR EFFECT APPARATUS

When a linearly polarized light is reflected by the surface of a magnetized sample, the plane of polarization is rotated through an angle which depends upon the magnetization direction in the sample.¹⁵ By applying this effect, which is known as the Kerr effect, the adjacent domains of a ferromagnetic sample will be revealed in contrasting intensity when the sample is illuminated with a plane polarized light and observed by reflection through a suitably oriented analyzer.¹⁶ The arrangement of the Kerr magneto-optic system for the direct observation of magnetic domains is shown in Fig. 16. The system utilizes the longitudinal Kerr effect in which the magnetization vector is in the plane of the film and parallel to the plane of incidence of the light beam. A collimated light after being polarized by a Glan-Thomson prism, illuminates the sample at about a 60° angle of incidence. The reflected light passes through another Glan-Thomson prism which acts as the analyzer and then through an objective lens to a television camera. The picture of the sample is shown on the screen of a television monitor.

The television system not only provides an easy way for observing the domain patterns of the sample, but it also enables the contrast of the domains to be increased electronically. The image of the sample was directly focused on the videcon tube of the television camera. The video

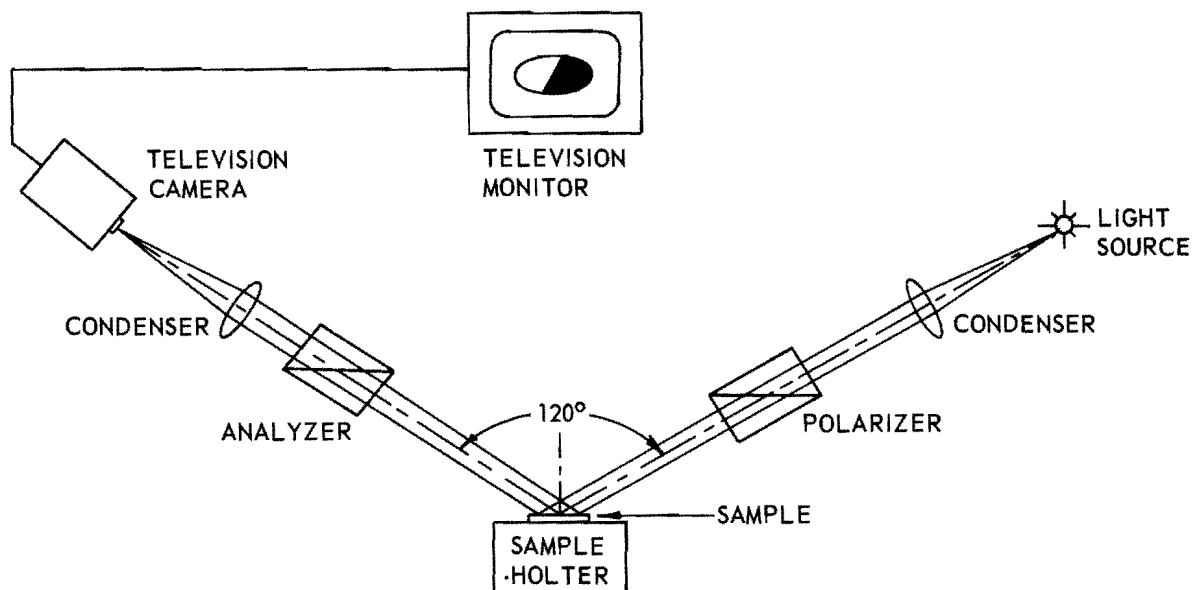


Figure 16. Diagram of Kerr Magneto-optic Apparatus.

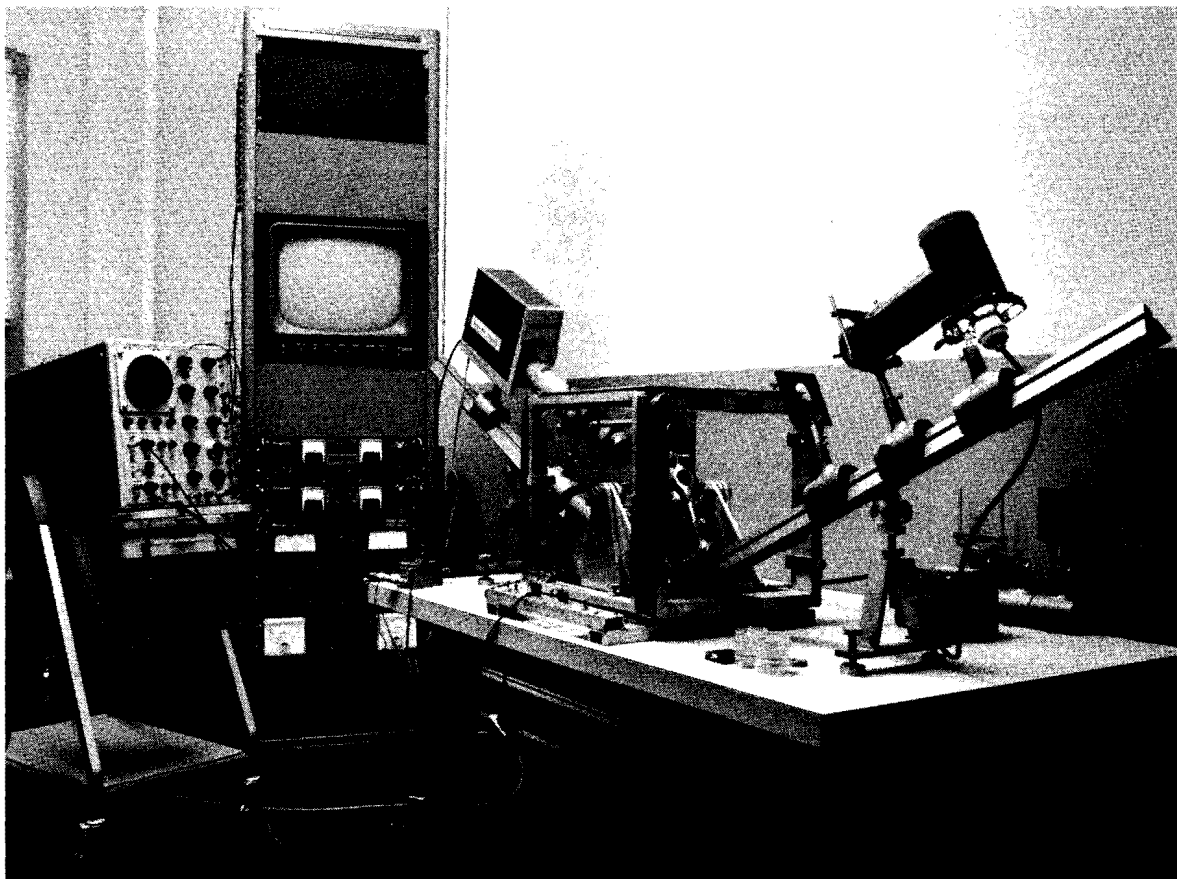


Figure 17. Photograph of Kerr Magneto-optic Apparatus.

signals from the television camera are amplified and some of the DC component is clamped to increase contrast.

The entire system is mounted on an optical bench which is adjustable in slope angle. The sample holder, which can be rotated 360° in the plane of sample, is a modified surplus Air Force Astro-Compass. A photograph of the Kerr apparatus is shown in Fig. 17.

The Kerr technique has the advantages of not disturbing the sample during the observation and of having less limitation on the thickness of the sample. It has, however, the disadvantage of relatively low contrast and magnification of domain structure. Some improvement in contrast can be achieved through employing a transparent ZnS dielectric layer deposited on the surface of the sample.¹⁷ This technique increases the Kerr rotation by multiple reflections within the dielectric film and reduces the intensity of the normal component by a proper choice of the dielectric film thickness. Another technique for improving contrast is to eliminate the ellipticity of the reflected light by adjusting the Kerr component to be in phase with the normal component. Monochromatic light is required for the latter technique.

The light source is a very important factor in determining the contrast and resolution of domain structure from a Kerr magneto-optic system. A collimated light beam will give better results and can be approximated by using a pinhole shutter at the source; however, the intensity is also reduced. A high intensity collimated beam can be achieved by using a laser as a source.

A 150 W high pressure mercury arc lamp and a Spectro-Physics Model 115 gas laser have been used at Georgia Tech for light sources. The laser is a very effective light source because the output beam from the laser itself is monochromatic, collimated, and polarized.

Much improvement in contrast and resolution of domain structure is observed when using the laser beam. Diffraction patterns appear on the screen however as a result of the coherent characteristics of the laser beam and defects in the lenses now in use. High quality diffraction limited lenses can be used to eliminate the diffraction patterns. It is possible that a high intensity laser beam is a solution to the contrast and magnification deficiencies of the magneto-optic technique.

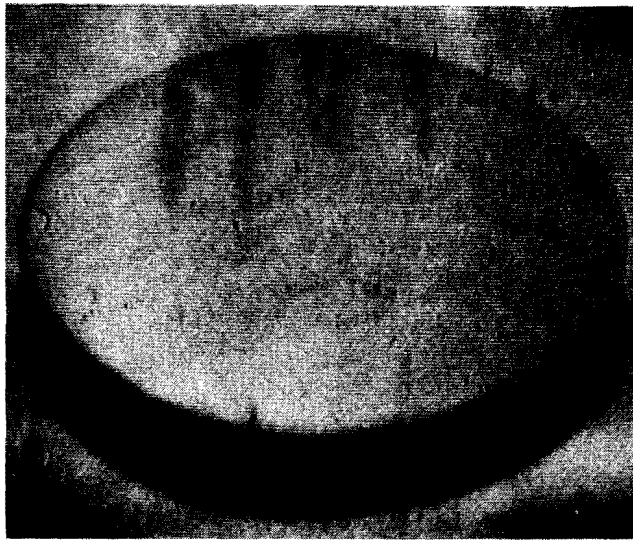
A pair of 18 inch square coils with sensitivity of 2.75 oe/amp produces a DC field in the longitudinal direction for the observation of the static domain structure. The direction of the field can be changed by a switch. The magnitude of the field can be adjusted smoothly by using the voltage adjustment in a Kepco power supply voltage regulating circuit. The direction control switch and the magnitude adjustment potentiometer are mounted on a small control box with a long cable. The stray field at the location of the sample is nulled to less than 0.002 oe by two pairs of 18 inch square coils.

A pair of Helmholtz coils 1.5 inches in diameter can be driven by a Rutherford pulse generator to produce a pulsing field in the longitudinal direction. The pulse field has a rise time of $0.5 \mu \text{ sec}$ and is adjustable both in duration and amplitude. An external triggering circuit is built to control the occurrence of single pulses. With this setup, we are able to study domain wall motion by applying a single pulse and observing the way the wall advances.

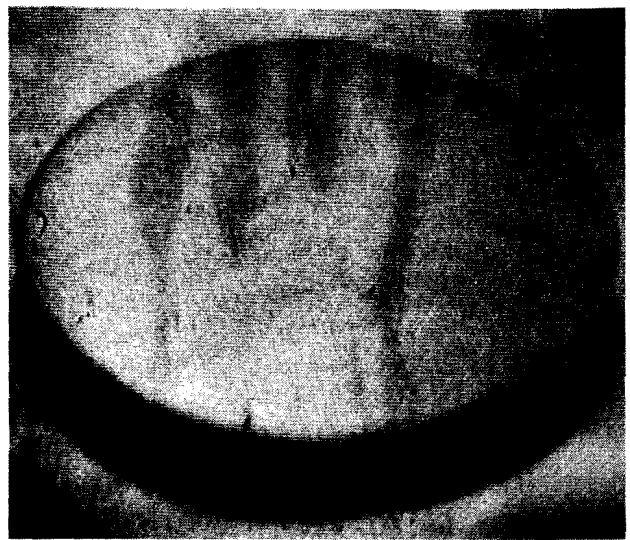
The Kerr apparatus is also very useful in measuring the tendency of domain walls to creep. The apparatus includes another pair of Helmholtz coils, 2 inches in diameter, driven by audio power in the transverse direction for the creeping experiments. The magnetization of a saturated uniaxial ferromagnetic film will reverse its direction by the slow movement (creep) of domain walls if an alternating field is applied along the hard direction, in the presence of a DC field in the easy direction opposite to the original magnetization direction, even though the total applied field is within the switching curve.

The mechanism of the creep phenomenon has been proposed by many authors and the measurements of the creep threshold have been presented in different papers, but so far there has been no generally accepted unique method to measure creep.^{18, 19, 20, 21, 22, 23, 24}

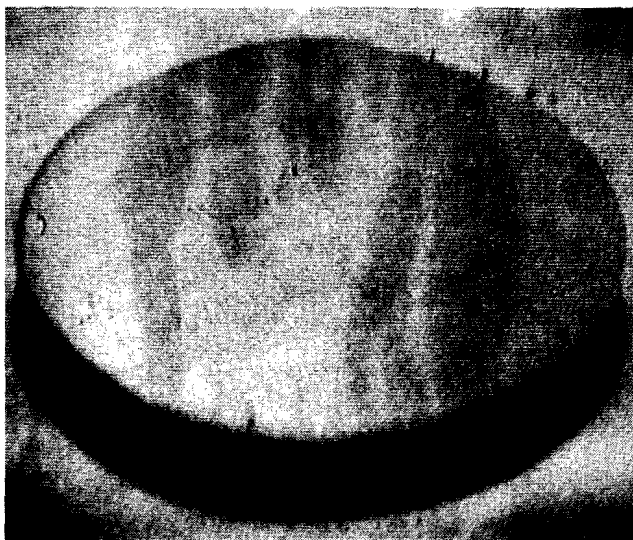
The creep threshold in this report is defined as the peak magnitude of the AC transverse field required to switch the magnetization of the film 25 per cent in 50 sec, in the presence of an easy-direction DC field of $0.2 H_k$ applied opposite to the saturation direction.



A



B



C



D

Figure 18. Kerr Photographs Showing Intermediate Stages in Switching by Domain Wall Motion.

Figure 18a-c shows a sequence of pictures taken of the Kerr apparatus video monitor as a film switched direction of magnetization by wall motion. Figure 18d shows a Kerr picture of a 5 mm film which was made using a 3 mm laser as a light source.

C. K. Kuo

C. FLUX REVERSAL MEASUREMENTS

The spontaneous magnetization in uniaxial permalloy films tends to lie in the plane of the film, due to the shape anisotropy, and parallel to one axis if viewed on a scale which is large compared to the crystallite size. If the film's thickness is less than about 10^{-4} times the easy axis dimension, the film can be changed from a single domain state with magnetization in one direction to a single domain state with magnetization reversed by applying a switching field, H_s , antiparallel to the original magnetization direction. As shown in Fig. 19, which was taken from Olson and Pohm,²⁵ the switching time, τ , can be greatly shortened by the presence of a transverse field, $H_{\perp}$.²⁶ The curves in Fig. 19 consist of two regions of H_s where the switching speed, $1/\tau$, is linearly related to the switching field but where the slope of the curve is different. In the higher field region the magnetization of the film rotates coherently while in the lower field region the magnetization rotates incoherently (i.e., the local magnetization vectors do not remain parallel).^{27,28,29}

Figure 20 shows a third region of H_s where the inverse switching time is linearly related to the switching field for many films.^{30,31} The slope of the curve in this region is much smaller (note the difference in vertical scale between Figs. 19 and 20). The film in this region of values for H_s reverses magnetization by domain wall motion. The velocity of the walls, V , can be described by

$$V = G(H_s - H_0) \quad (1)$$

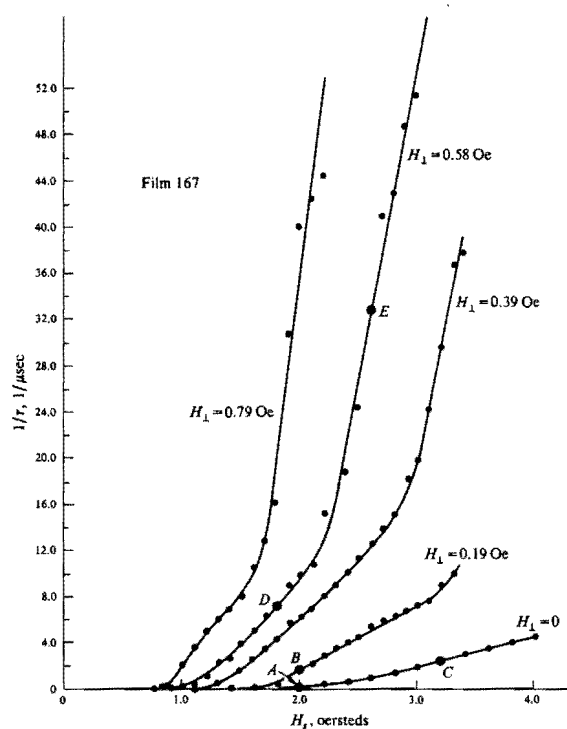


Figure 19. Inverse Switching Time vs. Drive Field for Permalloy Films.

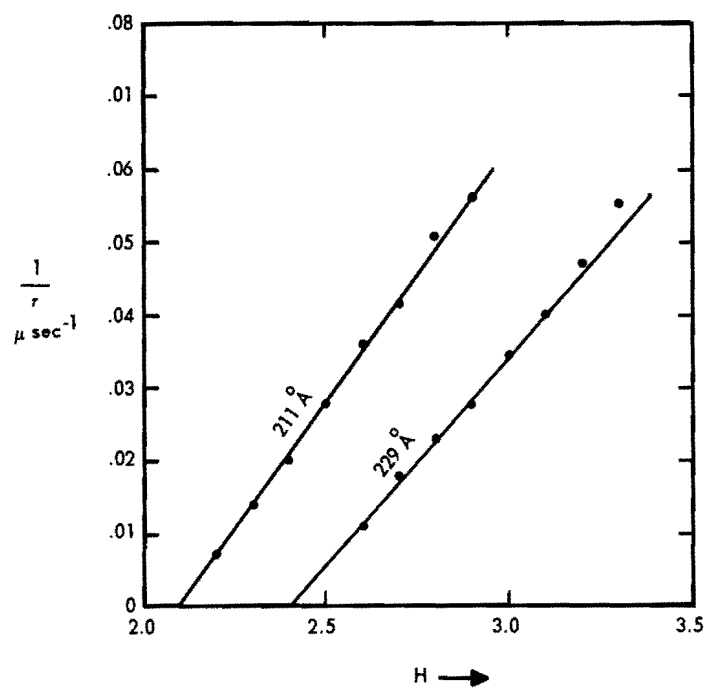


Figure 20. Inverse Switching Time vs. Drive Field for Permalloy Films in the Wall Motion Switch Range.

where G is wall mobility and depends primarily on the thickness of the film. If Eq. (1) is divided on both sides by the distance through which a wall moves during a flux reversal, X , the result is

$$\tau^{-1} = (G/X)(H_s - H_o) \quad (2)$$

The slope of curves such as those in Fig. 20 will vary from film to film because of different wall movement patterns and therefore different values for X . The value of X may be determined for a particular film by observing the domain wall movement pattern on a Kerr effect apparatus while switching the film with a series of $2\mu\text{sec}$.

The wall motion, Eq. (1), can be explained by two types of energy losses as the wall moves; one type in which the energy loss per unit area is proportional to the distance the wall moves ($E_1 = c_1 x$) and the other which gives a power loss per unit area in proportion to the velocity squared ($dE_2/dt = c_2(dx/dt)^2$). The energy per unit area put into the wall by the switching field is proportional to the distance the wall moves (actually to the volume a unit area sweeps through) and is given by $E = 2MHx$ or in terms of power, $dE/dt = MH dx/dt$. Since the power put into the wall must be equal to the power loss:

$$MH dx/dt = c_1 dx/dt + c_2 (dx/dt)^2 \quad (3)$$

If Eq. (3) is rearranged and V substituted for dx/dt

$$V = c_2^{-1} M (H - c_1/M) \quad (4)$$

Comparing Eq. (4) with Eq. (1) it seems that

$$G = c_2^{-1} M, \quad H_o = c_1/M \quad (5)$$

The second type of energy loss, the one that determines the value of G , is well understood. For films thicker than $700 \text{ \AA}$ this energy loss

is due mainly to eddy currents while for films thinner than $500 \text{ \AA}$ the loss is due mainly to intrinsic damping.^{30, 31} Figure 21 shows the value of G calculated from theory and experimental values for permalloy films as a function of film thickness.

The mechanism of the energy loss which is proportional to the distance the wall moves and independent of the velocity is not well understood. It is known that transverse fields, particularly alternating fields, reduce the value of c_1 or H_0 and let the wall move at values of H_s less than the value of H_0 for no transverse field (domain wall creep). This energy loss is probably due to microscopic inhomogeneities in the film.

For observing flux reversals that take place in periods from 1 nsec to $10 \mu\text{sec}$ it is necessary to put the film in a transmission line, either a strip or coaxial type, and sense the flux changes with a single turn pick-up.^{25, 26, 27, 28} A pulsed field with a rise time of 1 nsec is obtained by discharging several hundred feet of coaxial cable through a coaxial mercury-wetted relay.

For studying flux reversals which take place by wall motion in $10 \mu\text{sec}$ to $100 \mu\text{sec}$ it is possible to use a lumped constant transmission line made of a two inch diameter coil of wire, 8 turns/in with 120 picofarad capacitor from each turn to an external ground buss. This type of line will produce a pulsed field of about 3 oe with $0.5 \mu\text{sec}$ rise time when driven by a 50 volt, 1 amp pulse generator such as the Rutherford B7B and when termination in 50 ohms resistance. A multiturn pickup coil such as the one used for the hysteresis loop tracer described in Section III-A is used to sense the flux changes in the film. Other external coils can be used to reset the magnetization, to supply transverse fields, and to cancel the earth's field.^{30, 32}

For flux reversals which take place in more than $100 \mu\text{sec}$, which generally would be by domain wall creep, the Kerr apparatus can be used as described in Section III-B. Also a small-spot Kerr apparatus with a photomultiplier tube can be used to measure domain wall velocities.³¹

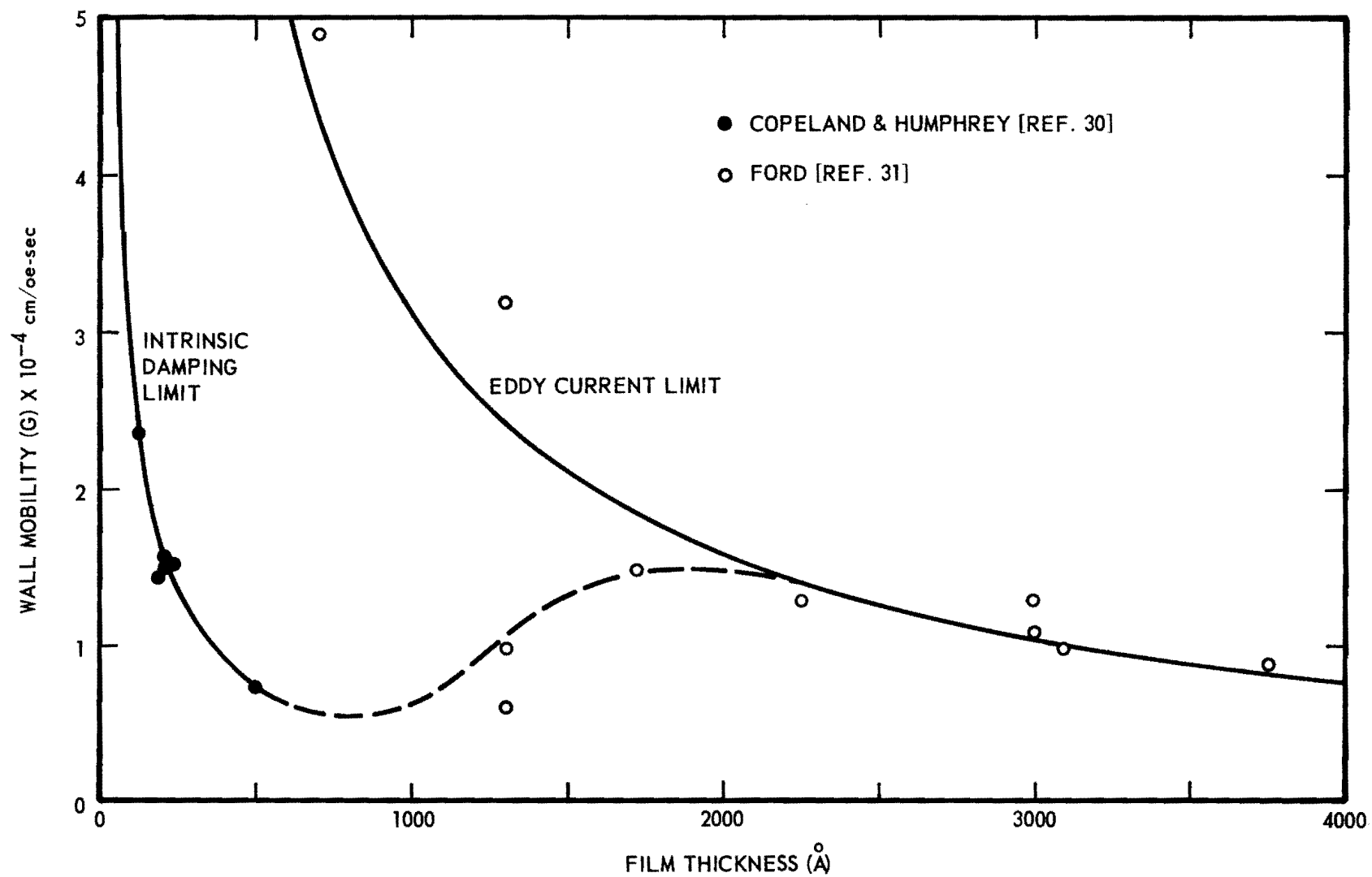


Figure 21. Mobility Factor of Domain Walls as a Function of Film Thickness.

For a review of flux reversal experiments and theory see the appropriate chapters of the books by Soohoo³³ and Prutton.³⁴

J. A. Copeland

D. TORQUE MEASUREMENTS

Torque measurements on thin films are used to determine several important properties. An absolute determination of the spontaneous magnetization is possible by making measurements about an axis lying in the plane of the film and normal to the field direction. Torque measurements made on films with the rotation axis normal to the film and with the field vector lying in the plane of the film are used to determine angular anisotropies and to evaluate anisotropy constants. These measurements are greatly facilitated by the use of a self balancing torque magnetometer. The magnetometer and some of the techniques of measurement are described briefly in this section.

Measurement of Saturation Magnetization

The saturation magnetization is measured by suspending the film vertically in a horizontal magnetic field and then making torque measurements about the vertical axis. A torque is exerted on the film essentially because of the shape anisotropy of the thin layer. If the orientation of the film is such that the plane of the film makes an angle of 45° with the applied field, the energy may be written:

$$E = -MHV \cos \theta + (2\pi M^2 + K)V \sin^2(\pi/4 - \theta) \quad (1)$$

where θ is the angle between the magnetization M and the applied field, H , V is the volume of the film, and K is an anisotropy constant. The torque acting on the film is

$$L = MHV \sin \theta \quad (2)$$

When equations (1) and (2) are combined after minimizing the energy in (1), the following expression is obtained

$$\frac{L}{H} = - \frac{MV}{4(2\pi M^2 + K)} \left(MH - \left[M^2 H^2 + 8(2\pi M^2 + K)^2 \right]^{1/2} \right) \quad (3)$$

The spontaneous magnetization may be determined from an extrapolation of the experimental $\frac{L}{H}$ vs. H curves to obtain the zero field intercept $\left(\frac{L}{H} \right)_0$. It is seen from equation (3) that the spontaneous magnetization may be computed from this intercept according to

$$M = \frac{\sqrt{2}}{V} \left(\frac{L}{H} \right)_0 \quad (4)$$

A more detailed discussion of the techniques involved appear in the papers by Neugebauer³⁵ and Livesay and Scheibner³⁶ who have studied the dependence of the magnetic properties of thin nickel films on thickness.

Determination of Anisotropy

Torque measurements have long been used to evaluate anisotropic behavior in magnetic materials.^{37, 38, 39} A uniaxial anisotropy in the plane of thin films is usually expressible in the form

$$E_k = K \sin^2 \theta \quad (5)$$

where E_k is the anisotropy energy per unit volume, k the anisotropy constant and θ the angle between the magnetization vector, M , and the easy direction. When a magnetic field, H , is applied at an angle, θ , with the easy direction, the magnetic energy of the film is

$$E = -H MV \cos(\theta - \phi) + KV \sin^2 \phi \quad (6)$$

For fields much greater than the anisotropy field, H_k , ($H_k = \frac{2K}{M}$) $\theta \sim \phi$ and the torque $L = \frac{\partial H}{\partial \phi}$ is approximately

$$L = -KV \sin 2\theta \quad (7)$$

The amplitude of the L vs. θ curves may therefore be used to evaluate the anisotropy constant, K , provided V is determined independently.

An anisotropy energy involving a $\sin^4 \phi$ term results in a $L \sim \sin 4\theta$ yielding a biaxial anisotropy. A unique approach for determining H_k from measurements of the torque with H at 90° to the easy axis has recently been described by Robinson.⁴⁰

Torque Measurements on Permalloy Films

Torque measurements are made with a system capable of providing a known counter-torque to give rotational equilibrium at some desired orientation. Our torque measurements have been made with a system which uses either of two methods for applying the counter torque. The simpler method involves a fine torsion fiber while a more convenient arrangement is obtained by using a galvanometer coil and sensing circuit to provide a self-balancing method. Both methods have been used for torque measurements on permalloy films. Saturation magnetization values of approximately 700 emu have been determined and all films examined, including the oxidized ones, show only a uniaxial anisotropy.

B. R. Livesay

E. LORENTZ MICROSCOPY

Since the pioneering work by Fuller and Hale⁴¹ in 1960 numerous studies have been made of domains and domain walls in thin ferromagnetic films by out-of-focus electron microscopy (Lorentz microscopy). Recently at the 1965 Intermag Conference, Cohen⁴² summarized the advantages of the technique and showed that it could be used for quantitative measurements of certain magnetic properties which agreed well with microscopic measurements made on other films prepared simultaneously with the

films for Lorentz studies. Thus, the technique offers high resolution, it provides an unequivocal identification of the local magnetization direction and it permits correlation studies between the magnetic structure and the crystallographic structure of the film. Quantitative measurements that can be made for comparison with magnetic properties of simultaneously prepared films are: (1) the Curie temperature; (2) anisotropy field H_k by the standard hysteresis method or by the Feldtkeller technique;⁴³ (3) wall motion by labyrinth propagation⁴⁴ and by creep;⁴⁵ and anisotropy dispersion by the Crowther⁴⁶ and Torok⁴⁷ techniques.

Basically the technique involves imaging the pattern formed by the magnetic deflection of electrons passing through different regions of the thin film. In a simple picture of the process the Lorentz force on the electron, $-(e/c \vec{v} \times \vec{B})$ will cause an angular deflection proportional to the film thickness and magnetization and inversely proportional to the velocity of the electrons. In Fig. 22 is shown schematically an array of domains separated by 180° domain walls. For a large diameter parallel beam it is seen that two cases can be distinguished, the divergent case where the wall is imaged as a deficiency of electrons and the convergent case where the wall is imaged as an excess of electrons. Such walls appear then in Lorentz micrographs as light and dark bands (Fig. 23a). Fine structure within a domain is also resolved as for example the magnetization ripple shown in Fig. 23b. The ripple is normally associated^{48, 49, 50} with random local variations in the direction of the easy axis as a result of the magnetocrystalline anisotropy⁵¹ or magnetostriction anisotropy⁵² of individual crystallites. Since the mean magnetization direction in the domain is always perpendicular to the ripple it is possible to map accurately the magnetization directions in the film. Cross-tie walls proposed by Huber, Smith and Goodenough⁵³ and studied by them with the Bitter technique show clearly in Lorentz micrographs of thin permalloy films (Fig. 23c). In the figure the dot on the wall which is the same color is the Bloch line in the position where flux closure is along the same direction as the magnetizations of the domains on either side of the wall. The cross-ties which are the opposite color from the wall,

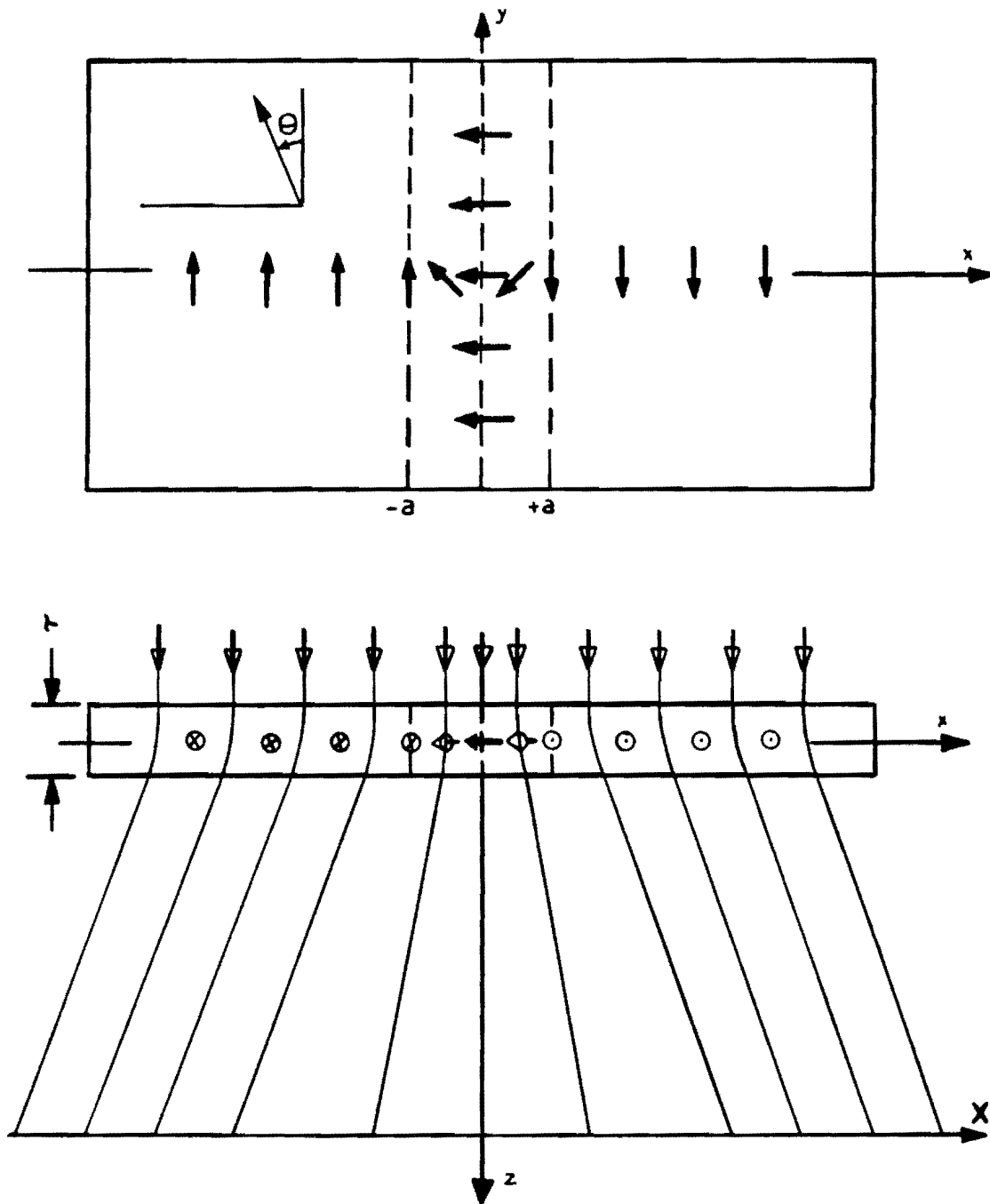
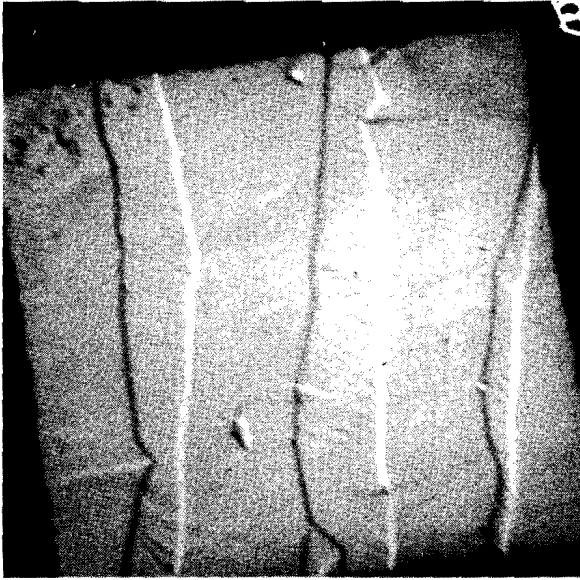
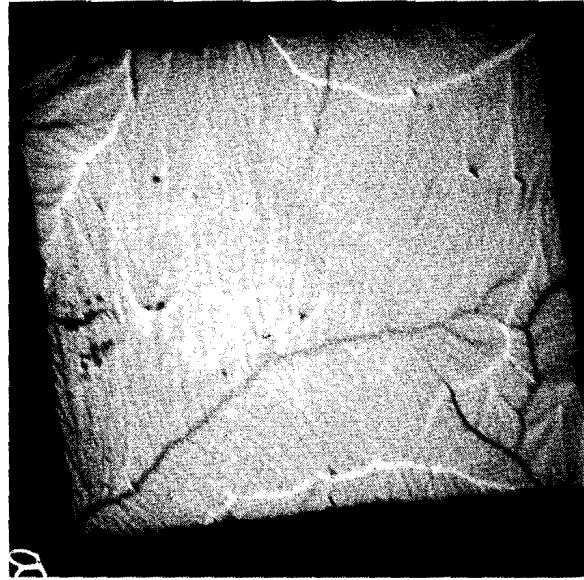


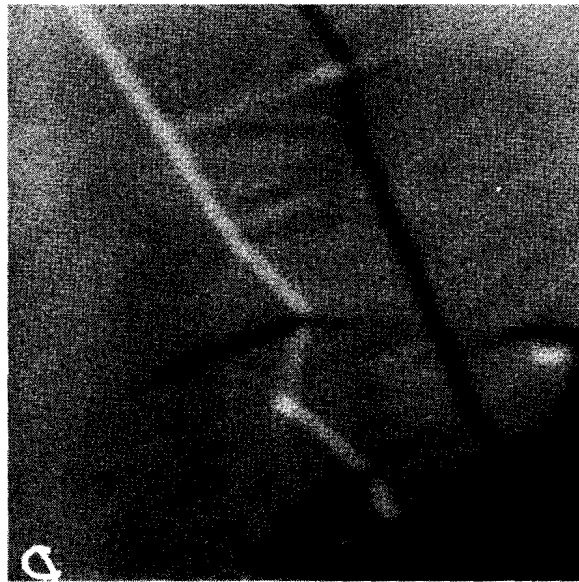
Figure 22. Schematic of Lorentz Deflection by a Magnetic Film.



A



B



C

Figure 23. Typical Lorentz Micrographs.

occur at Bloch line positions where the flux closure is opposite to that of the domain magnetizations.⁵⁴

Several modes of operation of the electron microscope are possible in Lorentz microscopy. In the Foucault method the objective aperture is positioned off the optical axis so that it will intercept electrons deflected toward it from some domains and not those from other domains. As a result there will be intensity contrast between entire domains of differing magnetization directions.⁵⁵ This technique is similar therefore to the dark-field technique normally used in electron microscopy.⁵⁶ The more conventional methods of Lorentz microscopy utilize the imaging of an out-of-focus pattern either with the objective lens off and pole piece removed or with the objective lens excited. In the first method as described by Cohen⁵⁷ the specimen is inserted in place of the intermediate lens and the projector lens is operated to magnify the image. Somewhat higher magnification and greater convenience in examining in-focus images of the specimen is afforded by the second method. This is the method we have used.

In our study, observations of magnetization conditions in permalloy films under the influence of externally applied fields at different angles with the easy direction was desired. No suitable commercial holder was available and the Cohen type attachment didn't provide high enough magnification or the capability of imaging the specimen plane in exactly the same region where magnetic features were observed. Therefore a specimen holder was constructed for the RCA EMU-2 electron microscope whose radial field due to the objective lens is negligible in the region of the specimen. The holder, shown in Fig. 24, accommodates a standard microscope grid mounted on a rotatable tube so that its position is slightly above the normal specimen position and two pairs of Helmholtz coils, one for magnetizing the sample (A-A) and one for beam compensation (B-B). When the field due to coils A-A is increased the image-forming beam is deflected; coils B-B provide a field in the opposite direction to compensate for this effect. Fields of up to 50 oersteds can be applied with a field variation over the sample of less than 10 percent as measured by a Bell Hall effect gauss-meter. The power supply for the unit provides the required magnetizing

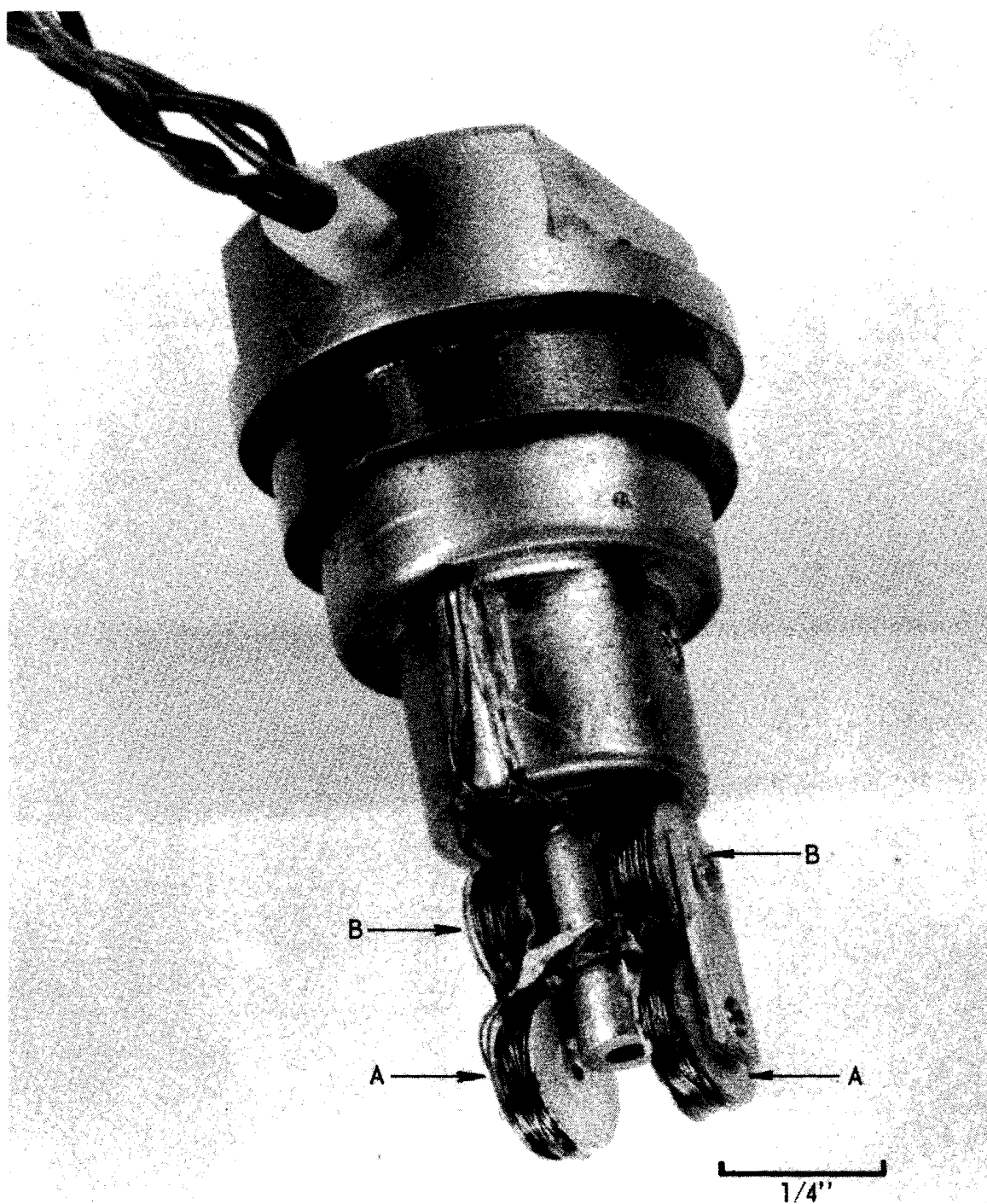


Figure 24. Lorentz Specimen Holder for RCA-EMU-2 Electron Microscope.

current from dry-cell batteries with automatic compensation for beam deflection. A panel meter was calibrated directly in oersteds with the Bell gaussmeter.

It is planned to continue the development of the instrumentation for Lorentz microscopy. A similar holder to that described will be made for the RCA EMU-3. The double-condenser system of this microscope provides a smaller apparent electron source resulting in sharper domain patterns. The higher accelerating voltage which is available will also enable the examination of thicker films. In the holder itself an additional set of coils orthogonal to the magnetizing coils will permit the application of a. c. fields for creep studies. Rotation of the specimen with respect to the applied fields will be possible without removing the holder as now necessary.

E. J. Scheibner
J. L. Brown

IV. EVALUATION OF RESULTS

The purpose of this chapter is to discuss the results of the experimental study of films made by vacuum deposition since the results of the preliminary study of films made by chemical vapor deposition are included in Section II-B.

The primary objective of the study of vacuum deposited films was to investigate the effect of post-deposition oxidation. Eighty-four films were made without post-deposition oxidation to determine the magnetic characteristics of normal films made in the vacuum system used (see Section II-A for details on deposition system). Another 110 films were made with post-deposition oxidation at a substrate temperature of 250°C . The total number of films includes 120 films 5 mm in diameter deposited in thirty groups of four onto separate Corning 7059 substrates for use in the hysteresis loop tracer (III-A) and Kerr effect apparatus (III-B). With each group of four circular films, a $1/16$ inch $\times$ $5/8$ inch film for resistance monitoring (R-1 through R-30) and a $1/16$ inch $\times$ 2 inch for interferometer thickness measurements were deposited. During 14 of the 30 depositions, films E-1 through E-14 were deposited onto a carbon film stretched over an electron microscope grid for use in the Lorentz microscope (III-E).

The experimental data is tabulated in the appendix. The rest of this section will discuss different properties of films which were observed.

Effect of Substrate Orientation

The Corning 7059 glass substrate surfaces have a slight waviness which can barely be seen with the eye when looking at a reflection from the surface from about a 20° angle of incidence and which appears to be parallel to two sides of the square substrate. The waviness has an apparent wavelength of about 10^{-2} cm. The amplitude has not been measured but is obviously larger in some substrates than others.

The main effect of the anisotropic substrate surfaces seems to be a dispersion of the average easy axis between films that were deposited simultaneously. The angle between the edge of substrate and the magnetic easy axis is shown in Table III in the appendix under the heading θ . In all groups of four films that were deposited simultaneously and not oxidized the angle θ varies by amounts on the order of 5° while the dispersion within the films, α_{50} , is smaller by a factor of about five to ten. The angle θ appears to be random with respect to substrate position in the mask which implies the variation of θ is not an angle-of-incidence or stray effect. Also the average value of H_k for films with the substrate waviness parallel to the easy axis is 10 percent higher than films with the two directions perpendicular. While this condition was discovered late in the project and was not investigated thoroughly, it may be largely responsible for the variation in the magnetic properties within the groups of films that were deposited simultaneously.

Effect of Substrate Temperature

The non-oxidized films deposited at a substrate temperature of 250°C (1-4, 13-16, 45-48) had H_c/H_k ratios which ranged from 0.5 to 0.9 and values of easy-axis dispersion, α_{50} , that ranged from 0.5° to 2.5° . The films deposited at lower temperatures (17-20, 33-44) generally had lower values of H_c/H_k and the group deposited at room temperature (33-36) had larger values of α_{50} (3° to 11°). The films deposited onto substrates at 350°C and 450° (5-12, 21-24, 29-32) showed a little magnetic anisotropy and had larger values of H_c , 10 oe to 25 oe. Since a substrate temperature of 250° seems to give the best results, this value was used for films 49 through 120.

Post-Deposition Oxidation

The primary effect of the post-deposition oxidation was to increase the dispersion of the local easy axis directions within the film. The films which were exposed to dry oxygen at 0.1 Torr after deposition and while the substrates were still at 250°C had values of α_{50} which generally ranged from

10° to 30° . Most of these films (65-80, 85-120) were inverted ($H_w > H_k$) with values of H_w/H_k which ranged from 0.6 to 4.0. The hysteresis loops of the post-oxidized films would suddenly open up as the film was rotated in the hysteresis loop tracer so the drive field was parallel to the hard-axis of the film. Both the inverted characteristic and the open hard-axis hysteresis loop can be explained as effects of the easy-axis dispersion.^{58, 59} Figure 25 shows the value of H_c as a function of angle between the hysteresis loop tracer drive field and the film's easy-axis and the remanent magnetization as a function of the same angle for film 69. Figure 26 shows a sequence of hysteresis loops at different angles for an oxidized film.

The deposition and oxidation conditions were varied during the preparation of films 81 through 120 and E-5 through E-14 to determine the effect of the applied field during oxidation and substrate cooling. These conditions are summarized in Table II in the appendix. The effect of the oxide observed to form on the permalloy evaporant during an oxidation on the next film was also studied. None of these factors had appreciable influence on the magnetic characteristics.

The films which were deposited in layers and oxidized between the deposition of each layer (65-84) showed essentially the same magnetic characteristics as the single layer oxidized films (85-116). Films 53 through 64 were deposited in layers with 5 minutes exposure to 10^{-6} Torr vacuum between each layer. These films have essentially the same characteristics as single layer, unoxidized films with the same total thickness. It appears that evaporation by layers or evaporation and oxidation by layers produced no change in magnetic characteristics from that observed in single layer films.

Domain Wall Motion

The techniques used to study domain motion were described in Sections III-B and III-E. The creep threshold was normally measured on the Kerr apparatus using a 10 kc alternating transverse field. The creep threshold seemed to be fairly constant over a range of transverse field frequencies from 10 cps to 100 kc. Even though most of the oxidized films were inverted,

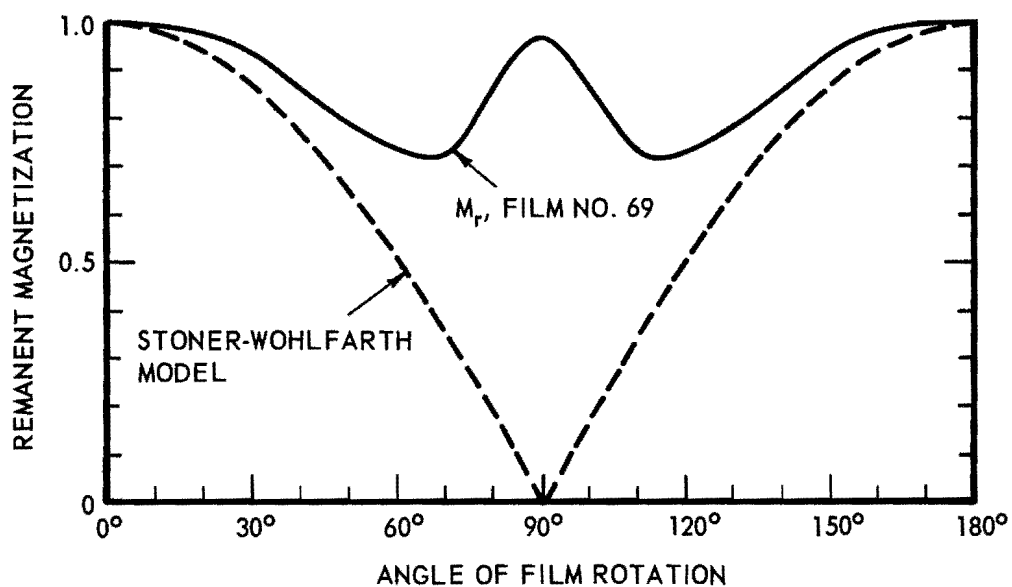
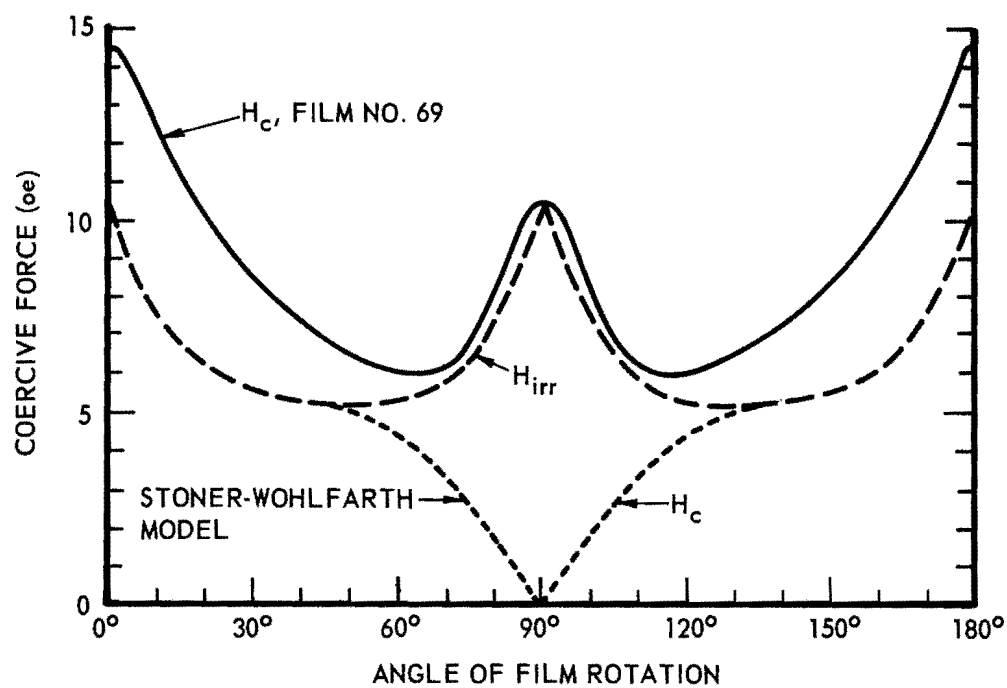
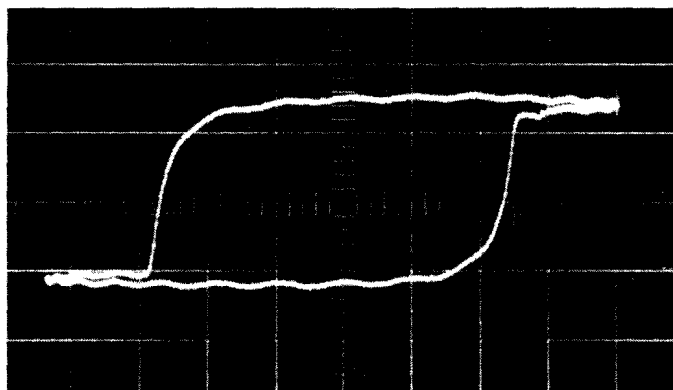
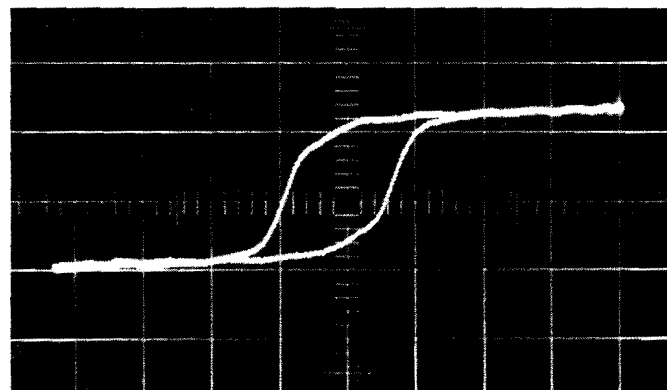


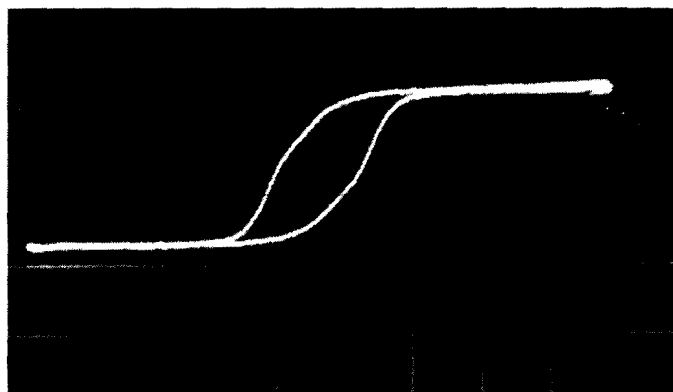
Figure 25. Plots of Coercive Force (H_c) and Remanent Magnetization (M_r) as a Function of Angle Between the Drive Field of the Loop Tracer and the Easy Axis.



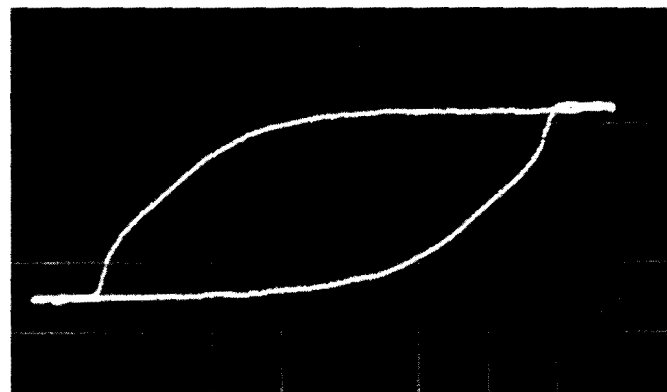
A. EASY DIRECTION



B. ROTATED 45°



C. ROTATED 62°



D. ROTATED 90°

Figure 26. Hysteresis Loops of a Post-deposition Oxidized Film at Various Angles Between the Drive Function and the Easy Axis.

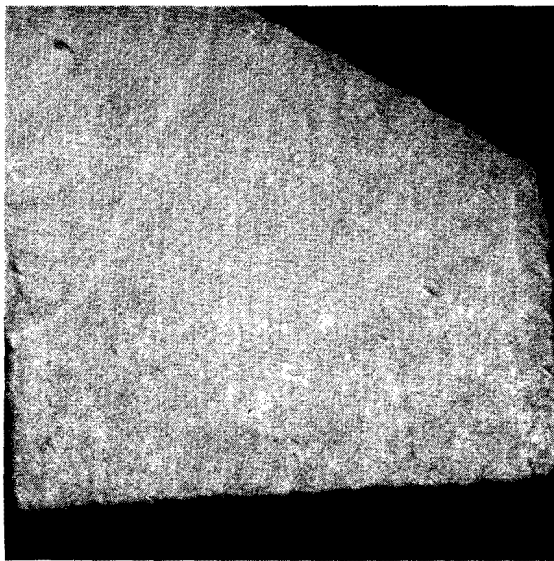
the creep thresholds for these films were in the same range as the creep thresholds observed in the films which were not oxidized.

The Lorentz micrograph shown in Fig. 27a shows the type of dispersion regions that form in a film with high easy-axis dispersion. These dispersion regions, which are on the order of 10^{-3} cm wide in the easy direction and much longer in the hard direction, tend to lock up the magnetization and prevent flux reversal until drive fields several times H_k are applied. As can be seen in Fig. 27b when a field is applied parallel to the easy axis, dispersion regions start to rotate in different directions and set up demagnetizing fields that oppose further rotation. Another effect of easy-axis dispersion is shown in Fig. 27c and 27d. The single wall shown in Fig. 27c becomes a complex structure when a 2.7 oe field is applied parallel to the wall and to the easy axis. A dc transverse field eliminates the locking up effect by causing all areas to rotate the same way.

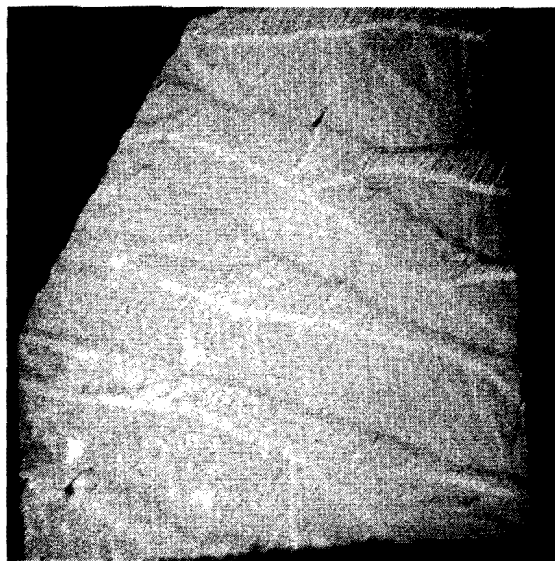
One possible cause of creep is the movement of cross-ties along a domain wall. This is shown in Fig. 28 as a sequence of four pictures taken as a transverse was applied and slowly increased. The walls do not move because there is no field applied parallel to the wall. As the field increases the segments of the two cross-tie walls with magnetization in the center of wall parallel to the field fade out and begin to grow, at the expense of the segments with center magnetization antiparallel to the field, by the movement of the cross-ties or Bloch lines. The single white wall on the other side of the microscope grid wire changes from a pure Neel wall at $H = 3$ oe (b) to a cross-tie wall at 4 oe(c) and movement of the cross-ties can be seen at 5 oe (d).

Aging

The values of H_w and H_k increased with age for all films. Figure 29 shows aging effects in three films which are typical of most of the films studied. The easy-axis dispersion, α_{50} , generally decreased. The aging effects were more pronounced in the thicker films and in films made at higher substrate temperatures. The films were not overcoated and were kept in

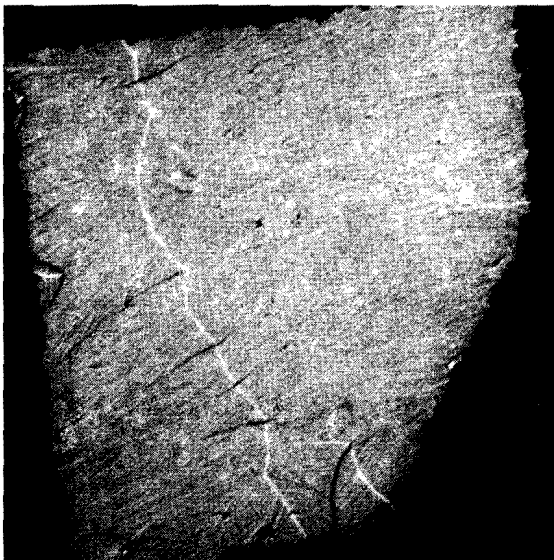


A. $H = 0$ oe

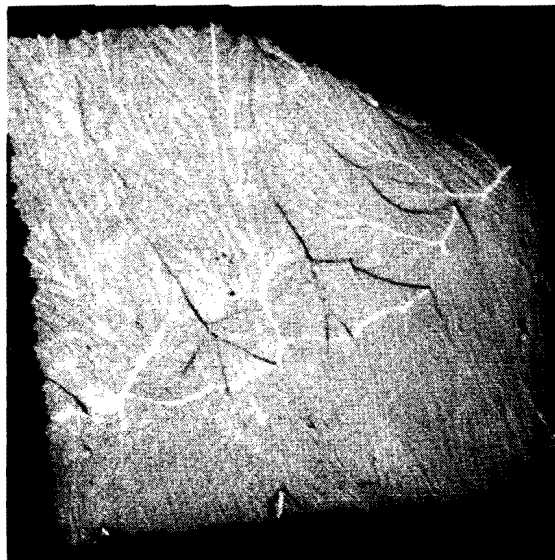


B. $H = 3.3$ oe

FILM E - 12



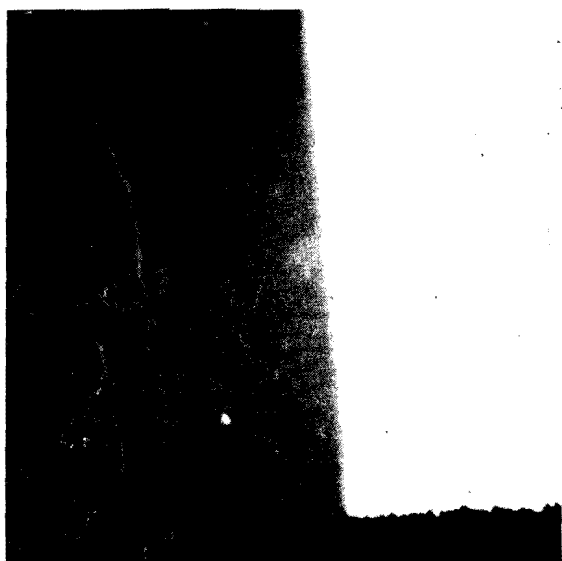
C. $H = 0$ oe



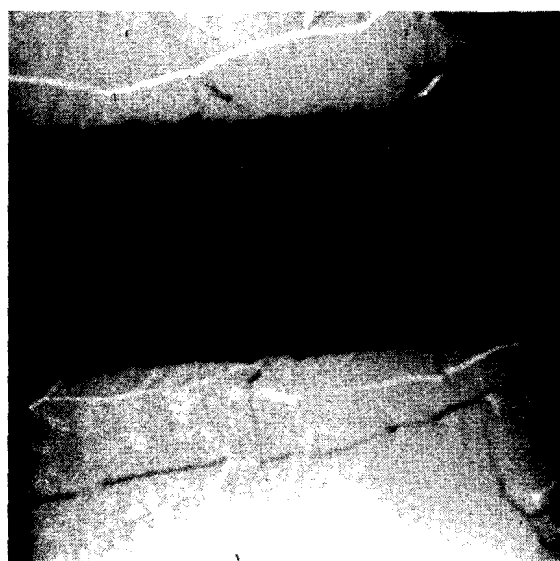
D. $H = 2.7$ oe

FILM E - 9

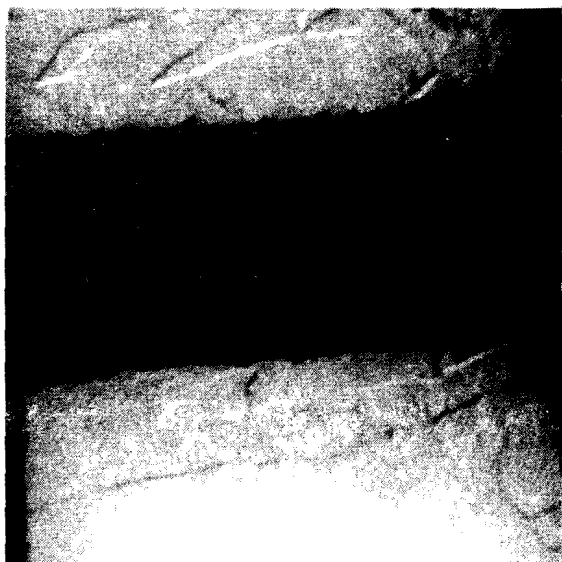
Figure 27. Lorentz Micrographs Showing the Effects of an Applied Field in the Easy Direction.



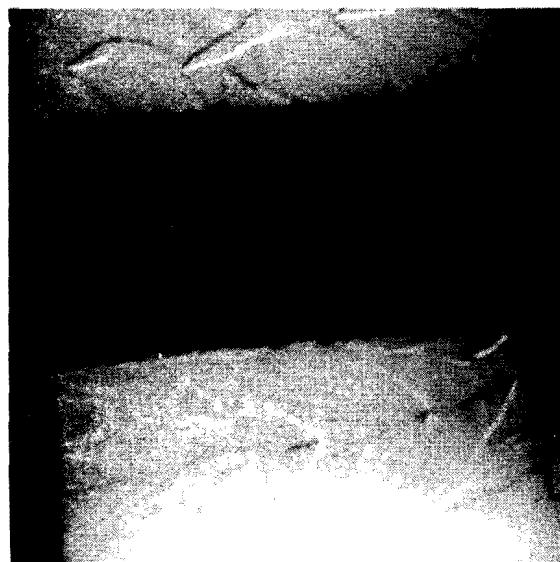
A. $H = 0$ oe



B. $H = 3.0$ oe



C. $H = 4.0$ oe



D. $H = 5.0$ oe

Figure 28. Lorentz Micrographs of Cross-tie Wall in a Permalloy Film, showing Movement of Cross Ties when Field (H) is Applied Perpendicularly to Wall and Easy Axis.

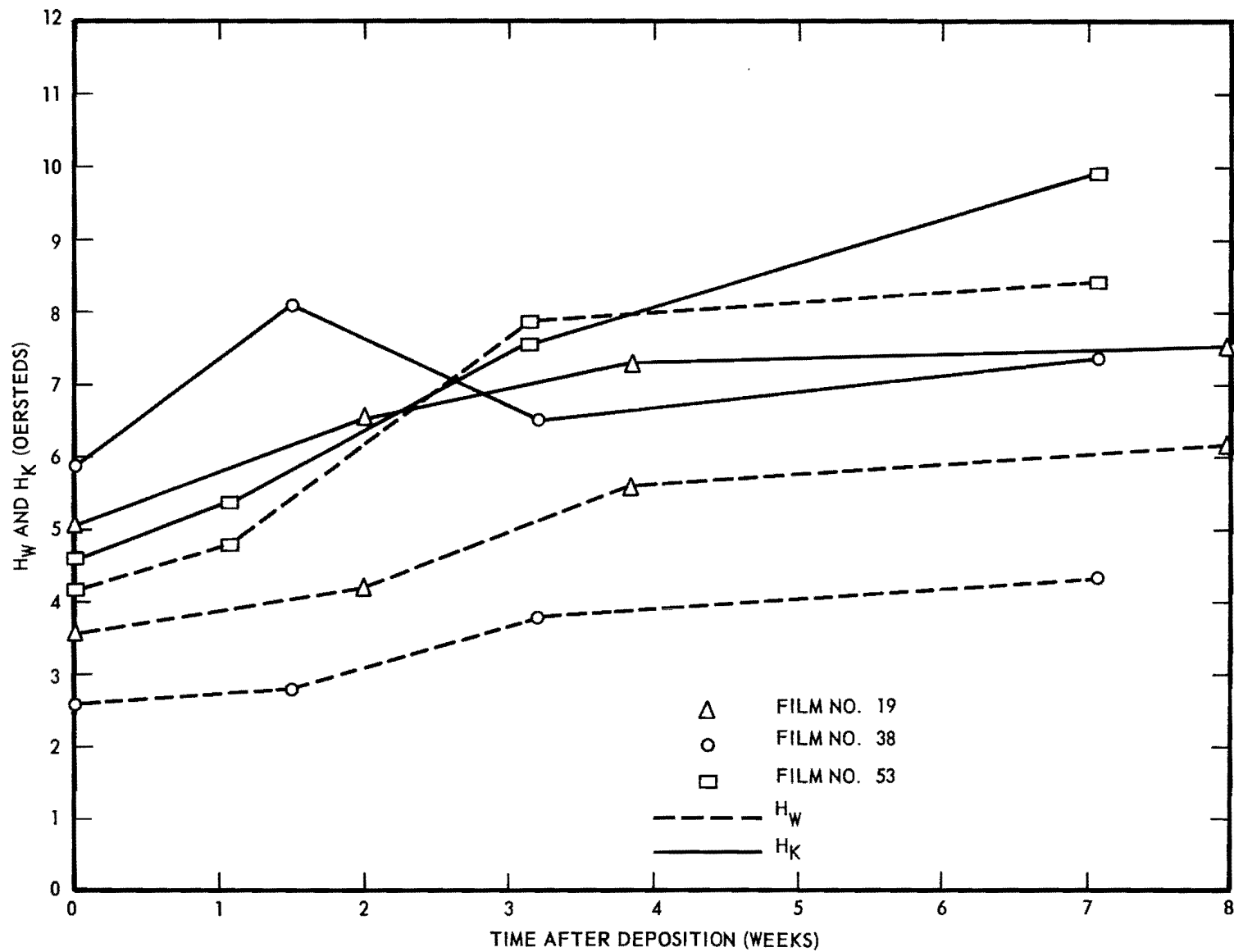


Figure 29. Changes in Coercive Force (H_c) and Anisotropy Constant (H_w) as Films Aged.

cardboard pill boxes and therefore were exposed to high humidity conditions present on some days when the laboratory was not air conditioned. No tests were made to determine whether the changes in magnetic properties were due to room temperature annealing or to chemical changes.

Film Resistivity

Metal films are usually deposited in a state of strain and are thermodynamically unstable regardless of the method of deposition; hence, the physical properties of films usually change with time and with exposure to temperatures above that experienced during growth. In general, if films are protected against corrosion, the degree of aging can be reduced to an insignificant amount for temperatures below the recrystallization temperature by properly annealing during deposition or by post-deposition annealing. The effects of annealing on film resistivity, temperature coefficient of resistance, structure and stability with respect to time and temperature have been treated extensively in the literature.^{60, 61, 62, 63, 64} More recently, annealing effects on magnetic properties of nickel films have been reported.³⁶

The permalloy film on resistance monitor R-1 was deposited during the deposition of specimens 1 through 4. The substrate temperature during deposition was 250°C, and the room temperature resistivity of R-1 was initially 8.35 ohms per square. The resistor was heated to 240°C in vacuum over a period of one hour and cooled to room temperature in a relatively short time. The temperature coefficient of resistivity, TCR, was determined from the cooling cycle as +0.0016/°C and the resistivity did not change. The resistor was heated a second time in vacuum to 425°C and held at that temperature for approximately 30 minutes before cooling; this caused the resistivity to decrease to a value of 5.77 ohms per square. The resistor was cycled again to 250°C without a change in resistivity. The TCR increased during the heat treatment to a final value of +0.0022/°C.

Monitor resistor R-2 was deposited at a substrate temperature of 350°C, and its initial resistivity value was 5.04 ohms per square. It was heated to 375°C in vacuum over a 30 minute period and cooled to room

temperature. The resistivity value did not change. The TCR value of R-2 was $+0.0025/^{\circ}\text{C}$.

The films of R-1 and R-2 were approximately equal in thickness; hence, the higher TCR and lower resistivity of R-2 obtained initially compared to that of R-1 indicate that considerably more annealing occurred with the higher substrate temperature during deposition.

Relation Between Dispersion and Wall Motion

For the purpose of making memory devices it is desirable to have a small amount of dispersion of the easy axis ($\alpha_{50} \leq 1^{\circ}$) and a high threshold for wall motion ($H_w/H_k \geq 1$). As shown in Fig. 30, α_{50} and H_w/H_k generally increased together for the films used for this study. The films thinner than $600 \text{ \AA}$ where the domain walls are mainly of the Neel type followed the relation:

$$H_w/H_k = 0.25 (\alpha_{50} + 1.6^{\circ}) \pm 0.25 \quad (T < 600 \text{ \AA})$$

The films with thickness greater than $1300 \text{ \AA}$ where the domain walls are mainly of the Bloch type followed the relation:

$$H_w/H_k = 0.10 \alpha_{50} \pm 0.20 \quad (T > 1300 \text{ \AA})$$

It seems that the threshold for motion of a Neel wall is about 2.5 times more sensitive to an increase in dispersion than is the coercive force of a Bloch wall. It appears that one must accept a compromise on the values of α_{50} and H_w/H_k .

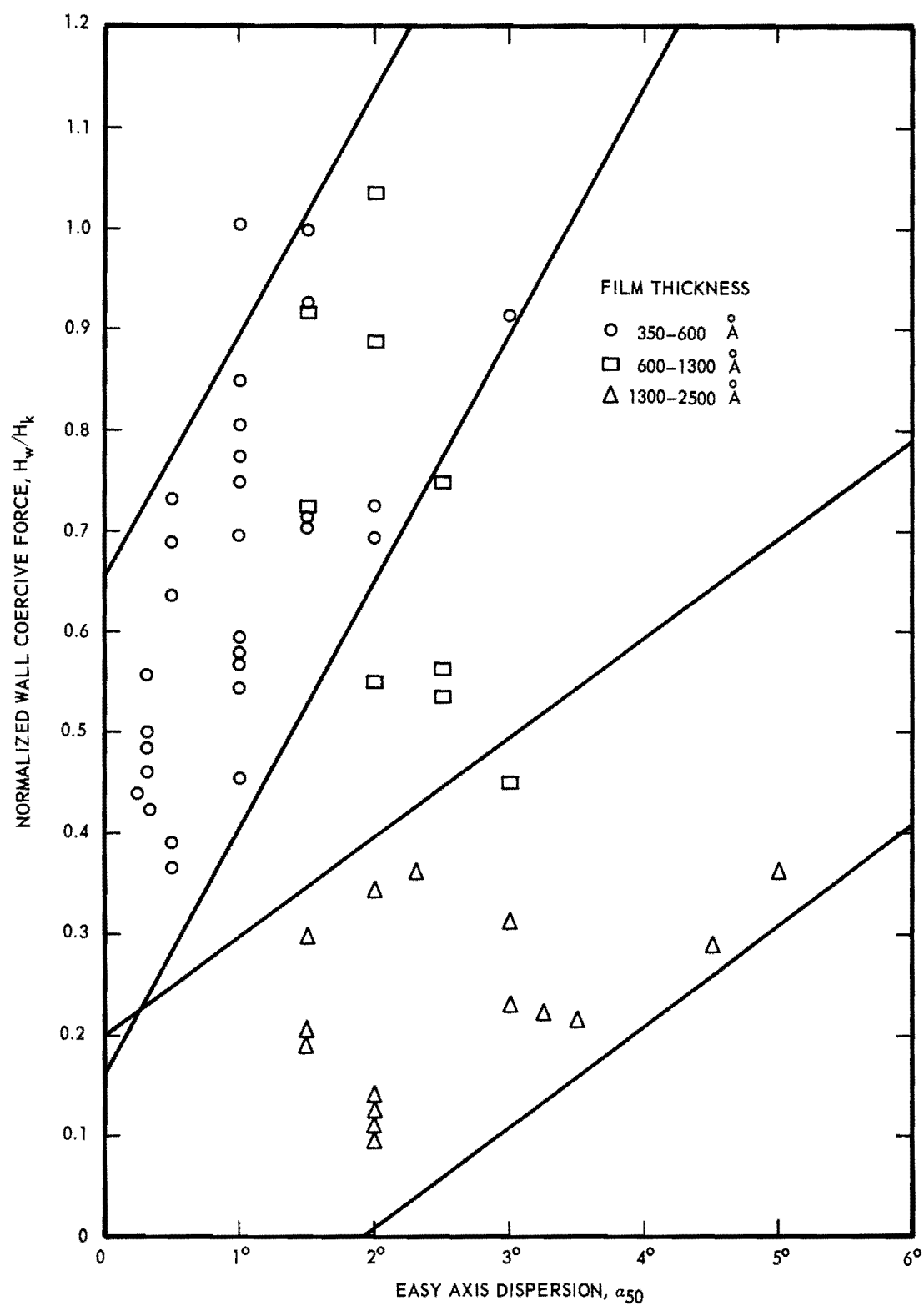


Figure 30. Normalized Wall Coercive Force (H_w/H_k) vs. Easy-Axis Dispersion (α_{50}) for Films in Different Thickness Ranges.

V. REFERENCES

Bibliographies:

"Magnetic Memory Devices and Surfaces Related to Solid State Devices, " Bibliography No. 824, prepared by the Scientific and Technical Information Facility, operated for the National Aeronautics and Space Administration by Documentation Incorporated, 12 January 1965.

"State of the Art on the Effects of Gas Occlusions and Strains on the Magnetic Properties of Thin Permalloy Films, " RSIC-416, B. J. Nicholson and G. B. Gaines, Battelle Memorial Institute, Columbus, Ohio, 28 May 1965.

"Bibliography of Thin Magnetic Films, " Report of the Bibliography Subcommittee of the Nonlinear Magnetics Committee IEEE, November 1964.

References:

1. S. Tolansky, Multiple Beam Interferometry of Surfaces and Films, Clarendon Press, Oxford (1948).
2. G. D. Scott, T. A. McLaunchlan, and R. S. Sennett, J. Appl. Phys., 21, 843 (1950).
3. I. W. Wolf, J. Appl. Phys., 33, 1152 (1962).
4. D. B. Dove, T. R. Long, U. F. Gianola, Proc. 1965 Intermag Conf., 8, 1 (1965).
5. M. Prutton, Thin Ferromagnetic Films, Butterworths, Washington (1964), pp. 95-101.
6. C. J. Kriessman, T. J. Matcovich, W. E. Flannery, IEEE Transactions on Communications Electronics, 519 (Sept. 1964).
7. T. R. Long, J. Appl. Phys., 31, 123S (1960).
8. I. W. Wolf, T. S. Crowther, J. Appl. Phys., 34, 1205 (1963).

9. J. T. Chang, U. F. Gianola, M. W. Sagal, J. Appl. Phys., 35, 830 (1964).
10. E. C. Crittenden, Jr., A. A. Hudimac, and R. I. Strough, Rev. Sci. Inst., 22, 872 (1951).
11. H. J. Oguey, Rev. Sci. Inst., 31, 701 (1960).
12. J. A. Copeland, C. K. Kuo, and E. J. Scheibner, Rev. Sci. Inst., 36, 291 (1965).
13. T. S. Crowther, J. Appl. Phys., 34, 580 (1963).
14. E. J. Torok, R. A. White, A. J. Hunt, and H. N. Oredson, J. Appl. Phys., 33, 3037 (1962).
15. J. Kerr, Rep. Brit. Assoc., 40 (1876).
16. C. A. Fowler and E. M. Fryer, Phys. Rev., 94, 52 (1954).
17. M. Prutton, Phil. Mag., 4, 1063 (1959).
18. S. Middelhoek, Z. Angew. Phys., 14, 191 (1962).
19. E. Fuchs and H. Pfisterer, Proceedings of the 5th International Congress for Electron Microscopy (Academic Press Inc., New York, 1962) p. 114.
20. T. H. Beeforth and P. J. Hulyer, Nature, 199, 793 (1963).
21. A. Green, K. D. Leaver, and M. Prutton, J. Appl. Phys., 35, 812 (1964).
22. S. Middelhoek, IBM Journal, p. 140, Jan. 1962.
23. A. L. Olson and E. J. Torok, J. Appl. Phys., 36, 1058 (1965).
24. G. P. Gagnon and T. S. Crowther, J. Appl. Phys., 36, 1112, (1965).
25. C. D. Olson and A. V. Pohm, J. Appl. Phys., 29, 274 (1958).
26. F. B. Humphrey and E. M. Gyorgy, J. Appl. Phys., 30, 935 (1959).
27. D. O. Smith, J. Appl. Phys., 29, 264 (1958).
28. F. B. Humphrey, J. Appl. Phys., 29, 286 (1958).

29. K. J. Harte, J. Appl. Phys., 36 (Part 2), 960 (1965).
30. J. A. Copeland and F. B. Humphrey, J. Appl. Phys., 34 (Part 2), 1211 (1963).
31. N. C. Ford, J. Appl. Phys., 31, 300S (1960).
32. C. E. Patton and F. B. Humphrey, J. Appl. Phys., 35 (Part 2), 921 (1964).
33. R. F. Soohoo, Magnetic Thin Films, Harper and Row, New York (1965), Chap. 3, 4, 9.
34. M. Prutton, Thin Ferromagnetic Films, Butterworths, Washington (1964), Chap. 3, 4, 5.
35. C. A. Neugebauer, in Structure and Properties of Thin Films, Proceedings of an International Conference held at Bolton Landing, New York, September 9-11, 1959, John Wiley and Sons, Inc., New York, 1959.
36. B. R. Livesay and E. J. Scheibner, J. Appl. Phys., Accepted for Publication (1965).
37. J. S. Kouvel and C. D. Graham, Jr., Proceedings of the Conference on Magnetism and Magnetic Materials, Boston, Massachusetts, October 16-18, 1956, p. 85.
38. I. S. Jacobs and F. E. Luborsky, Proceedings of the Conference on Magnetism and Magnetic Materials, Boston, Massachusetts, October 16-18, 1956, p. 145.
39. W. D. Doyle, J. Appl. Phys., 33, 1769 (1962).
40. G. Robinson, J. Phys. Soc. Japan, 17, Suppl. B-I 558 (1962).
41. H. W. Fuller and M. E. Hale, J. Appl. Phys., 31, 238 (1960).
42. M. S. Cohen, 1965 Intermag Conference held in Washington, D. C. April 21-23, 1965.
43. E. Feldtkeller, Z. Angew. Phys., 15, 206 (1963).
44. D. O. Smith, Magnetism, Vol. III edited by G. T. Rado and H. Suhl Academic Press, New York (1963) p. 499.
45. A. Green, K. D. Leaver and M. Prutton, J. Appl. Phys., 35 812 (1964); see also Section III-E of this report.

46. T. S. Crowther, J. Appl. Phys., 34, 580 (1963).
47. E. J. Torok, R. A. White, A. J. Hunt, H. N. Oredson, J. Appl. Phys., 33, 3037 (1962).
48. H. Hoffmann, J. Appl. Phys., 35, 1790 (1964).
49. A. Baltz and W. D. Doyle, J. Appl. Phys., 35, 1814 (1964).
50. H. B. Callen, R. L. Coren, W. D. Doyle, J. Appl. Phys., 36, 1064 (1965).
51. S. Middelhoek, Z. Angew. Phys., 13, 151 (1961).
52. D. O. Smith, J. Appl. Phys., 32, 70S (1961).
53. E. E. Huber, D. O. Smith and J. B. Goodenough, J. Appl. Phys., 29, 294 (1958).
54. S. Methfessel, S. Middelhoek, H. Thomas, IBM J. Res. and Dev., 4, 96 (1960).
55. H. W. Fuller and M. E. Hale, J. Appl. Phys., 31, 1699 (1960).
56. C. E. Hall, Introduction to Electron Microscopy, McGraw Hill, New York (1953) p. 279.
57. M. S. Cohen, Proc. Fifth Int. Congress for Electron Microscopy Vol. I, Academic Press, New York (1962) p. E-9.
58. D. O. Smith, Structure and Properties of Thin Films, ed. C. A. Neugebauer, John Wiley and Sons, Inc., New York (1959), p. 380. Also J. Appl. Phys. 32, 70S (1961).
59. S. Middelhoek, Ferromagnetic Domains in Thin Nickel Iron Films, Ph.D. Thesis, University of Amsterdam (1961), p. 117.
60. R. J. Raudebaugh, W. A. Edson, and R. B. Belser, "Aging Study of Metal Plating on Quartz Crystals," Final Report, Contract No. DA-36-039-SC-147, U. S. Army Signal Corps, May 1952-ASTIA Document No. 6964.
61. R. B. Belser, "Aging Study of Metal Plating on Quartz Crystals," Interim Report Contract No. DA-36-039-SC-42453, U. S. Army Signal Corps, April 1954-ASTIA Document No. AD 48261.

62. R. B. Belser and W. H. Hicklin, "Investigation on the Aging Effects Resulting from Plating Methods and Materials," Final Report, Contract No. DA-36-039-SC-56753, U. S. Army Signal Corps, March 1955-ASTIA Document No. AD 81420.
63. R. B. Belser and W. H. Hicklin, Electrical Conductivity Studies of Metallic Films, Wright Air Development Center Technical Report 57-660, November 1957.
64. R. B. Belser, "Electrical Resistances of Thin Metal Films Before and After Artificial Aging by Heating," J. of Appl. Phys., 28, 109 (1957).

VI. RECOMMENDATIONS FOR FUTURE WORK

The research conducted during the last six months has provided further insight into the practical problems in preparing good permalloy thin films for memory applications. Many of the subjects which were discussed briefly in the last chapter could be developed more extensively; however, considering the immediate needs of the Astrionics Laboratory of NASA's Marshall Space Flight Center it is recommended that future effort be directed toward developing a specific technique for preparing magnetic films with the necessary properties for use in a digital computer memory system. In order to evaluate the thin magnetic films properly, they should be used in a prototype memory system and judged on the performance of the system. Such an approach would be a logical step from the properties of single magnetic film elements.

A conference at Goddard Space Flight Center on July 20, 1965 with Dr. R. W. Rochelle, head of the Data Systems Branch, brought out the need for a small buffer memory system of about 1024 bits that could operate at a 20 kc read or write rate with an average power consumption of about one watt. It is suggested that these specifications be used for the prototype memory system.

Also it is recommended that further studies of the preparation of magnetic films by chemical vapor deposition be done to extend the preliminary study which was part of this project.

VII. IDENTIFICATION OF PERSONNEL

Dr. E. J. Scheibner, Research Professor of Physics, is chief of the Physical Sciences Division of the Engineering Experiment Station and head of the Solid State Branch. He is known for his work in fields of low energy electron diffraction and physics of solid surfaces.

Dr. J. A. Copeland, Research Physicist, is supervisor of the Solid State Electronics Laboratory and was Project Director for this research project.

Mr. M. D. Carithers, Research Physicist, was responsible for the fabrication of the thin films studied in this project. He is supervisor of the Microcircuit Laboratory and carried out much of the design of the special vacuum evaporation apparatus required.

Mr. B. R. Livesay, Research Physicist, has used torque measuring techniques to study the behavior of nickel and gadolinium films near their Curie temperatures.

Mr. J. L. Brown, Senior Research Physicist, is head of the Analytical Instrumentation Laboratories whose electron microscopy and x-ray facilities were used in the research. Mr. Brown designed and constructed the Lorentz attachment for the RCA electron microscope and carried out most of the Lorentz studies.

Dr. J. D. Fleming, Associate Professor of Chemical Engineering, has done research on chemical vapor deposition of many materials.

Dr. W. J. Corbett, Research Engineer, in the Materials Division of the Experiment Station was associated with Dr. Fleming on the chemical vapor deposition studies.

Mr. C. K. Kuo, Graduate Research Assistant, is working on a Ph.D. in Electrical Engineering and was responsible for the magnetic measurements in this report.

VIII. PUBLICATIONS

A paper on the experimental study entitled "Effects of Post-deposition Oxidation on Permalloy Films" has been submitted for presentation at the Conference on Magnetism and Magnetic Materials in November 1965. The conference papers will be published in the magnetics conference issue of the Journal of Applied Physics in the spring of 1966.

IX. TRIPS AND VISITS

Mr. G. A. Bailey of the Astrionics Laboratory of MSFC visited Georgia Tech on April 8 and 9, 1965 to discuss the research program.

Dr. E. J. Scheibner and Dr. J. A. Copeland attended the International Conference on Magnetism in Washington, D. C. on April 21 through 24.

Mr. C. K. Kuo visited MSFC on April 27 through 30, 1965, to discuss magnetic measurements.

Mr. G. A. Bailey of MSFC visited Georgia Tech on June 7 through 11, 1965, to discuss the research program.

Dr. T. P. Janis and Dr. N. Kinney of Materials Research Corporation visited Georgia Tech on June 8 and 9, 1965 to discuss magnetic foil.

Dr. E. J. Scheibner and Dr. J. A. Copeland visited MSFC on June 28 and 29, 1965.

Dr. E. J. Scheibner and Dr. J. A. Copeland met Mr. G. A. Bailey at Goddard Space Flight Center, Greenbelt, Maryland, to discuss uses of thin-film memory systems in space vehicles with Dr. R. W. Rochelle, head of Data Systems Branch on July 20, 1965.

Dr. J. A. Copeland visited MSFC on August 3, 1965.

Dr. John Blades of Burroughs' Research Laboratory visited Georgia Tech on August 26, 1965 to discuss research on magnetic films.

APPENDIX

TABLE I
DETAILED DATA FOR VACUUM PROCESSED FILMS

SPECIMEN (CODE NO.)	SUBSTRATE (TYPE)	SUBSTRATE CLEANING (CODE NO.)	PRE-DEPOSITION SUBSTRATE HEATING		POWER SOURCE	CRUCIBLE	CRUCIBLE TO SUBSTRATE DISTANCE (INCHES)	EVAPORANT		SUBSTRATE TEMP. (°C)	HELM- HOLTZ'S FIELD (± 20e)	DEPOSITION TIME (MIN.)	FILM THICKNESS OF MONITOR (± 50 Å)	DEPOSITION RATE (Å/MIN.)	INITIAL PRESSURE (TORR)	FINAL PRESSURE (TORR)	MONITOR RESISTOR		DATE	REMARKS	
			TIME (MIN.)	TEMP. (°C)				COMPO- SITION	TEMP. (°C)								CODE NO.	VALUE			
																		(Ω)			(Ω ₀)
1	Corning	4	60	286.250	High	Fused	6 1/2	Permalloy	1375±25	250	27	2	770		2x10 ⁻⁷	2x10 ⁻⁵	R-1	73.3	7.36	6-10	SUBSTRATES OF 1 THRU 16 CLEANED SIMULTANEOUSLY. COMMON EVACUATION CYCLE ON THRU 16. COMMON EVAPORANT AND SOURCE USED ON 1 THRU 16. FILMS DEPOSITED SUCCESSIVELY FROM LOWEST TO HIGHEST ORDER IN 4 SIMULTANEOUS GROUPS 1, 2, 3, & 4; 5, 6, 7, & 8; 9, 10, 11, & 12; 13, 14, 15, & 16.
2	Glass	4	"	"	Frequency	Quartz	"	80.55%Ni	"	"	"	"	"	"	"	"	"	"	"	"	
3	#7059	4	"	"	Generator	7/16" Id.	"	19.45%Fe	"	"	"	"	"	"	"	"	"	"	"	"	
4	"	4	"	"	Lepel	1/2" deep	"	Purchased	"	"	"	"	"	"	"	"	"	"	"	"	
5	"	4	30	286.350	Model	"	"	from	"	350	"	"	702	"	"	"	R-2	50.4	5.04	"	
6	"	4	"	"	T-2, S-1	"	"	Hamilton	"	"	"	"	"	"	"	"	"	"	"	"	
7	"	4	"	"	(450KC)	"	"	Waltz Co.	"	"	"	"	"	"	"	"	"	"	"	"	
8	"	4	"	"	"	"	"	Lancaster	"	"	"	"	"	"	"	"	"	"	"	"	
9	"	4	"	286.400	"	"	"	Penn.	"	400	"	"	787	"	"	"	R-3	51.2	5.12	"	
10	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
11	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
12	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
13	"	4	"	286.250	"	"	"	"	1375±25	250	(Zero 1st Min.)	1 Min.	334	"	"	"	R-4	175.1	17.5	"	
14	"	4	"	"	"	"	"	"	First Min., approx.	"	27 last	Interrupted 15 Sec.	"	"	"	"	"	"	"	"	
15	"	4	"	"	"	"	"	"	1200 to 1350 last	"	1 1/4 Min.	Followed by 1 1/4 Min. EVAP	"	"	"	"	"	"	"	"	
16	"	4	"	"	"	"	"	"	1 1/4 Min.	"	"	"	"	"	"	"	"	"	"	"	
E-1	Electron M Grid	—	"	286.195	"	(New)	"	"	1380±25	195	27	1	372	2x10 ⁻⁷	1x10 ⁻⁴	R-5	174.2	17.4	6-15	SPECIMENS 17 THRU 32 CLEANED SIMULTANEOUSLY. COMMON EVACUATION CYCLE FOR FILMS DEPOSITED ON 6/15. FILMS ON E-1, E-2, E-3, E-4, AND 17 THRU 32 DEPOSITED SUCCESSIVELY FROM LOWEST TO HIGHEST ORDER IN 4 SIMULTANEOUS GROUPS E-1, 17, 18, 19, & 20; E-2, 21, 22, 23, & 24; E-3, 25, 26, 27, & 28; E-4, 29, 30, 31, & 32. HELMHOLTZ COIL LEFT ON UNTIL SPECIMENS COOLED BELOW 100°C FOR ALL SPECIMEN DEPOSITED ON 6/15.	
17	#7059 Glass	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"		"
18	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"		"
19	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"		"
20	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"		"
E-2	E.M.Grid	—	"	286.350	"	"	"	"	"	350	"	1	418	1.5x10 ⁻⁶	1x10 ⁻⁴	R-6	139.8	13.9	"		"
21	#7059 Glass	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"		"
22	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"		"
23	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"		"
24	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"		"
E-3	E.M.Grid	—	"	286.200	"	"	"	"	"	200	"	1/2	Est. 100	3x10 ⁻⁷	7x10 ⁻⁵	R-7	706.1	70.6	"		"
25	#7059 Glass	4	"	"	"	"	"	"	"	"	"	1/2	"	"	"	"	"	"	"		"
26	"	4	"	"	"	"	"	"	"	"	"	1/2	"	"	"	"	"	"	"	"	
27	"	4	"	"	"	"	"	"	"	"	"	1/2	"	"	"	"	"	"	"	"	
28	"	4	"	"	"	"	"	"	"	"	"	1/2	"	"	"	"	"	"	"	"	

NOTES: 1. MEASURED WITH OPTICAL PYROMETER.
2. ROOM TEMPERATURE VALUE AFTER REMOVAL FROM VACUUM CHAMBER.

(Continued)

TABLE I (Continued)

SPECIMEN (CODE NO.)	SUBSTRATE (TYPE)	SUBSTRATE CLEANING (CODE NO.)	PRE-DEPOSITION SUBSTRATE HEATING		POWER SOURCE	CRUCIBLE	CRUCIBLE TO SUBSTRATE DISTANCE (INCHES)	EVAPORANT		SUBSTRATE TEMP. (°C)	HELM- HOLTZ'S FIELD (±20G)	DEPOSITION TIME (MIN.)	FILM THICKNESS OF MONITOR (±80Å)	DEPOSITION RATE (Å/MIN.)	INITIAL PRESSURE (TORR.)	FINAL PRESSURE (TORR.)	MONITOR CODE NO.	RESISTOR		DATE 1966	REMARKS
			TIME (MIN.)	TEMP. (°C)				COMPO- SITION	TEMP. ⁽¹⁾ (°C)									VALUE ⁽²⁾ (Ω) (Ω/□)			
E-4	E-M Grid	—	45	28 to 350	RF Gen.	Fused	6 1/2"	Permalloy Wire	1380±25	350	27	1/2	233		7x10 ⁻⁷	7x10 ⁻⁵	R-8	3189	319	6-15	MEISSNER TRAP OFF FOR SPECIMENS 17 THRU 24 & E-1 & E-2. MEISSNER TRAP ON OTHER 6/15 DE- POSITIONS. SPECIMENS 33 THRU 48 CLEANED SIMULTANEOUSLY AND HAD COMMON EVACUA- TION CYCLE. FILMS ON 33 THRU 48 DEPOSITED SUCCESSIVELY FROM LOWEST TO HIGHEST ORDER IN 4 SIMULTA- NEOUS GROUPS 33 THRU 36, 37 THRU 40, 41 THRU 44, AND 45 THRU 48. HELMHOLTZ COIL LEFT ON FOR THE GREATER TIME OF 5 MIN. OR THAT TO COOL TO -100° C. FOR SPECIMENS 33 THRU 48, A 6 FT. LENGTH OF PERMALLOY WIRE WAS ADDED TO THE EVAPO- RANT MELT USED FOR THE PREVIOUS DEPOSI- TIONS.
29	7059 Glass	4	11	11	ntor, LePel	Quartz,	11	.010" dia,	11	11	11	1/2		11	11	11			11		
30	11	4	11	11	Model	7/16" I.D,	11	80.55% Ni,	11	11	11	1/2		11	11	11			11		
31	11	4	11	11	T-2 1/2-1	1/2 deep	11	19.45% Fe	11	11	11	1/2		11	11	11			11		
32	11	4	11	11	(450 KC)	11	11	Purchased	11	11	11	1/2		11	11	11			11		
33	11	4	(1) none	none	11	11	11	From	1380±25	28	11	4	1372	4x10 ⁻⁷	8x10 ⁻⁵	R-9	356	356	6-17		
34	11	4	(1) 11	11	11	11	11	Hamilton	11	11	11	4		11	11	11			11		
35	11	4	(3) 11	11	11	11	11	Watch Co	11	11	11	4		11	11	11			11		
36	11	4	(3) 11	11	11	11	11	11	11	11	11	4		11	11	11			11		
37	11	4	(3) 30	28 to 150	11	11	11	11	(4) 11	150	11	4	350	11	3x10 ⁻⁵	R-10	1434	143	11		
38	11	4	(3) 11	11	11	11	11	11	(4) 11	11	11	4		11	11	11			11		
39	11	4	(3) 11	11	11	11	11	11	(4) 11	11	11	4		11	11	11			11		
40	11	4	(3) 11	11	11	11	11	11	(4) 11	11	11	4		11	11	11			11		
41	11	4	(3) 30	28 to 200	11	11	11	11	(4) 11	200	11	4	401	3x10 ⁻⁷	1x10 ⁻⁵	R-11	800	8.0	11		
42	11	4	(3) 11	11	11	11	11	11	(4) 11	11	11	4		11	11	11			11		
43	11	4	(3) 11	11	11	11	11	11	(4) 11	11	11	4		11	11	11			11		
44	11	4	(3) 11	11	11	11	11	11	(4) 11	11	11	4		11	11	11			11		
45	11	4	(3) 30	28 to 250	11	11	11	11	(4) 11	250	11	4	368	4x10 ⁻⁷	1.8x10 ⁻⁵	R-12	971	9.71	11		
46	11	4	(3) 11	11	11	11	11	11	(4) 11	11	11	4		11	11	11			11		
47	11	4	(3) 11	11	11	11	11	11	(4) 11	11	11	4		11	11	11			11		
48	11	4	(3) 11	11	11	11	11	11	(4) 11	11	11	4		11	11	11			11		
													</								

- NOTES: 1. MEASURED WITH OPTICAL PYROMETER.
2. VALUE AT ROOM TEMPERATURE AFTER COOLING FROM DEPOSITION TEMPERATURE.
3. PUMPED TO 1×10^{-6} AND BAKED AT 28-450°C OVER A 10 MINUTE PERIOD, COOLED TO ROOM TEMPERATURE, AND THEN BAKED FOR THE INDICATED TIME AND TEMPERATURE IMMEDIATELY BEFORE FILM DEPOSITION.
4. APPARENTLY THE QUARTZ CRUCIBLE TILTED AND A MUCH LOWER DEPOSITION RATE WAS OBTAINED THAN ANTICIPATED.

(Continued)

TABLE 1 (Continued)

SPECIMEN (CODE NO.)	SUBSTRATE (TYPE)	SUBSTRATE CLEANING (CODE NO.)	PRE-DEPOSITION SUBSTRATE HEATING		POWER SOURCE	CRUCIBLE	CRUCIBLE TO SUBSTRATE DISTANCE (INCHES)	EVAPORANT		SUBSTRATE TEMP. (°C)	HELM- HOLTZ'S FIELD (± 200)	DEPOSITION TIME (MIN.)	FILM THICKNESS OF MONITOR (± 80 Å)	DEPOSITION RATE (Å/MIN)	INITIAL PRESSURE (TORR)	FINAL PRESSURE (TORR)	MONITOR RESISTOR		DATE	REMARKS	
			TIME (MIN.)	TEMP. (°C)				COMPO- SITION	TEMP. (°C)								CODE NO.	VALUE (Ω) (V/D)			
49	#7059 Glass	4	30	28-250	H.F. Gener- ator Leptel Model T-24-1 (450 KC)	Fused Quartz 7/16" I. Q. 1/2" deep	6 1/2	Permalloy Wire 0.10 dia., 80.55% Ni, 19.45% Fe	1390±25	250	27	4	2507		3.5x10 ⁻⁷	5x10 ⁻⁵	R-13	11.6	1.2	6/22	SPECIMENS 49 THRU 64 CLEANED SIMULTANE- OUSLY AND PLACED IN VACUUM SYSTEM. #49 THRU #52; #53 THRU #56, #57 THRU #60; AND #61 THRU #64 WERE DEPOS- ITED IN 4 SUCCESSIVE SIMULTANEOUS GROUPS WITH THE VACUUM SYS- TEM OPENED AND EACH GROUP REMOVED AT THE END OF ITS DEPOSITION CYCLE. HELMHOLTZ COIL LEFT ON UNTIL SPECIMENS COOLED TO < 100°C. A TOTALLY NEW PERMALLOY MELT WAS EMPLOYED BEGINNING WITH SPECIMEN 49. SPECIMENS 65 THRU 80 CLEANED SIMULTANE- OUSLY AND PLACED IN VACUUM SYSTEM. #65 THRU #68, #69 THRU #72, #73 THRU #76, AND #77 THRU #80 WERE DEPOSITED IN 4 SUCCESSIVE SIMULTANE- OUS GROUPS WITH THE VACUUM SYSTEM OPENED AND EACH GROUP RE- MOVED AT THE END OF ITS DEPOSITION CYCLE. A TOTALLY NEW PER- MALLOY MELT WAS USED BEGINNING WITH #65. THE PERMALLOY MELT WAS HOT (600-800°C) WHEN O ₂ WAS INITIALLY ADMITTED INTO BELL JAR WHICH RESULTED IN THE FORMATION OF A RATHER HEAVY OXIDE SURFACE OF EVAPORANT (SEE NOTES 6 & 7).
50	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
51	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
52	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
53	"	4	"	"	"	"	"	"	"	"	"	(5) Two ea.	636		4.5x10 ⁻⁷	3x10 ⁻⁵	R-14	5475	5.5	"	
54	"	4	"	"	"	"	"	"	"	"	"	one min.	"	"	"	"	"	"	"	"	
55	"	4	"	"	"	"	"	"	"	"	"	Intervals	"	"	"	"	"	"	"	"	
56	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
57	"	4	"	"	"	"	"	"	"	"	"	(5) Two ea.	1491		5x10 ⁻⁷	5x10 ⁻⁵	R-15	198	2.0	"	
58	"	4	"	"	"	"	"	"	"	"	"	two min.	"	"	"	"	"	"	"	"	
59	"	4	"	"	"	"	"	"	"	"	"	Intervals	"	"	"	"	"	"	"	"	
60	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
61	"	4	"	"	"	"	"	"	"	"	"	(5) Four ea.	1830		5x10 ⁻⁷	1x10 ⁻⁵	R-16	14.7	1.5	"	
62	"	4	"	"	"	"	"	"	"	"	"	one min.	"	"	"	"	"	"	"	"	
63	"	4	"	"	"	"	"	"	"	"	"	Intervals	"	"	"	"	"	"	"	"	
64	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
65	"	4	"	"	"	"	New	"	1360±25	"	"	(6) Two ea.	588		5x10 ⁻⁷	2.5x10 ⁻⁵	R-17	1384	13.8	6/25	
66	"	4	"	"	"	"	"	"	"	"	"	one min.	"	"	"	"	"	"	"	"	
67	"	4	"	"	"	"	"	"	"	"	"	Intervals	"	"	"	"	"	"	"	"	
68	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
69	"	4	"	"	"	"	"	"	1390±25	"	"	(6) Two ea.	702		5x10 ⁻⁷	5x10 ⁻⁵	R-18	450	4.5	"	
70	"	4	"	"	"	"	"	"	"	"	"	two min.	"	"	"	"	"	"	"	"	
71	"	4	"	"	"	"	"	"	"	"	"	Intervals	"	"	"	"	"	"	"	"	
72	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
73	"	4	"	"	"	"	"	"	"	"	"	(6) Four ea.	1060		4x10 ⁻⁷	1x10 ⁻⁵	R-19	439	4.4	6/26	
74	"	4	"	"	"	"	"	"	"	"	"	one min.	"	"	"	"	"	"	"	"	
75	"	4	"	"	"	"	"	"	"	"	"	Intervals	"	"	"	"	"	"	"	"	
76	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	
77	"	4	"	"	"	"	"	"	"	"	"	(7) Four ea.	1552		4x10 ⁻⁷	"	R-20	340	3.4	"	
78	"	4	"	"	"	"	"	"	"	"	"	one min.	"	"	"	"	"	"	"	"	
79	"	4	"	"	"	"	"	"	"	"	"	Intervals	"	"	"	"	"	"	"	"	
80	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	

- NOTES: 5. FIVE MINUTE OXIDIZING INTERVAL AT $(1 - 10) \times 10^{-6}$ TORR INTERPOSED BETWEEN EACH DEPOSITION INTERVAL TO FORM LAYERED STRUCTURE.
6. OXIDIZED AT 250°C AFTER EACH LAYER IN O₂, ONE TO TWO MINUTES FROM $\sim 1 \times 10^{-6}$ TORR TO 1 TORR, 10 MINUTES AT 1 TORR, 5 MINUTES FROM 1 TORR TO 3×10^{-6} TORR, 5 MINUTES TO 5×10^{-7} TORR WITH HELMHOLTZ FIELD APPLIED. HELMHOLTZ FIELD ON AFTER FINAL OXIDATION UNTIL SPECIMEN TEMPERATURE COOLED TO < 100°C.
7. SAME OXIDATION PROCEDURE AS FOR NOTE (6) EXCEPT WITH HELMHOLTZ FIELD OFF DURING OXIDATION AND SWITCHED OFF AT 250°C AFTER FINAL LAYER AND BEFORE COOLING TO < 100°C AND OPENING BELL JAR.

(Continued)

TABLE I (Continued)

SPECIMEN (CODE NO.)	SUBSTRATE (TYPE)	SUBSTRATE CLEANING (CODE NO.)	PRE-DEPOSITION SUBSTRATE HEATING		POWER SOURCE	CRUCIBLE	CRUCIBLE TO SUBSTRATE DISTANCE (INCHES)	EVAPORANT		SUBSTRATE TEMP. (°C)	HELM- HOLTZ'S FIELD (± 200)	DEPOSITION TIME (MIN.)	FILM THICKNESS OF MONITOR ($\pm 80\text{\AA}$)	DEPOSITION RATE ($\text{\AA}/\text{MIN.}$)	INITIAL PRESSURE (TORR)	FINAL PRESSURE (TORR)	MONITOR RESISTOR		DATE	REMARKS	
			TIME (MIN.)	TEMP. (°C)				COMPO- SITION	TEMP. ⁽¹⁾ (°C)								CODE NO.	VALUE (2)			
																		(Ω)			(Ω)
E-5	E.M.Grid	—	30	28-250	H.F. Gen.	Fused	6 1/2	Permalloy Wire	1380 ± 25	250	27	2/3	323		4×10^{-7}	4×10^{-5}	R-21	2265	226	7/3	SPECIMENS 81 THRU 96 CLEANED SIMULTANE- OUSLY AND PLACED IN VACUUM SYSTEM. SPECIMENS E-5 & 81 THRU 84, E-6 & 85 THRU 88, E-7 & 89 THRU 92, AND E-8 & 93 THRU 96 WERE DEPOSITED IN 4 SUCCESSIVE SIMULTANE- OUS GROUPS WITH THE VACUUM SYSTEM OPENED AND EACH GROUP RE- MOVED AT THE END OF ITS DEPOSITION CYCLE. A TOTALLY NEW PER- MALLOY MELT USED ON GROUPS E-5, E-9, & E-13.
81	#7059	4	"	"	erator, Leppel	Quartz	"	.010" dia.	"	"	"	2/3		"	"	"	"	"	"	"	
82	Glass	4	"	"	Model T-2 1/2-1	7/16" I.D.	"	8035% Ni	"	"	"	2/3		"	"	"	"	"	"	"	
83	"	4	"	"	(450 KC)	1/2" deep	"	1945% Fe	"	"	"	2/3		"	"	"	"	"	"	"	
84	"	4	"	"	"	"	"	"	"	"	"	2/3		"	"	"	"	"	"	"	SPECIMENS 97 THRU 112 CLEANED SIMULTANE- OUSLY AND PLACED IN VACUUM SYSTEM. SPECIMENS E-9 & 97 THRU 100, E-10 & 101 THRU 104, E-11 & 105 THRU 108, AND E-12 & 109 THRU 112 WERE DEPOSITED IN 4 SUCCE- SSIVE SIMULTANEOUS GROUPS WITH EACH GROUP REMOVED AT END OF THE DEPOSI- TION/OXIDATION CYCLE.
E-6	E.M.Grid	—	"	"	"	"	"	"	"	"	"	(6) 2/3	246		5×10^{-7}	4×10^{-5}	R-22	2910	291	"	
85	#7059	4	"	"	"	"	"	"	"	"	"	(6) 2/3		"	"	"	"	"	"	"	
86	Glass	4	"	"	"	"	"	"	"	"	"	(6) 2/3		"	"	"	"	"	"	"	
87	"	4	"	"	"	"	"	"	"	"	"	(6) 2/3		"	"	"	"	"	"	"	
88	"	4	"	"	"	"	"	"	"	"	"	(6) 2/3		"	"	"	"	"	"	"	
E-7	E.M.Grid	—	"	"	"	"	"	"	"	"	"	(6) 2/3	292		4×10^{-7}	6×10^{-5}	R-23	3087	309	"	
89	#7059	4	"	"	"	"	"	"	"	"	"	(6) 2/3		"	"	"	"	"	"	"	
90	Glass	4	"	"	"	"	"	"	"	"	"	(6) 2/3		"	"	"	"	"	"	"	
91	"	4	"	"	"	"	"	"	"	"	"	(6) 2/3		"	"	"	"	"	"	"	
92	"	4	"	"	"	"	"	"	"	"	"	(6) 2/3		"	"	"	"	"	"	"	
E-8	E.M.Grid	—	"	"	"	"	"	"	"	"	"	(7) 2/3	342		4×10^{-7}	3×10^{-5}	R-24	2838	284	"	
93	#7059	4	"	"	"	"	"	"	"	"	"	(7) 2/3		"	"	"	"	"	"	"	
94	Glass	4	"	"	"	"	"	"	"	"	"	(7) 2/3		"	"	"	"	"	"	"	
95	"	4	"	"	"	"	"	"	"	"	"	(7) 2/3		"	"	"	"	"	"	"	
96	"	4	"	"	"	"	"	"	"	"	"	(7) 2/3		"	"	"	"	"	"	"	
E-9	E.M.Grid	—	"	"	"	"	"	"	"	"	"	(8) 2/3	296		4×10^{-7}	2×10^{-5}	R-25	3070	307	"	
97	#7059	4	"	"	"	"	"	"	"	"	"	(8) 2/3		4×10^{-7}	2×10^{-5}	"	"	"	"	"	
98	Glass	4	"	"	"	"	"	"	"	"	"	(8) 2/3		"	"	"	"	"	"	"	
99	"	4	"	"	"	"	"	"	"	"	"	(8) 2/3		"	"	"	"	"	"	"	
100	"	4	"	"	"	"	"	"	"	"	"	(8) 2/3		"	"	"	"	"	"	"	
E-10	E.M.Grid	—	"	"	"	"	"	"	"	"	"	(6) 2/3	248				R-26	2400	240	7/4	
101	#7059	4	"	"	"	"	"	"	"	"	"	1/3 min.			"	"	"	"	"	"	
102	Glass	4	"	"	"	"	"	"	"	"	"	Intervals		"	"	"	"	"	"	"	
103	"	4	"	"	"	"	"	"	"	"	"	"		"	"	"	"	"	"	"	
104	"	4	"	"	"	"	"	"	"	"	"	"		"	"	"	"	"	"	"	

NOTES: 8. DURING INITIAL FIVE SECONDS OF FILM DEPOSITION HELMHOLTZ FIELD WAS OFF, THEREAFTER, SAME AS FOR NOTE (7).

(Continued)

TABLE I

SPECIMEN (CODE NO.)	SUBSTRATE (TYPE)	SUBSTRATE CLEANING (CODE NO.)	PRE-DEPOSITION SUBSTRATE HEATING		POWER SOURCE	CRUCIBLE	CRUCIBLE TO SUBSTRATE DISTANCE (INCHES)	EVAPORANT		SUBSTRATE TEMP. (°C)	HELM- HOLTZ'S FIELD (± 20e)	DEPOSITION TIME (MIN.)	FILM THICKNESS OF MONITOR (± 80 Å)	DEPOSITION RATE (Å/MIN.)	INITIAL PRESSURE (TORR)	FINAL PRESSURE (TORR)	MONITOR CODE NO.	RESISTOR		DATE	REMARKS
			TIME (MIN.)	TEMP. (°C)				COMPO- SITION	TEMP. (1) (°C)									VALUE (Ω)	VALUE (Ω)		
E-11	EM Grid	—	30	28-250	HF Gen-erator Leppel Model T-24-1 (450KC)	Fused Quartz 7/16 ID, 1/2" deep	6 1/2 "	Permalloy Wire, .010 dia., 80.55% Ni, 1945% Fe	1380±25	250	27	(9) Two ea. 1/3 min. Intervals	28.3		3x10 ⁻⁷	2x10 ⁻⁵	R-27	296.0	296	7/5	
105	#7059	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"
106	Glass	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"
107	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"
108	"	4	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"	"
E-12	EM Grid	—	"	"	"	"	"	"	"	"	"	2/3	350		With Helmholtz Field applied, Comp in O ₂ at 4x10 ⁻⁴	R-28	>10M		7/6		
109	#7059	4	"	"	"	"	"	"	"	"	"	2/3			at 250°C and cooled to <100°C	"			"		
110	Glass	4	"	"	"	"	"	"	"	"	"	2/3			"	"	"		"		
111	"	4	"	"	"	"	"	"	"	"	"	2/3			"	"	"		"		
112	"	4	"	"	"	"	"	"	"	"	"	2/3			"	"	"		"		
E-13	EM Grid	—	"	"	"	"	"	"	1390±25	"	"	(7) 2/3	412		4x10 ⁻⁷	3x10 ⁻⁵	R-29	197.0	19.7	7/7	
113	#7059	4	"	"	"	"	"	"	"	"	"	(7) 2/3			"	"	"		"		
114	Glass	4	"	"	"	"	"	"	"	"	"	(7) 2/3			"	"	"		"		
115	"	4	"	"	"	"	"	"	"	"	"	(7) 2/3			"	"	"		"		
116	"	4	"	"	"	"	"	"	"	"	"	(7) 2/3			"	"	"		"		
E-14	EM Grid	—	"	"	"	"	"	"	"	"	Zero	(9) Two short Intervals Of Under- mined Length	399		4x10 ⁻⁷	4x10 ⁻⁵	R-30	116.9	11.7	7/8	
117	#7059	4	"	"	"	"	"	"	"	"	"	"			"	"	"		"		
118	Glass	4	"	"	"	"	"	"	"	"	"	"			"	"	"		"		
119	"	4	"	"	"	"	"	"	"	"	"	"			"	"	"		"		
120	"	4	"	"	"	"	"	"	"	"	"	"			"	"	"		"		
							</														

NOTES: 9. NO MAGNETIC FIELD APPLIED DURING OR SUBSEQUENT TO FILM DEPOSITION. TWO DEPOSITION INTERVALS WERE SEPARATED BY ABOUT 10 MINUTES OF OXIDIZING AT A CHAMBER PRESSURE OF 5×10^{-6} TORR.

TABLE 2
SIGNIFICANT DEPOSITION VARIABLES OF VACUUM PROCESSED PERMALLOY FILMS

SPECIMENS (CODE NO.)	Ni-Fe (1) EVAPORANT	SUBSTRATE TEMPERATURE (°C DURING DEPOSITION)						FILM		OXIDATION	HELMHOLTZ FIELD					
		20	150	200	250	350	400	NO. OF Ni-Fe LAYERS	TOTAL THICKNESS (± 50 Å)		DURING DEPOSITION		DURING OXIDATION		DURING COOLING	
											ON	OFF	ON	OFF	ON TO ≤ 100 °C	OFF 60 250 °C
1-4	NEW MELT				X			1	770	NONE	X				X	
5-8	After Evap. of 1-4					X		1	702	"	X				X	
9-12	After Evap. of 5-8						X	1	787	"	X				X	
13-16	After Evap. of 9-12				X			2	334	15 Seconds Interruption @ H.V.	2 nd half	1 st half	X		X	
E-1 & 17-20	NEW MELT			X				1	372	NONE	X				X	
E-2 & 21-24	After Evap. of E-1gp					X		1	418	"	X				X	
E-3 & 25-28	After Evap. of E-2gp			X				1	395	"	X				X	
E-4 & 29-32	After Evap. of E-3gp					X		1	233	"	X				X	
33-36	Ni-Fe Wire added to Previous Melt	X						1	1372	"	X				X	
37-40	After Evap. of 33-36		X					1	350	"	X				X	
41-44	After Evap. of 37-40			X				1	401	"	X				X	
45-48	After Evap. of 41-44				X			1	368	"	X				X	
49-52	NEW MELT				X			1	2507	"	X				X	
53-56	After Evap. of 49-52				X			2	636	5 Min. @ 250 °C and 1×10^{-6}	X		X		X	
57-60	After Evap. of 53-56				X			2	1491	Torr after each layer	X		X		X	
61-64	After Evap. of 57-60				X			4	1830	"	X		X		X	
65-68	NEW MELT				X			2	588	250 °C Film Exposed to Dry O ₂	X		X		X	
69-72	Exposed to O ₂ during Oxid. of 65-68				X			2	702	at 1 Torr after each layer	X		X		X	
73-76	Exposed to O ₂ during Oxid. of 65-72				X			4	1060	"	X		X		X	
77-80	Exposed to O ₂ during Oxid. of 65-76				X			4	1552	"	X			X		X
E-5 & 81-84	NEW MELT				X			1	323	NONE	X				X	
E-6 & 85-88	After Evap. of E-5 gp.				X			1	246	250 °C Film Exposed to Dry O ₂	X		X		X	
E-7 & 89-92	Exposed to O ₂ during Oxid. of E-6 gp.				X			1	292	at 1 Torr after deposition	X		X		X	
E-8 & 93-96	Exposed to O ₂ during Oxid. of E-6 & E-7gps				X			1	342	"	X			X		X
E-9 & 97-100	NEW MELT				X			1	296	"	Final 35 sec.	Initial 5 sec.		X		X
E-10 & 101-104	Exposed to O ₂ during Oxid. of E-9gp.				X			2	248	250 °C Film Exposed to Dry O ₂	X		X		X	
E-11 & 105-108	Exposed to O ₂ during Oxid. of E-9 & E-10 gps.				X			2	283	at 1 Torr after each layer	X		X		X	
E-12 & 109-112	Exposed to O ₂ during Oxid. of E-9, 10, & 11 gps.				X			1 layer of Ni-Fe oxide	350	Reactively Evaporated in O ₂ @ 4×10^{-4} Torr	X				X	to ≤ 75 °C
E-13 & 113-116	NEW MELT				X			1	412	250 °C Film Exposed to Dry O ₂ at 1 Torr after deposition	X			X		X
E-14 & 117-120	Exposed to O ₂ during Oxid. of E-13gp.				X			2	399	Oxid. about 10 min. at 5×10^{-6} Torr after each layer		X		X	not applied	

Notes: 1. When O₂ was admitted to oxidize films, the evaporant was approximately 600 to 700 °C but cooled to less than color temperature in about 2 minutes.

2. The thickness is the thickness value obtained for the thickness monitor coated with each group.

MAGNETIC PROPERTIES OF FILMS

FILM	S. T.	THICKNESS	H _W	H _K	H _W /H _K	M _{RR}	θ	m (MV)	a ₅₀	H _T	H _T /H _K	REMARKS
1	250°	770	2.50	3.40	.736	40%	+11°	10.6	1.0°			
2	"	"	2.63	4.87	.541	20%	+15°	10.7	2.5°			
3	"	"	2.98	5.40	.552	15%	+4°	10.0		0.13	0.024	
4	"	"	2.95	4.30	.686	18%	+9°	10.0	0.5°	0.14	0.032	
5	350°		—	—	—	—	—	—	—			
6	"	700	9.54	12.26	.767	100%	+4°	13.0	—			
7	"	"	9.87	11.42	.862	100%	+52°-38°	11.8	—	2.45	0.214	
8	"	"	10.57	9.96	1.05	100%	+40°-50°	11.8	—	2.10	0.217	
9	450°	790	28.0	—	—	100%	—	12.0	—			
10	"	"	25.5	—	—	100%	—	10.4	—			
11	"	"	25.0	—	—	100%	—	9.8	—			
12	"											
13	250°											
14	"	330	4.28	4.55	.941	50%	+10°	4.7	—	1.53	0.336	
15	"	"	3.40	4.00	.850	50%	+5°	4.4	1.0°	1.57	0.390	
16	"	"	3.70	4.25	.870	50%	-2°	4.3	2.5°			
17	195°	370	4.15	5.26	.789	18%	+7°	5.40	1.0°			
18	"	"	3.95	5.31	.726	17%	-3°	5.20	0.5°			
19	"	"	3.60	5.03	.716	17%	-3°	6.0	1.5°	1.2	0.24	
20	"	"	3.82	4.77	.801	20%	+3°	5.8	1.0°	1.0	0.21	
21	350°	420	53.8			ISOTROPIC						
22	"	"	58.5			"						
23	"	"	36.0			"						
24	"	"	41.8			"						
25	200°		BROKEN			SUBSTRATE						
26	"	100	2.90					0.8				
27	"	"	1.88					1.30				
28	"	"	2.60					1.07				
29	350°	230				NO OBSERVABLE SIGNAL						
30	"	"				"						
31	"	"	4.93					1.70				
32	"	"	3.48					1.50				

(Continued)

TABLE 3 (Continued)

FILM	S. T.	THICKNESS	H _W	H _K	H _W /H _K	M _{RH}	θ	m _(MV)	α_{80}	H _T	H _T /H _K	REMARKS
33	Rm. Tp.	1370	3.61	9.08	.364	15%	- 9°	5.78	5.0°			
34	"	"	1.39	8.79	.197	18%	+28°	24.4	1.5°			
35	"	"	1.37	10.15	.117	6%	+ 3°	25.8	2.0°			
36	"	"	1.25	10.81	.144	1%	+11°	21.3	2.0°			
37	150°	350	2.87	5.76	.498	20%	+ 4°	4.7	0.3°			
38	"	"	2.55	5.85	.436	8%	- 1°	5.0	0.2°	0.7	0.12	
39	"	"	2.83	6.30	.449	14%	+ 3°	5.5	0.3°	1.05	0.167	
40	"	"	2.25	6.00	.375	7%	+ 9°	5.3	0.5°			
41	200°	400	2.42	5.69	.426	6%	+ 7°	7.6	0.3°	0.165	0.029	
42	"	"	2.42	5.20	.465	20%	+ 3°	6.9	0.3°	0.55	0.106	
43	"	"	3.12	5.42	.577	33%	+ 6°	7.6	0.9°			
44	"	"	2.62	5.81	.452	10%	+ 5°	8.0	1.0°			
45	250°	370	2.62	4.73	.554	2%	+ 7°	5.9	0.3°			
46	"	"	2.52	4.70	.537	15%	+ 5°	6.1	1.0°	1.25	0.22	
47	"	"	2.20	5.72	.385	17%	+ 3°	6.4	0.5°	1.28	0.224	
48	"	"	2.31	5.05	.458	12%	+ 1°	6.9	1.0°			
49	250°	2500	.83	6.62	.276	2%	+ 3°	35.1	4.5°			4 minute deposition time
50	"	"	.74	5.69	.306	2%	+ 3°	41.5	3.0°			
51	"	"	.49	6.30	.238	2%	+ 3°	37.6	3.2°			
52	"	"	.60	7.10	.226	3%	+ 3°	45.0	3.5°			
53	250°	1280	4.15	4.59	.905	17%	+ 3°	10.9	1.5°	0.92	0.20	2 layers one minute each
54	"	"	5.09	4.99	1.04	30%	- 1°	10.3	2.0°	1.03	0.206	
55	"	"	4.12	4.60	.893	20%	+ 4°	12.5	2.0°	1.45	0.315	
56	"	"	3.72	4.65	.714	20%	0°	13.9	1.5°	0.53	0.114	
57	250°	1500	1.85	5.62	.330	12%	- 1°	26.0	2.0°			2 layers two minutes each
58	"	"	1.34	4.75	.282	10%	+ 3°	25.5	1.5°			
59	"	"	1.61	4.70	.343	16%	+ 1°	28.7	2.2°			
60	"	"	1.23	5.85	.210	10%	+ 1°	27.7	1.5°			
61	250°	1830	1.98	5.61	.353	12%	+ 9°	33.3	4.0°			4 layers one minute each
62	"	"	1.30	5.49	.237	9%	0°	36.0	3.0°			
63	"	"	.67	5.75	.132	7%	+ 4°	35.8	2.0°			
64	"	"	.83	5.23	.159	6%	+ 5°	35.0	2.0°			

(Continued)

TABLE 3 (Continued)

FILM	S. T.	THICKNESS	H _W	H _K	H _W /H _K	M _{RH}	θ	m _(MV)	α_{60}	H _T	H _T /H _K	REMARKS
65	250°	590	15.51	20.68°	.751°	100%	+10°	5.1	10.0°			Two one-minute layers oxidized
66	"	"	15.98	19.03°	.839°	"	+9°	5.4	7.0°	1.68	0.088	
67	"	"	18.19	17.39°	1.047°	"	+7°	6.5	13.0°			
68	"	"	15.22	13.89°	1.098°	"	+3°	5.8	7.0°			
69	250°	700	12.88	10.58°	1.217°	100%	+2°	14.7	6.0°	1.38	0.130	Two two-minute layers oxidized
70	"	"	10.57	4.60°	2.320°	"	-2°	14.1	5.0°	0.98	0.213	
71	"	"	7.88	9.26°	.851°	"	-1°	15.5	3.0°			
72	"	"	8.10	3.65°	2.226°	"	+1°	17.7	3.0°			
73	250°	1060	6.20	6.21°	.999°	100%	-1°	14.2	3.0°	0.98	0.158	Four one-minute layers oxidized
74	"	"	6.80	5.25°	1.294°	"	0°	16.0	3.5°	1.13	0.216	
75	"	"	8.45	6.80°	1.242°	"	+3°	16.7	8.0°			
76	"	"	8.45	6.81°	1.242°	"	+5°	16.5	5.0°			
77	250°	1550	3.70	4.60°	.805°	100%	+1°	12.5	22.0°	0.925	0.205	Four one-minute layers. Oxidized with Helmholtz coils off.
78	"	"	3.38	6.32°	.525°	OFF CENTER				0.70	0.111	
79	"	"	3.04	5.38°	.564°	40%	+1°	17.0	2.5°	0.65	0.121	
80	"	"	2.77	6.39°	.434°	60%	+1°	14.8	3.0°	0.53	0.083	
81	250°	320	4.78	6.80°	.703°	60%	+2°	4.0	1.5°	1.275	0.187	Single layer, not oxidized
82	"	"	5.81	10.29°	.555°	50%	+1°	4.6	0.3°	0.88	0.086	
83	"	"	5.37	7.81°	.686°	60%	+3°	3.3	1.0°	1.08	0.138	
84	"	"	5.84	9.19°	.636°	40%	0°	2.8	0.5°	0.75	0.082	
85	250°	250	10.81	15.64°	.692°	80%	0°	2.0	2.0°	0.58	0.037	Oxidized
86	"	"	10.61	14.19°	.749°	50%	0°	2.3	1.0°	0.28	0.020	
87	"	"	7.53	10.44°	.721°	60%	+4°	1.7	2.0°	0.38	0.036	
88	"	"	6.88	11.98°	.575°	80%	+2°	2.3	1.0°	0.25	0.021	
89	250°	300	11.40	15.37°	.743°	90%	+1°	2.2	3.0°	1.10	0.072	Oxidized
90	"	"	10.81	15.04°	.720°	100%	+5°	1.8	3.0°	0.65	0.044	
91	"	"	8.82	10.48°	.842°	"	-1°	2.4	2.5°	0.48	0.046	
92	"	"	8.55	13.49°	.633°	"	+2°	2.3	4.0°	0.53	0.036	
93	250°	350	5.04	4.80°	1.050°	30%	+3°	3.3	1.0°	1.28	0.267	Oxidized
94	"	"	5.01	5.49°	.914°	50%	+4°	2.8	1.5°	0.68	0.124	
95	"	"	4.21	4.60°	.916°	30%	+2°	3.1	3.0°	1.20	0.262	
96	"	"	4.70	4.70°	1.000°	50%	+4°	2.9	1.5°	1.15	0.245	

(Continued)

TABLE 3

[illegible]