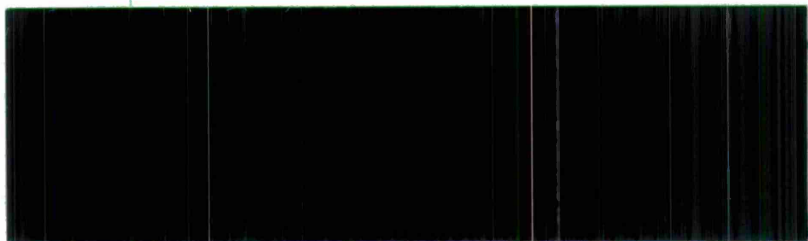


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ELECTRODEPOSITED MAGNETIC FILM DEVELOPMENT

Semi-Annual Technical Report No. 2

(Final Report - Second Edition)

by

I. W. Wolf, N. Ballard, L. Banzet

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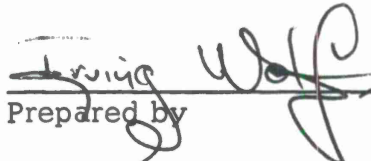
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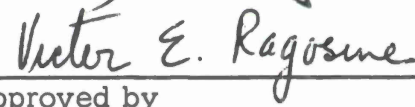
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SEMI-ANNUAL TECHNICAL REPORT NO. 2
(FINAL REPORT - SECOND EDITION)

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LINCOLN LABORATORY


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1.0 INTRODUCTION

The intent of the development program on electrodeposited magnetic film, undertaken by Ampex Research, under the sponsorship of Lincoln Laboratory, has been to make use of novel and conventional electrodeposited film structures as memory elements. This program has been closely coupled with the film and device development efforts at Lincoln Laboratory.

Effort during 1964-65 was directed toward the solution of problems in three major areas: 1) thin film sandwich structures, 2) closed flux structure element development, and 3) inverted film fabrication. During the second half of the year the major effort was devoted to the closed flux structure development including the production of large area samples of high coercivity film uniformly plated on a ground plane structure but separated from this structure by pinhole free insulation.

The major portion of the work in the first area, work on development of thin film sandwich structures, had been completed during the course of the first half of the program and was reported in the first semi-annual report. The results of this work indicated that the "Neél-wall" sandwich structure, which was successfully fabricated and photoetched, did indeed promise improved creep resistance for open flux structure elements in memory planes. In order to test the creep resistance, several planes were etched in the configurations used for the FX-1 memory.

Preliminary work was also completed on a closed flux structure which would incorporate a plated copper conductor in a thin permalloy sheet. The results of these efforts are presented in this report under the headings Neel Wall Sandwich Structure, Ground Plane High Density Structure and Closed Flux Structure.

2.0

NEEL WALL SANDWICH STRUCTURE

A procedure for preparing a double magnetic film structure separated by a nonmagnetic intermediate layer was described in the first semi-annual report. By using a nickel-phosphorus intermediate layer, good films consisting of 350Å thick permalloy, 2000Å thick nickel-phosphorus and a second layer of 350Å permalloy were fabricated such that excellent squareness, low easy axis, dispersion films could be made. Hysteresis data on a series of samples indicated that the magnetic properties were well within the limits required for memory application. However, one problem remained to be solved before the structures could be tested as memory elements. The ferric-chloride etching procedure normally used for permalloy films was found inadequate for this composite film. Since a layer of nickel-phosphide residue remained in the exposed regions of the films when the nickel-phosphorus layers were removed by the etchant. This residual nickel-phosphide film protected the lower layers from the etchant. Thus, it was necessary to develop a special photoetch procedure for this composite structure.

Among the etch procedures examined, two appeared promising. The first used a nitric acid rinse as a second etchant. Nickel-phosphide is soluble in nitric acid, and thus, a two step etch or a single etch employing more concentrated nitric acid was possible. The second technique employed an electrolytic etch in a dilute nitric acid. This technique provided a very low undercutting ratio and thereby, considerably improved image resolution. Details of these two procedures are given in Appendix I.

Having developed a successful etch procedure, arrangements were made to produce several samples of Neél wall sandwich structure elements arranged in the configuration used for the FX-1 memory. These samples were then submitted to Lincoln Laboratory for testing. The results of the hysteresis measurements for three samples are given in Table I. Subsequent pulse testing indicated that film (1) had only one creeping bit out of a total 448, using the same value of transverse drive for reading, writing and disturbing. This disturbed bit was located at the edge of the array. Film (2) had several bits with low output in a cluster in the vicinity of the plating contact clip. Two of the spots were over-etched but the rest of the plane was good. The third plane had a considerable over-etched area and some insufficiently written spots which could be fully written with more digit current. However, when R_d was increased to give an equal disturb digit field, creeping occurred.

Film No.	1	2	3
θ_1 (deg.)	-1	+1	0
α_q (ma)	6	5	6.5
α (90%) ma	14	14	15
H_d (oe)	2.1	2.2	1.5
H_c (oe)	2.4	2.6	2.4
H_k (oe)	2.4	3.3	3.0
$2\phi_{\max}$ (mv)	25	25	25
Thickness	830Å	830Å	830Å

TABLE I

Thus, though the sandwich films showed a good deal of promise for open flux structure memory planes, some work on perfecting the etch technique, and eliminating the cause of the low output clusters seen in film (3) (believed to be due to the plating clip), would have to be carried out before good yields of perfect planes were possible.

Because of more pressing problems on closed flux structures, this phase of the effort was discontinued.

3.0 DEVELOPMENT OF LARGE AREA HIGH DENSITY GROUND PLANE STRUCTURES

Work began at Ampex in conjunction with the program at Lincoln Laboratory which had concentrated on the development of copper coated 2-mil permalloy lines on 4-mil centers in a 2in. x 10 in. plane, on an electrodeposited counterpart having the following characteristics:

$H_{cd} \leq 10$ Oe; easy axis dispersion low; film in close proximity to a conductive ground plane.

The development of such a structure required a departure from past operating procedures in several ways:

1. A high coercivity material, presumably of the cobalt-nickel-iron family, would have to be developed and tested for memory application.
2. The substrate would have to be altered from that normally used, i.e., a change from thin glass to a conductor coated with thin insulator.

3. The development of a plating technique for large areas of uniform magnetic film would be required.
4. The development of photoetch techniques for utilizing the plated structure in a memory configuration would be required.

In approaching the problem of a high coercivity material, reference* was made to earlier work with the cobalt-nickel-iron system. In that work, it was shown that H_k and H_c could be increased with the addition of cobalt while maintaining good dispersion characteristics and low magnetostriction sensitivity. A series of tests were carried out to optimize the composition for a film having an H_c of approximately 12 Oe, and H_k of approximately 15 Oe and a thickness of 750Å.

It was shown during this study that the coercivity varied more rapidly than the magnetoelastic sensitivity with nickel-to-iron ratio. Indeed, whereas the latter could be used in the permalloy system as a good composition control indicator, such was not the case in the cobalt-nickel-iron system. Moreover, departures from optimum nickel-iron ratios gave increased dispersion when the cobalt concentrations were sufficiently high to provide H_k 's of 15 Oe or above. Another factor which appeared during the course of the development was a greater plating bath pH dependency than had been encountered in the permalloy system.

* I. W. Wolf, Conference on Electrical and Magnetic Properties of Thin Metal Layers, 1961, Louvain, Belgium

As a result of the development, the following bath was found acceptable for producing the high coercivity films used in the ground plane structure:

NiFeCo Solution

500 ml/l Ni sulfamate concentrate

6 g/l NaCl

37.5 g/l HBO_3

0.1 g/l saccharin

0.1 g/l sodium lauryl sulfate

4 g/l FeSO_4

10 g/l CoSO_4

50 ma/in² cathode current density

The second development in this project was that of the ground plane structure itself. It was necessary to find a material which was reasonably flat but sufficiently inexpensive for detailed experimentation. A special sheet aluminum was found¹ to serve the purpose well, and thus, was used throughout the experiment.

The aluminum was cut into strips of suitable size and dipped into a liquid organic resin diluted with solvent. Upon curing, the resin polymerized forming a hard pinhole free smooth coating on the aluminum. From the standpoint of smoothness and mechanical characteristics the resin-coated aluminum substrate appeared to be an ideal configuration for the ground plane structure. Unfortunately, a problem arose later with regard to adhesion of metal films to the resin coated surface.

¹Alcoa 1100-H25 One Side Bright

The first resin used, a silicone compound², was chosen because of its ability to withstand the high temperatures to which the material is subjected in the sputtering cycle. Experiments were first done with 1-inch square coated aluminum substrates. Initially, a nichrome-gold sputtered conductor was used as the substrate material. A number of samples were prepared and were found to have excellent magnetic and physical properties. In order to improve the probabilities of good photoetch properties, copper was substituted for gold. Samples 1-inch square of nichrome-copper were prepared and also appeared to have good characteristics. However, when larger samples (2 inch x 10 inch) of nichrome-copper were prepared, it was found that poor adhesion between the nichrome and resin occurred. Blistering and peeling of the metal film from the resin substrate resulted. A good deal of effort was then devoted to overcoming this problem. Further, the problem had not been anticipated at the beginning of the program and thus, created unexpected delays in carrying out the final preparation of the ground plane structure.

At first, it was believed that the difference in success between the small and large ground plane structures was significant. It was thought then that perhaps the introduction of a large undergassed surface into the sputtering system created in organic impurities in the sputtering atmosphere at the time the initial sputtered metal film was being deposited. This, in turn, might have created a thin organic layer with poor substrate to the adhesion as an underlayer to the metal film. (Attempts at precleaning the resin substrate by exposing it to vacuum heating and presputtering electron-bombardment conditions were partially successful.) However, when attempts were made at a quasi-production batch of samples, the adhesion problems, originally observed, reappeared.

²Dow Corning CP77 Tri Var #7 Thinner 3:1

A second approach was made substituting an epoxy resin for the silicone. It was reasoned that the epoxy linkages themselves might be opened, producing much better adhesion to the metal film during sputtering. Again, initial results looked favorable. However, continued examination indicated only slight improvement.

A chromium complex material³ which was known to improve adhesion of electroless metals to resin surfaces was incorporated into the material. However, again, little improvement in ultimate adhesion was achieved.

The next approach resulted from experiments indicating that much better adhesion could be obtained to a resin which had not been completely cross-linked (cured at high temperatures) was obtained, since presumably the resin had surface linkage bonds free for linking to the sputtered surface. A number of samples were prepared using a low temperature cure (10 minutes at 100°C), subsequent sputtering and finally recuring at high temperature (180°C).

The effect on adhesion was marked. The films showed good bonding to the surface. On the other hand, the sputtering of the low-temperature cured material resulted in degradation of the polymer. This degradation was exhibited in several ways. First, where solvents were present, the polymer tended to blister. Next, after all solvents had been removed before sputtering, cracking occurred. This cracking was believed to be the result of surface hardening which occurred during the sputtering process (that is, a cross-linking of the surface molecules)

³Dupont Beta-Resorcylo Chromic Chloride

while the bulk of the film remained uncured. During further curing, either while sputtering or in the subsequent curing process, the bulk resin would shrink creating large tensile stresses in the surface film. Thus, while much had been learned about resin metal film interfaces and side effects, no acceptable process for obtaining good adhesion between resin and sputtered film had been achieved.

It should be pointed out that the extent of adhesion which was necessary to prevent undercutting during the photoetching of these fine-line structures exceeds considerably that normally needed in film memories. A semi-quantitative description of the required adhesion may be deduced from the following test: When Scotch tape is applied to the metal surface, it is necessary that film adhesion be good enough to withstand a rapid pull of the Scotch tape. Adhesion which withstands a slow pull of Scotch tape will not prevent etchant undercutting.

It had been observed, during the course of the experiment, that often when a resin which had been exposed to the sputtering chamber but not actively sputtered on (i.e., that which was on the underside of a sample) and was then exposed to direct sputtering, it showed much less tendency to crack. Attempts to precondition the resin with a vacuum heating process instead of with the sputtering chamber were unsuccessful. When, indeed, the underside sputtering process was introduced as a step in the procedure, good adhesion of copper-nichrome resin was finally achieved.

Films were prepared and tested for photoetchability according to the techniques given in Appendix II. The results were unfavorable indicating that even though copper had been assumed to be the easier to photoetch, undercutting was severe; thus a return to the gold-nichrome substrate was made. Similar tests were then conducted

with the gold-nichrome which resulted in some improvement over the copper-nichrome. However, photoetching results were, at best, marginal. Failure resulted from an inability to remove the base nichrome layer with the weak etchant necessary for preserving the image areas of much more readily etchable materials above this layer.

Attempts were made to reduce the base nichrome layer's thickness to a value which would yield a sufficiently high electrical resistance so as not to affect the electrical characteristics of the image structure. However, the thinnest film which yielded sufficient adhesion had too much conductance to be useable.

Attempts were made to remove, by chemical means, this very thin nichrome layer prior to removal of the upper images without success because of rapid etching and undercutting of the more easily etched materials.

It became apparent that a completely different system was necessary for the production of the substrates for these samples. A system was needed which provided the adhesive qualities of the nichrome yet which was more easily etched. Various different combinations, mixtures, and compounds were re-evaluated as the underlying insulation layer in an attempt to provide a surface to which sputtered gold would adhere directly. Although it is undoubtedly possible to provide such a system, the attempts were dropped when it was noted that excellent adhesion and etchability could be achieved with the use of an evaporated film of aluminum deposited on a Hysol epoxy insulation layer. The first series of tests on this new technique were made to determine the insulating layers adhesion requirements and its ability to withstand sputtering. It was found that the films must be

cured for at least 18 hours to provide physical strength and heat resistance sufficient to allow sputtering. Also, it was found that it is not necessary to include the chromium complex in the epoxy to provide adhesion of the evaporated aluminum layer.

A mixture of two parts Hysol epoxy to one part Hysol hardener to three parts Hysol solvent, draw-coated at a rate of approximately 1 in./minute and cured at 200°C for 16 hours provided an insulating layer with the desired characteristics.

It should be noted also that aluminum strips which had been prepared by removing all sharp edges, washed thoroughly with a mixture of detergent (super NZL) and BPA #1 fine non-abrasive buff powder, rinsed thoroughly with hot water while scrubbing to remove all traces of soap, rinsed with reagent grade methanol with gentle scrubbing to insure clean surfaces, re-rinsed with reagent grade methanol, and finally baked at 100°C for 15 minutes immediately prior to coating, yielded samples showing no chip-off around the edges, good over-all adhesion, and smooth, contaminate free coatings.

When the aluminum coatings were tested on epoxy, there was a marked improvement over any other previously mentioned film materials. Thus, it was decided to attempt using the aluminum as an intermediate substrate since bonding to it would be much easier.

In the first attempt, nichrome-gold was sputtered directly on the aluminum. This approach led to severe corrosion of the aluminum during plating in an acid bath. A second attempt was made utilizing the naturally protective coating of aluminum oxide on

the aluminum. This coating was achieved with a commercial process known as chromatizing⁴. The results were successful.

Thus, a process was evolved for producing magnetic films on the resin ground plane substrate. The films were found to have sufficiently high adhesion and were etchable. However, the processing techniques and procedures involved, particularly in the chromatizing process, required such control that the samples produced showed a relatively large rejection rate during subsequent plating and etching procedures due to partial or complete loss of adhesion at the chromatized-aluminum/nichrome-gold interface. Therefore, during the final weeks of the project, attempts were made to improve or replace this procedure such that a greater degree of simplicity, a higher reliability, and an improved layer-to-layer adhesion would result.

A completely different procedure for the substrate production was, by this time, under study. It was realized that the original necessity of coating the aluminum with epoxy, that of providing a smooth, high resistive layer between the ground plane and the upper magnetic and conductive sandwich structure, could also be provided by anodizing the surface of the aluminum as well as providing a surface upon which the nichrome-gold transition layer could be applied with a significant improvement in adhesion. Several samples were anodized in various baths and temperatures and current densities, with the result that two samples anodized in a solution containing 220 g/l H_2SO_4 , 10 g/l oxalic acid, in water at room temperature and relatively high current densities, were produced which showed nearly a smooth enough surface to be useable and resistances in the order of hundreds of megohms. However, even though the system showed promise, the investi-

⁴Using Alodyne 1200S (Amchem Products, Inc.)

gation was dropped since only days remained in the available time for work to progress before the project was to have been concluded. Work was resumed on producing substrates using the chromatized-aluminum system to facilitate the possible solution of the more important plating, photoresist image and etching problems.

Samples were aluminized such that loss of adhesion and surface deterioration during chromatizing could be re-evaluated and several different immersion times, sealing, and drying techniques reviewed.

Next, a series of four tests were made to complete the evaluation of etching techniques and procedures. Successful substrates were anodized using the same bath containing 220 g/l H_2SO_4 , utilizing a liquid nitrogen dewar technique and increasing the current density to 250 ma/square inch.

These samples showed excellent surface quality, resistivity and provided improved adhesion at the nichrome-gold substrate interface. Therefore, a successful method for substrate production was accomplished.

A second successful method for substrate production was discovered when the evaporation of the aluminum was done with a crucible which was found to have contained residual amounts of copper from previous use. Because of the resulting alloy color, it had been incorrectly assumed that the samples had been chromatized and the nichrome-gold transition layer had been sputtered directly onto the copper-aluminum alloy with resultant improved adhesion equivalent to the anodized system. Later plating of these samples showed

absolutely no adverse effects from electrolysis of the aluminum-copper alloy layer. Therefore, two completely successful techniques were evolved for the production of the required substrate materials.

Thus, samples have been prepared using the techniques described above. Attempts to photoetch these materials with the fine-line (2-mil wide) image are being made.

4.0 CLOSED FLUX STRUCTURE TECHNIQUES

Early conferences with Lincoln Laboratory investigators led to an expressed interest in storage devices having a closed flux path either in the rest state or during switching. One technique had been devised for achieving this effect in a plated flat film structure. However, the technique required a double etch with careful realignment of the photoresist during the second etch, and thus, was not ideal for large area structures.

With the evolution of the nickel-phosphorus layer, however, the possibility arose for developing a single-etch process. The process involved the plating of a multilayer structure as shown in Fig. 1-a in cross-section. Applying a photoresist after developing an image, as shown in Fig. 1-b, results. After etching, the configuration in Fig. 1-c is achieved and finally after plating in a solution containing magnetic ions, the closed flux configuration (Fig. 1-d) is arrived at.

The configuration in Fig. 1-d may be used to close flux in either the easy or hard axis directions. The advantage of the former is a reduction in digit disturb, creep disturb, and an increase in density along the word line. Closing the flux in the hard direction, on the other hand, resulted in decreased transverse drive requirements and lower

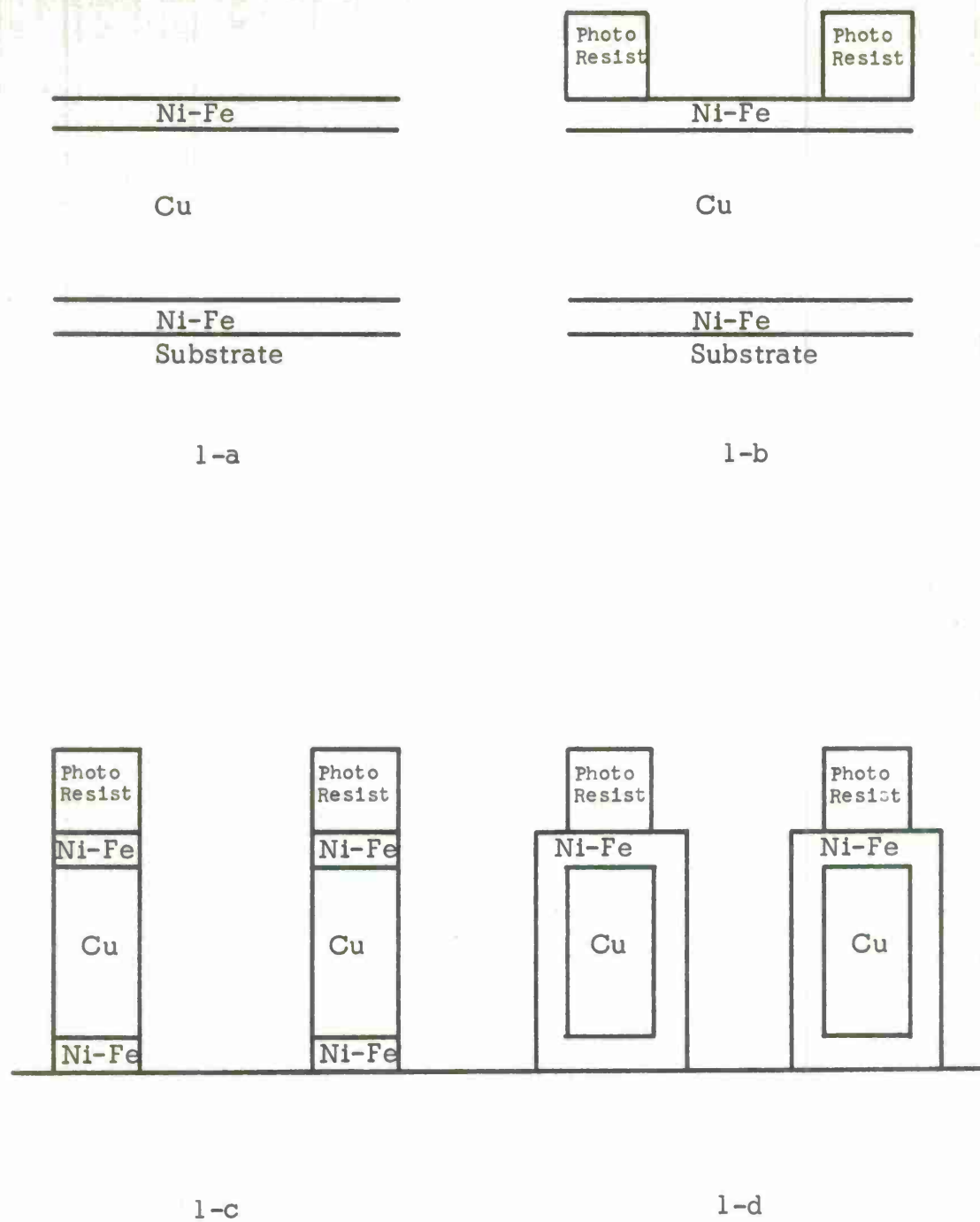


Fig. 1 Closed Flux Structure Preparation

magnetostatic "dispersion" as well as in increased density along the digit line.

The key consideration in attaining such a structure is the ability to deposit good thin anisotropic magnetic films on a thick (0.2 mil) conductor. As was indicated, the development of the nickel-phosphorus process made such a step feasible.

In order to test the technique, 0.2 mil of copper was electrodeposited on a 1-in. square glass substrate using an acid sulfate bath and a commercial brightener. The copper bath composition used was:

225 g/l $\text{CuSO}_4 \cdot 0.5 \text{H}_2\text{O}$
 56.3 g/l H_2SO_4
 4 ml/l UBAC* #1
 30 mg/l NaCl
 310 ma/in²
 (A bagged anode was used)

A layer 3000Å thick of nickel-phosphorus was deposited on the copper and followed by the 1000Å of permalloy deposited in a magnetic field. Results of hysteresis loop tests of a typical sample measured by T. Crowther of Lincoln Laboratory, are given in Table II. As can be seen from the results, a well-oriented film having a low dispersion was achieved. A number of such samples were prepared and etched into 10-mil strips on 20-mil centers. Flux closure was achieved in the way described above. However, the samples were not pulse tested.

Film No.	1
θ_1 (deg.)	0
α_q (ma)	5.0
α (90%) ma	13.0
H_d (oe)	2.4
H_c (oe)	3.0
H_k (oe)	5.3
$2\alpha_{\max}$ (mv)	120
100 mv/1000Å	

TABLE II

5.0 CONCLUSIONS

1. Results of switching tests on the Neel Wall sandwich structure show this structure to be quite promising for providing creep resistance for open flux bits.

2. In developing the 2 in. x 10 in. plane with 0.002 inch lines on a conducting ground plane several of the problems have been overcome including 1) the development of the high coercivity film, 2) uniformity of magnetic film properties, 3) development of a simple insulated ground plane substrate, and 4) development of a good copper-coating technique. Time was insufficient, however, to permit the solving of the problem of the fine-line photoetching of the film structure which had evolved.

3. The feasibility of fabricating a semi-closed flux structure was demonstrated.

6.0

SUMMARY

In the first half year, processes leading to the utilization of a new type of sandwich structure for "creep resistant" elements were developed.

During the second half of the year techniques for photo-etching the sandwich structure were developed and three memory planes containing 448 bits each were fabricated and pulse tested. Results indicated a promising, creep resistant capability for open flux structures but further development is required for high yield of good planes.

Techniques for producing a large ground plane structure (2 in. x 10 in.) with a high coercivity film were developed.

Feasibility of producing a semi-closed flux structure was shown.

Investigation of two types of inverted film structures has been carried out and preparations have been made for depositing large area planes of magnetic films.

APPENDIX I

Photoresist and Etching Procedures

1. Photoresist - Kodak Metal Etch Resist (KMER).
2. Spin coat at 800-1000 rpm.
3. Air dry for one hour.
4. Bake image for 15 minutes at 180-200°F.
5. Expose with P.E.K. Labs mercury arc vapor lamp 2 minutes.
6. Develop in KMER developer for 2 minutes.
7. Spray rinse with KMER developer from atomizer.
8. Rinse in aeriated tap water.
9. Air dry for 15 minutes.
10. Bake in image for 15-30 minutes at 180-200°F.
11. Electrolytically etch in a solution containing 500 ml of 10% HNO_3 /8 g NaCl, with a current density of 1-1.5 amps/sq. in. Completion of the etch should occur in 15 seconds or less. The rapid etch rate reduces the etch factor, undercutting, to nearly zero.

Due to the loss of electrical contact, residual material will be left in the exposed regions. This material can be successfully removed by immersion in a fresh solution of nitric acid, 1/1 - $\text{HNO}_3/\text{H}_2\text{O}$. This etch should not exceed 10 seconds. Small amounts of residue may still remain, but further etching will cause a rapid undercutting in the regions which should remain.

Alternative Chemical Etching Procedure

Immerse samples in a fresh nitric acid solution, 0.5/1.0 - $\text{HNO}_3/\text{H}_2\text{O}$. This etch should not require more than 30-45 seconds immersion time and will most often result in increased undercutting with respect to the electrolytic process.

APPENDIX II

Photoresistive Chemical Etching Procedure for 2 in. x 12 in. Memory Planes

1. Due to the high etching rates (etchant concentrations or current densities) which must be employed to etch these samples with satisfactory etch factors (undercutting), it is necessary to use a very thick photoresist image. The photoresist image thickness is the basis of the necessity for the stringent control as outlined in the image preparation procedure.
2. A check of the prevailing air temperature and relative humidity should be made prior to using these procedures since a definite increase in efficiency and decrease in the procedural control will be found if the room temperature is slightly higher than the usual 20°C and the relative humidity is close to 50%. The conditions prevailing when this procedure was established were at room temperature of 19°C, and a relative humidity of 70%. In particular, these two factors combined, force extremely long air drying times due to the low thermal energy available and the small change in composition which occurs under these conditions due to the water miscibility of one of the photoresists.

Procedure I
for
Photoresist Image Preparation

1. Prepare by mixture and filtration 900 ml of photoresist containing 2 parts KMER³, one part KTFR and one part KMER thinner¹.
2. Draw coat the photoresist mixture onto the samples at a rate of approximately 3 in./min¹.
3. Air dry with forced room temperature air for at least four hours².
4. Prebake this film for no longer than 15 minutes at a temperature not in excess of 185°F².
5. Place in a vacuum exposure frame; position desired image negative over sample and evacuate until best possible contact is achieved between sample and negative¹. (If this requires considerable time, do it in total darkness.)
6. Expose sample with a high intensity light source such as a PEK high pressure mercury-arc vapor lamp (with the PEK model 701 at a source to image distance of 3 feet, optimum exposure time will be 20 minutes.

¹Lighting provided by fluorescent or tungsten sources, covered by Kodagraph yellow or orange sheeting, can be used without effect on resulting image quality during these periods.

²Due to the time required, or the temperature prevailing, these procedures must be done in total darkness.

³Photoresists which are a product of Eastman Kodak Company, Rochester, New York.

⁴PEK Labs, Inc., Sunnyvale, California

7. Develop image by immersion in Stoddard solvent for one minute, followed by a thorough spray rinse development with clean Stoddard solvent for one minute¹.
8. Thoroughly rinse with cool, aireated, tap water until all signs of residual solvent and dissolved photoresist are gone¹.
9. Blow off excess water with contaminant-free compressed air¹.
10. Air dry for at least one hour using forced room temperature air².
11. Postbake image for 30 minutes at a temperature not in excess of 185°F.

Procedure II
for
Photoresist Image Preparation

Note: This procedure will provide a thicker more reliable photoresist image.

1. Prepare by mixture and filtration 900 ml of photoresist containing two parts KMER, one part KTFR and one part KMER thinner^{1,3}.
2. Draw coat the photoresist mixture onto the samples at a rate of approximately 3 in./min.¹ (from a 1000 ml graduate).
3. Air dry with forced room temperature air for at least four hours².
4. Prebake this film for no longer than 15 minutes at a temperature not in excess of 185°F².

For footnotes see page 24.

5. Prepare by mixture and filtration 900 ml of photoresist containing four parts KPR, two parts KPL and one part KPR thinner^{1,3}.
6. Draw-coat this photoresist mixture at a rate of approximately 4 in./min. (from a 1000 ml graduate).
7. Air dry the composite film for at least three hours using forced room temperature air².
8. Prebake the composite image for no longer than five minutes at a temperature not in excess of 185°F².
9. Place the samples in a vacuum exposure frame, position desired image negative over the sample and evacuate until best possible contact is achieved between sample and negative¹. (If this requires considerable time, do it in total darkness.)
10. Expose sample with a high intensity light source such as a PEK high pressure mercury arc vapor lamp (with the PEK model 701 at a source to image distance of 3 feet optimum exposure time will be 20 minutes).
11. Develop the upper portion of the composite image by immersion in KPR thinner for one minute followed by a thorough spray development with clean KPR thinner for 30 seconds¹.
12. Thoroughly rinse with cool aereated tap water until all signs of residual developer and dissolved photoresist are gone¹.
13. Blow off excess water using contaminant-free compressed air¹.
14. Air dry for 5-10 minutes using forced room temperature air¹.

For footnotes see page 24.

15. Develop the lower portion of the composite image by immersion in Stoddard solvent for one minute followed by a thorough spray development with clean Stoddard solvent for one minute¹.
16. Thoroughly rinse with cool, aireated tap water until all signs of residual developer and dissolved photoresist are gone¹.
17. Blow off excess water using contaminant-free compressed air¹.
18. Air dry for 15-30 minutes using forced room temperature air¹.
19. If desired, dye image by immersion in KPR dye, black or blue, for 15-20 seconds and rinse thoroughly with cool aireated tap water¹.
20. Postbake the image for 30 minutes at a temperature not in excess of 185°F.

For footnotes see page 24.

Procedure I
for
Chemical Etching of Structures on
Chromatized Aluminum, Copper
Aluminum Alloy or Anodized
Aluminum Substrates

1. Prepare a fresh nitric acid solution of 50% by volume HNO_3 ,
1 HNO_3 /1 H_2O .
2. Etch samples to the gold layer by rinsing with the nitric acid
solution applied from a hand pressurized wash bottle. (It may
be necessary to use alternating rinses of the HNO_3 solution and
a 30% by volume solution of ferric chloride; it has been found that
the nickel-phosphide content in the nickel-phosphorous layers
will vary to some degree and it is most easily removed by
alternate rinses. However, caution must be exercised since the
ferric chloride rinse will tend to lift the photoresist image
and cause severe under cutting during following applications of
the HNO_3 solution.)

Note: Stopping the etch by thoroughly rinsing in water gives rise
to no damaging effects and is very helpful by allowing careful
observation of the etch progress.

3. Etch through the gold layer by using a very rapid, 1-2 second,
rinse of aqua-regia. (A period in excess of this will cause
drastic lifting and undercutting of the upper structure.)
4. Etch through the aluminum layer using a concentrated solution of
sodium hydroxide. (Although care should be taken, this step
in the procedure usually does not require extreme control.)

5. Rinse immediately and thoroughly in tap water to stop all etching activity and dry.

Note: For etching of samples on anodized aluminum, simply delete step 4.

Procedure II
for
Electrolytic and Chemical Etching of
Structures on Chromatized Aluminum,
Copper Aluminum Alloy, or
Anodized Aluminum Substrates

1. Prepare an electrolytic etching solution containing 8 g NaCl, 2 g $\text{NaC}_{12}\text{H}_{25}\text{SO}_4^1$ and 500 ml of 10% HNO_3 in H_2O .
2. Electrolytically etch the uppermost nickel-phosphorous layer, at a current density of 200 ma/sq. in., or approximately 3.0 amps for an exposed region 6 in. long, containing the 2.0-mil line array.

Note: 1. Watch this etch step closely, and when the copper layer starts to be exposed or when only the black nickel-phosphide residue remains, immediately stop the etch. 2. A pulse plating technique in which current flows for five seconds out of each 15 seconds will complete the etch to the desired point in five minutes. 3. Continue to electrolytically etch only if a very uniform etch is proceeding and no lifting of the image or undercutting is apparent. All of the proceeding factors are dependent upon the adhesive quality of the photoresist image and the degree of residual contamination in the image.

¹Sodium Lauryl Sulfate

However, under no circumstances allow the sample to remain in the bath over eight minutes since the wetting agent and acid will by this time begin to cause lifting of the image and longer immersion will therefore result in undercutting.

3. The etching can, at this point, be finished by following the procedure outlined for chemical etching. However, the eventual image quality can be expected to be generally better using this system since most of the material to be etched is removed with little or no undercutting occurring.

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