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PROCESSES AND TECHNIQUES FOR FABRICATION OF MAGNETIC PLATED WIRE

by

William Wade
Gordon Sands

October 1968

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TECHNICAL REPORT ECOM-3029

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Electronic Components Laboratory

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ABSTRACT

This report details a facility for the fabrication of quality permalloy plated wires for computer memory applications. A description of the sequence of steps for a continuous plating system is included. Related parameters pertinent to production of reproducible uniform films and reduction of mechanical stresses are discussed; e. g. memory element, electrolytic cleaning, electropolishing, substrate surface, thickness, bath composition, and analytical techniques.

The instrumentation necessary for measurement of switching time is described briefly.

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PROCESSES AND TECHNIQUES FOR FABRICATION OF MAGNETIC PLATED WIRE

OBJECTIVE

The objective of this report is to indicate the level of achievement in the development of a continuous plating technique to be utilized for the fabrication of magnetic alloy films. This process, which is phase one of a permalloy film study, is considered to be the most feasible for obtaining lower and more uniform values of coercive force (2.5 oersteds) and anisotropy (5.0 oersteds). The coercive force and anisotropy along with a small magnetostriction determine the characteristics necessary for utilizing magnetic-film elements as memory devices. Such properties provide the means for nondestructive readout.

Phase two and subsequent phases will deal with the results and data obtained with investigating the binary alloy 81 Ni-19 Fe, ternary alloys 4 - 79 molybdenum, phosphorus-nickel-iron, etc. made possible through the development of this continuous multistep operation.

INTRODUCTION

When a non-magnetic wire is electroplated with a magnetic thin film in such a manner as to establish a circumferential magnetic easy axis, the film is capable of providing a nondestructive readout mode. The plated wire acts as its own sense line and information write line. The information is stored by clockwise or counterclockwise magnetization of the film which represents a stored 1 and a stored 0 position, respectively. A small current writes the information into the wire which provides the circumferential field used for steering the magnetization in the desired direction. The magnetization vector rests in the 1 or 0 position, dependent upon the direction of the bit current. The bit current in the wire is time-coincident with a word drive current flowing through a word strap at right angles to the plated wire. In order to accomplish nondestructive readout, a word drive field is applied which rotates the magnetization vector less than 90° from the easy axis. The amplitude of the word current is controlled so that when the current is turned off, the magnetization vector returns to its original rest position under the influence of the anisotropy and demagnetization fields. It is to be noted that the word drive current used for reading stored information has the same polarity and amplitude as for writing in new information.

Figure 1 shows information storage on plated wire which illustrates drive currents and resulting magnetic fields during information handling. Read and write operations are also portrayed which illustrate drive currents and plated-wire signals plotted against time.

Ferrite cores and planar thin films have been in the forefront for memory applications; however, the plated-wire technique may replace them in many memory applications. Plated wires are potentially more economical to produce, can be switched rapidly, and can be operated in a nondestructive readout mode.

To fabricate wires suitable for memory application, the effecting of a smooth, clean substrate surface, control of substrate residual stress, and amount of stress in the deposit must be given primary consideration. Investigation has shown that the best method for controlling these conditions is through a continuous plating process which, as described in this report, is subdivided into a substrate preparation process and a permalloy deposition process.

PRELIMINARY CONSIDERATIONS

1. Memory Element

The most likely configuration would be a solid permalloy wire, but this has a poor device geometry which does not provide uniform switching field at all radii. A tubular geometry is more desirable, but it is impossible to draw tubes of required wall thicknesses. A satisfactory tube geometry may be obtained by electrodepositing a thin magnetic plating on a non-magnetic wire. The present element consists of a wire substrate made of beryllium-copper drawn to a diameter of 0.005 inch, having minimum roughness and maximum randomness of orientation upon which the Ni-Fe alloy is electrodeposited.

2. Substrate Preplate

After removal of organic soil and loose dirt with an alkaline cleaner, and surface oxide with an acid dip, the Be-Cu wire still shows pits and small scratches under microscopic examination. A one-micron (10,000 angstroms) magnetic film plated onto this surface with no further preparation will sharply increase the values of dispersion and minimum bit writing current, depending on the local surface roughness of the substrate.[1] Therefore, a metallic preplate having randomness of orientation is used to smooth the surface although it does not eliminate surface imperfections completely. Both copper from a commercial plating bath and gold from a commercial proprietary plating bath have been employed in this study.

3. Thickness Measurement

Presently, the amount of preplate and permalloy are determined by a combination of the average thickness method and speed of the wire through the system.[2] A B-H loop tracer is being fabricated which will

show the flux characteristics (squareness ratio, coercive force, and anisotropy) of the plated wire. By comparison with a plated wire of known film thickness, it will be possible to estimate quickly the thickness of the in-house produced samples.

4. Analytical Techniques

A spectrophotometric technique has been used for the quantitative microanalysis of the nickel and iron content in electrodeposited nickel-iron alloy films, but this method proved to be time-consuming and impractical. For the determination of nickel, the dimethylglyoxime oxidizing agent method was used and the absorption of the solution measured at the wavelength of minimum transmission (4750 Å). For the determination of iron, the 1, 10-phenanthroline complex method was used, and the absorption measurements made at a wavelength of 4900 Å utilizing a Beckman Model DU spectrophotometer in both cases.

Presently, a microprobe technique is being used whereby an X-ray beam probes the surface of a standard nickel-iron permalloy, and qualitatively compares the nickel and iron intensities with those of the prepared test sample. With more complex manipulations, the microprobe can tell the exact quantitive ratios, and is being investigated to develop this capability.

Figure 2 shows the type of switching time pattern that can be obtained with this facility and is measured by a laboratory modification of standard techniques. An oscilloscope is used to read the switching time. Bare Be-Cu wire is used for noise cancellation and a printed circuit for the word strap.

5. Bath Composition

Iron and nickel can be codeposited from aqueous solution because their standard potentials lie close together. Iron behaves anomalously depositing from solution at a much faster rate than the more noble nickel; therefore, one can expect to find it in lesser proportions in the bath solution. The composition of the alloy is controlled by adjusting the relative concentration of the individual salts in solution. To obtain an 81 Ni-19 Fe permalloy film, the ratio is approximately 80 grams of nickel to 1 gram of iron from a salt in solution. Both nickelous and ferrous sulfate, and nickelous and ferrous sulfamate salts have been used. The parameters of additives, pH, temperature, and agitation also play important roles in the permalloy film deposition. Boric acid has been employed as a buffering

agent for controlling pH, and the sodium salt of a naphthalene sulfonic acid for reducing stress in the film. The temperature range studied was between 50 - 55°C, pH 2.9 to 2.5, and the agitation provided by a circulating pump at approximately 1700 milliliters per minute.[3, 4, 5]

PERMALLOY FILM PROCESSES AND TECHNIQUES

1. Early Methods

The first attempt at producing quality films can be considered a stationary method as the cylindrical wire substrate was attached to a plastic holder which was inserted by hand into a plastic container (Fig 3). It was found necessary to subject the wire to a series of treatments involving cleaning, deposition of a preplate, and finally permalloy plating. None of these operations involved circulation of solution. This multistage handling produced irregular and rough surfaces which resulted in inherent mechanical stresses, non-reproducibility, and non-uniformity of switching.

During electrodeposition, which involves reduction of nickel and iron ions from solution to a free metal at the cathode solution interface, a cation depletion region occurs in the vicinity of the cathode. Agitation influences this depletion layer which in turn varies the plate composition. Therefore the second attempt at producing quality films incorporated a laboratory-designed plating cell (Fig 4) for providing circulation of the plating solution. However, the results with this semi-continuous operation were no better than with the previous stationary method.

2. A Continuous Plating System[6]

Acceptable plated wires were produced using a continuous plating system. The treatments required for high quality are accomplished by passing the wire continuously through a series of treating cells where cleaning, straightening, polishing, gold-plating, and nickel-iron plating are carried out under controlled conditions. The system is divided into a substrate preparation process (Fig 5) in which the wire-feed rate is 9 inches per minute, and a permalloy deposition process (Fig 6) in which the wire-feed rate is 6 inches per minute.

Current is provided by power supplies located along the process path. A refurbished 24-inch bicycle wheel rotated by a motor winds the gold-plated wire during the substrate preparation process. The same wheel with the gold-plated wire is fitted into the permalloy deposition process and the wire is pushed through the system by means of a set of cylindrical rollers and adjustable speed motor control unit.

a. Substrate Preparation Process

The plating system consists of a series of identical cells, each two inches wide, two inches long, and three inches high. These cells

are machined from a solid lucite block and contain Teflon inserts for guiding the wire through the system. The inserts are attached to a Teflon plating chamber designed to give uniformly high agitation along the entire length of wire being operated upon. A centrifugal pump forces the electrolyte or rinse water through an inlet tube through the plating chamber(Fig 7) which has three staggered rows of holes drilled through the wall. The solution is forced through these holes and impinges on the surface of the moving wire at high velocity. It is removed through a low impedance discharge reservoir that connects with a drain or an electrolyte vessel. The electrolytes are held in vessels fitted with heaters and temperature controls.

During this process, a 5-mil beryllium-copper wire acting as cathode or anode is pulled through the system, and is subjected to the following treatments:

STEP 1 - Electrolytic Cleaning: The selection of an appropriate cleaning method depends on the type and quantity of the soil, metal composition and surface texture, and the degree of cleanliness required. In electrolytic cleaning, the generation of large quantities of gas close to the soil is effective in providing a high level of mechanical agitation, which is known as scrubbing action. In this step the gas is generated at the cathode or beryllium-copper wire surface. Oakite 191, a commercial detergent, is used in the amount of 60 gram per liter at 53°C.

STEP 2 - Straightening: The commercially prepared tempered 0.005-inch-diameter beryllium-copper wire appears to have an inherent twist and strain which is best removed by an annealing process. The annealing is accomplished after cleaning and water rinsing by moving the wire, which is under slight tension, through a furnace, at least 13 inches long, at 540°C, with an atmosphere of forming gas (88% nitrogen-12% hydrogen).

STEP 3 - Electropolishing: Oxide scale, pits, and deep scratch marks produced during annealing contribute to bad spots on the wire and are removed through a continuous electropolishing of the wire. The wire is made anodic in an electrolyte containing 18 grams of cupric pyrophosphate per liter of 85% ortho phosphoric acid. A copper-wire gauze cylinder used as the cathode is placed in a glass unit 4 inches in diameter and 0.3 mil of material is removed from the surface. The electrolyte temperature is 60°C and polishing current 450 mA. Figure 8 shows an electron microscope shadowgraph of the surface of the wire obtained from this polishing.

STEP 4 - Gold Plating: After rinsing, approximately 7000 angstroms of gold is deposited on the wire to form a substrate which does not produce

epitaxial growth in the subsequent permalloy deposition. A commercial acid-type electroplating formulation, which produces gold coatings of 99.9% purity, having hardness values between 130 and 170 knoop, was used. The plating current is set at 25 mA and bath temperature at 35°C. Figure 8-1 shows an electron shadowgraph of the gold-plated wire.

b. Permalloy Deposition Process

During this process, the gold-plated wire is cleaned electrolytically and a nickel-iron film approximately 10,000 angstroms in thickness is deposited. A 0.5 amp current equivalent to 15.7 oersteds through the wire during the deposition causes circumferential orientation of the magnetic easy axis of the film. The plate current was at 22 mA while the bath temperature employed to date was 50°C.

CONCLUSION

A laboratory plating apparatus has been established for producing continuous plated wire on an experimental basis. This facility uniformally reproduces high quality permalloy films (81 Ni - 19 Fe) on beryllium copper wire having near zero magnetostriction and comparable to the better plated wires produced by Industry. It was also found possible to eliminate the cooling process step in this apparatus due to the extremely close control of the electropolishing step which follows.

This facility will be used to prepare other promising permalloy films such as the ternary molybdenum alloy (known as 4-79 permalloy) and nickel-iron-phosphorus. It is anticipated that these alloys should have improved "creep" and aging characteristics which presently limit the packaging density and the read-write cycle time of plated wire memories. (Creep is the localized magnetization reversal caused by the cumulative effect of small disturb fields which are individually too weak to switch the magnetization in a single application.)

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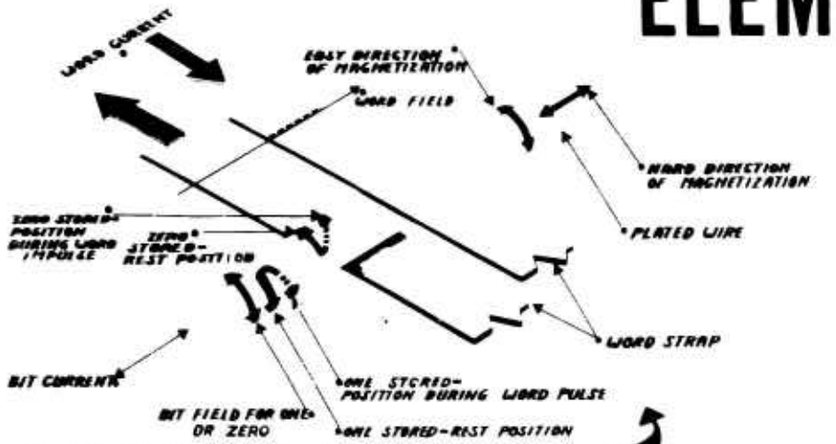
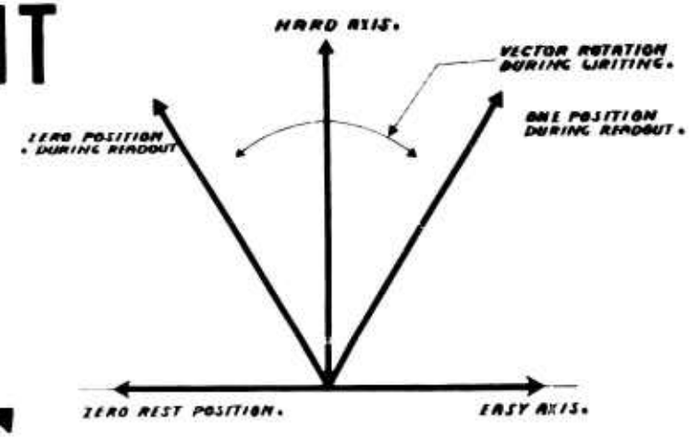
PLATED WIRE MEMORY ELEMENT



INFORMATION STORAGE ON PLATED WIRE.



READ AND WRITE OPERATIONS.

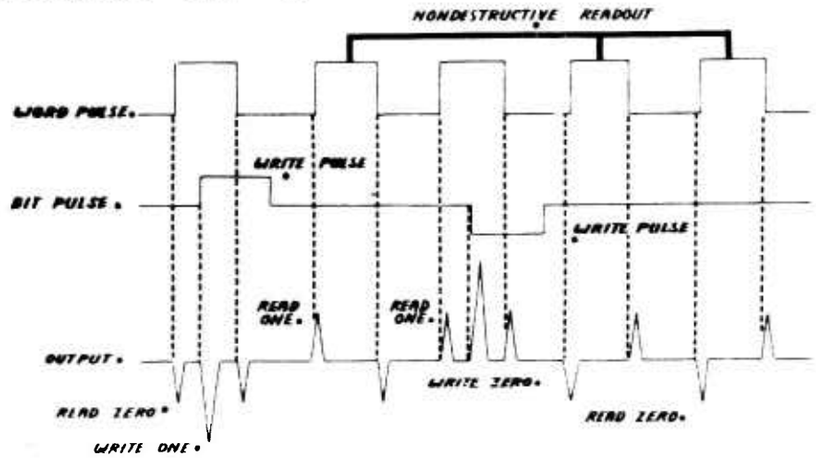


Fig. 1

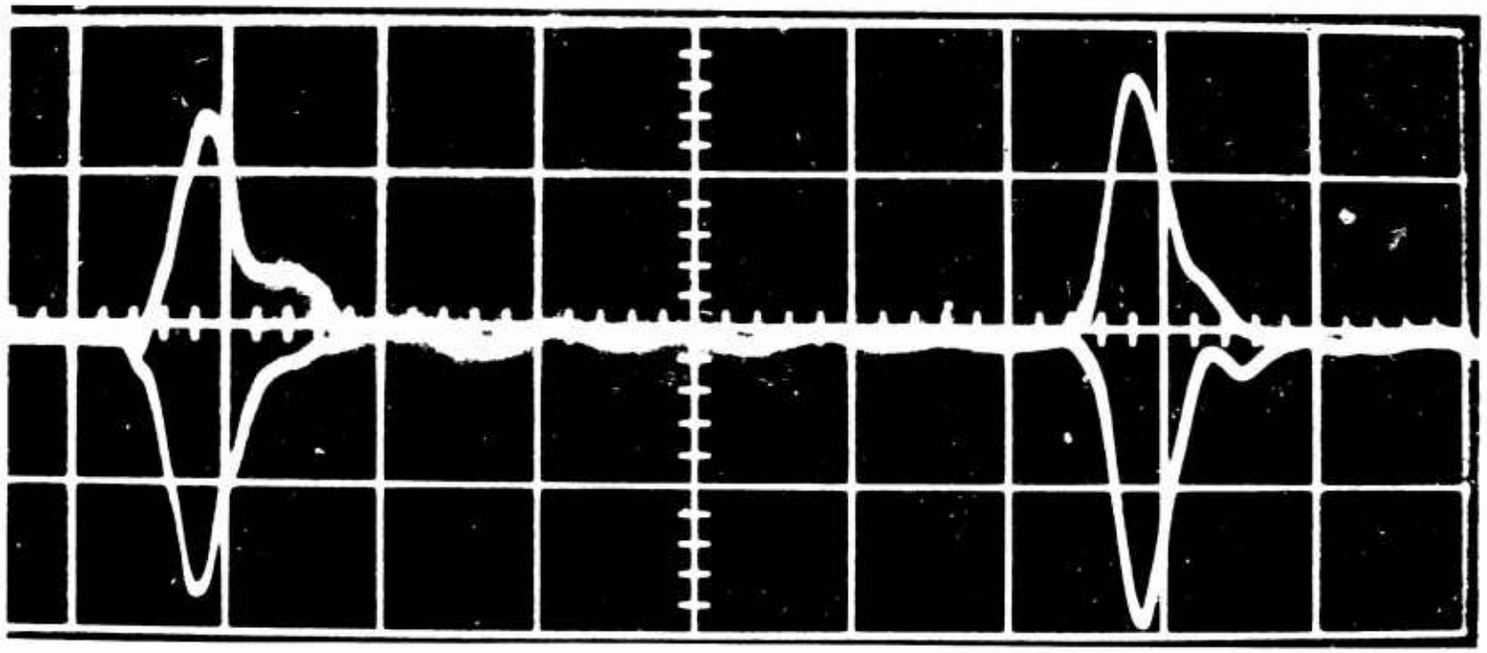


Fig. 2 Waveform of Plated-Wire Output

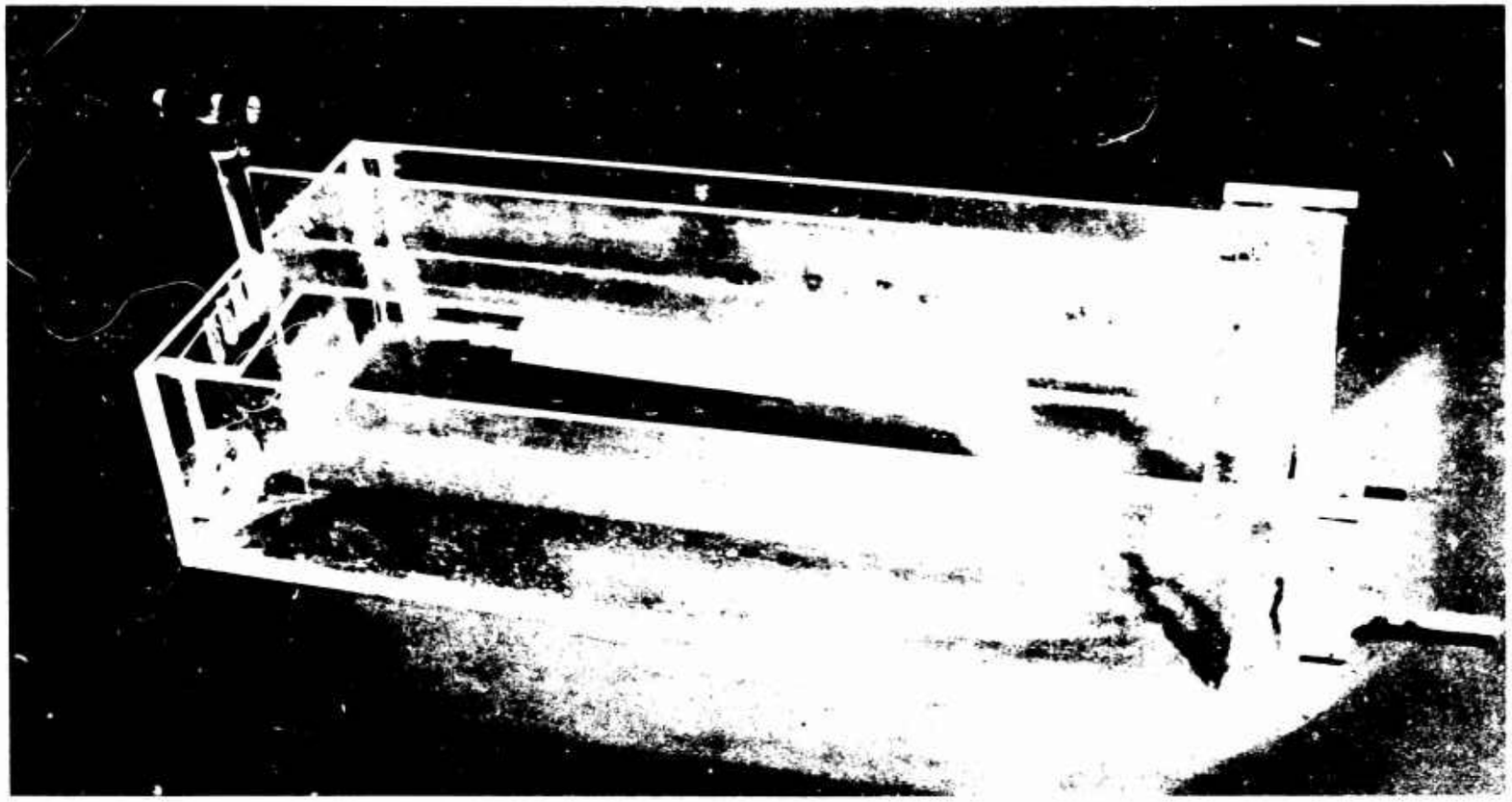


FIG 3 METHOD I - STATIONARY

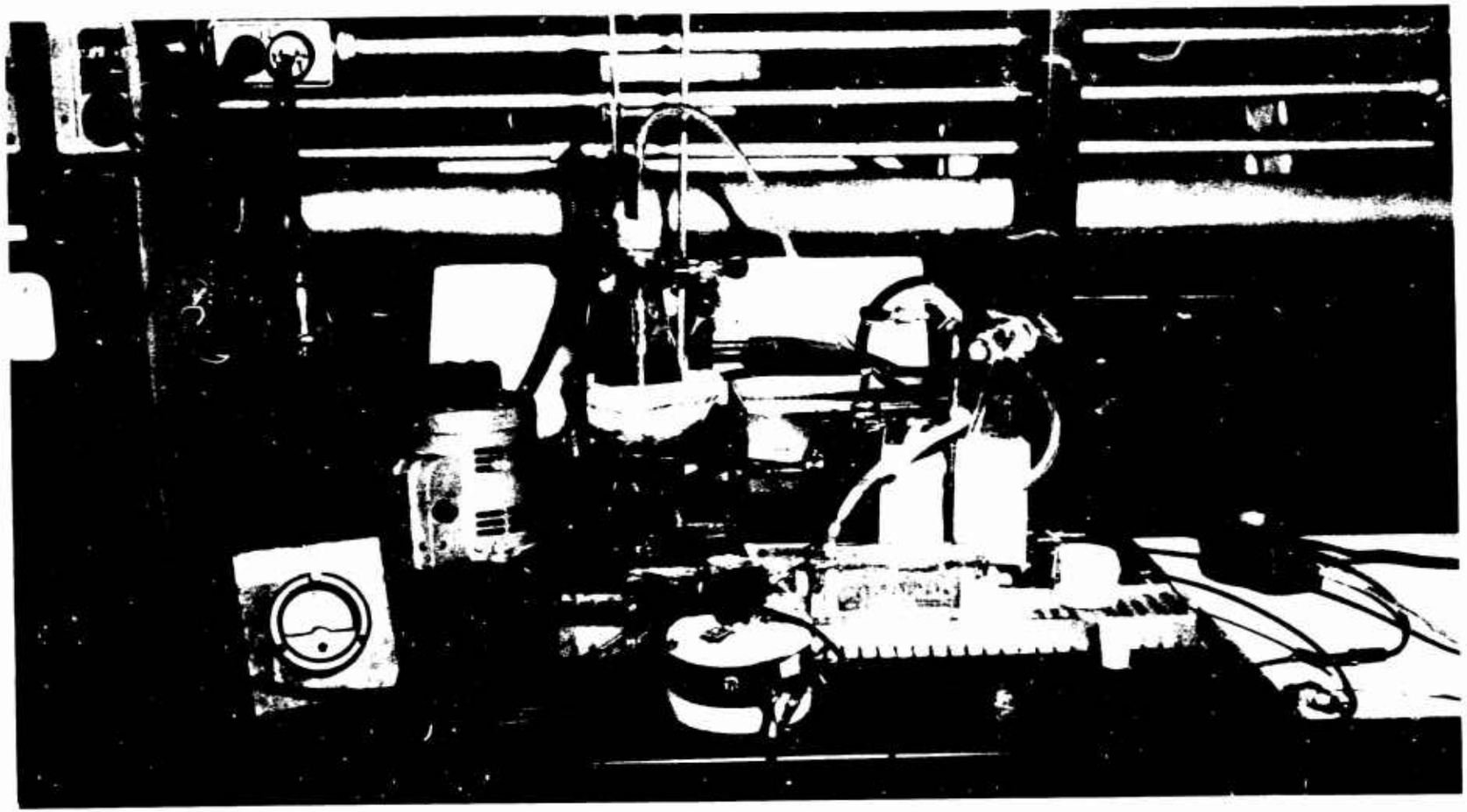


Fig. 4 Method II Semicontinuous

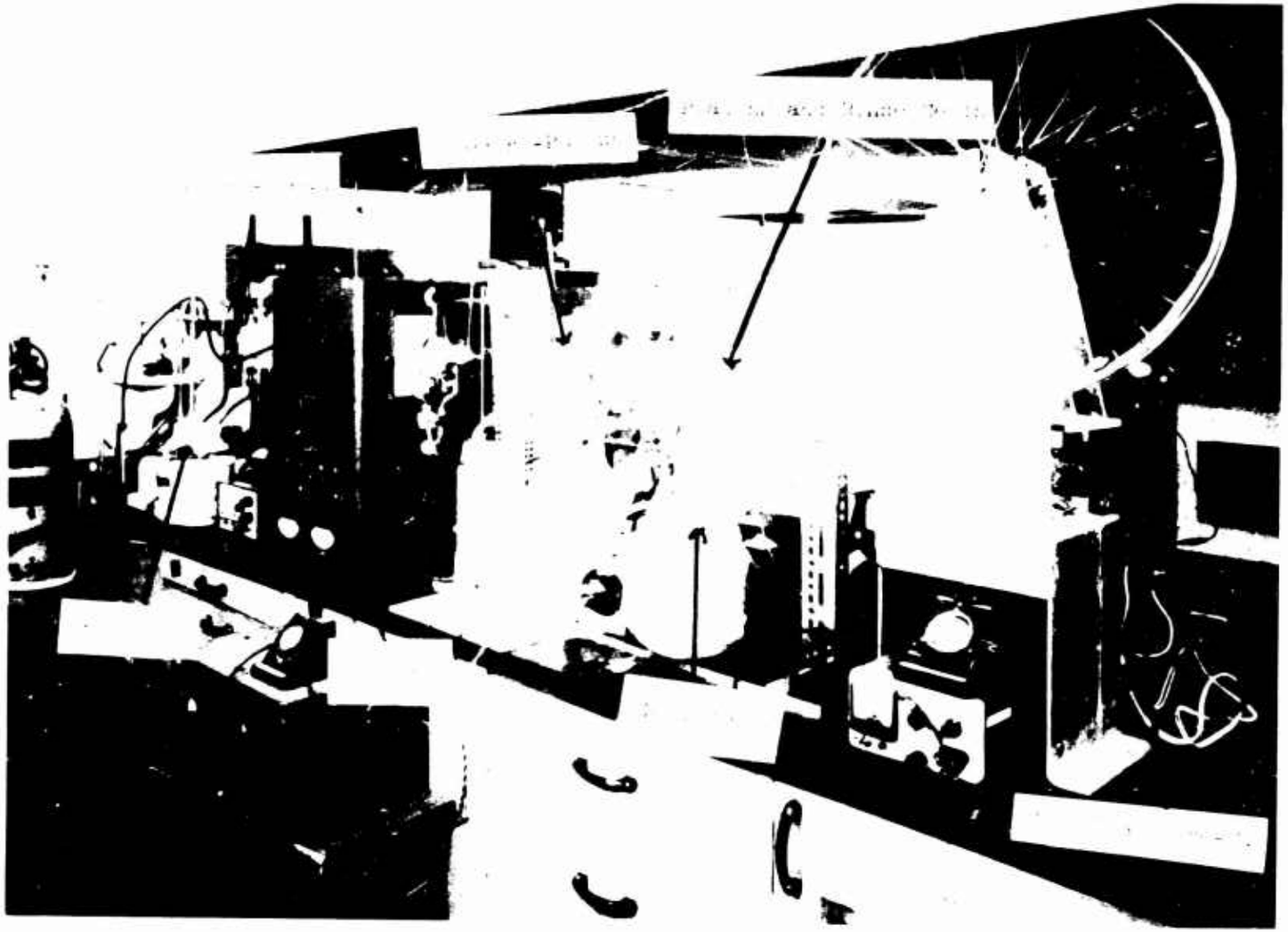


Fig. 5 Substrate Preparation

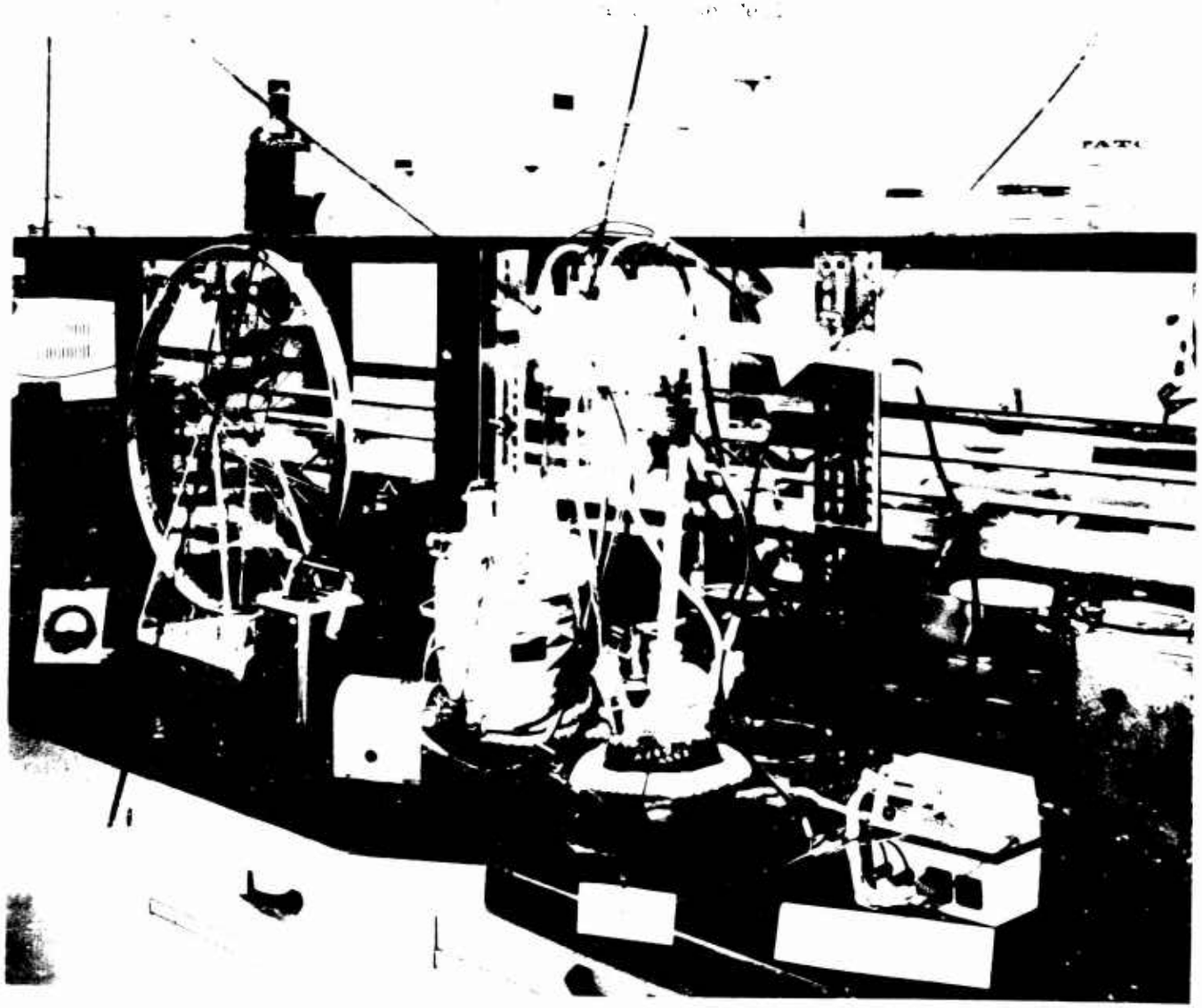


Fig. 6 Permalloy Deposition

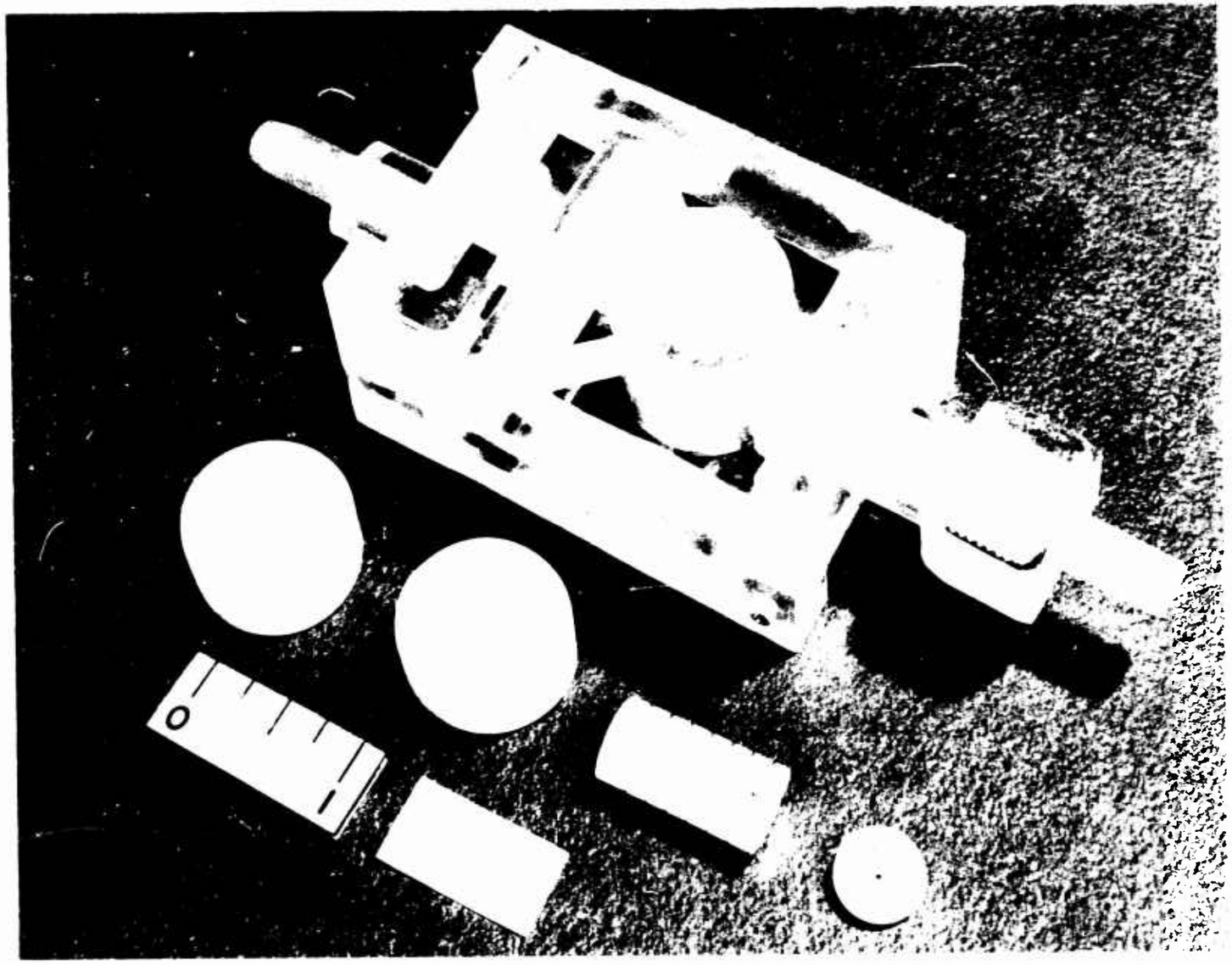


Fig. 7

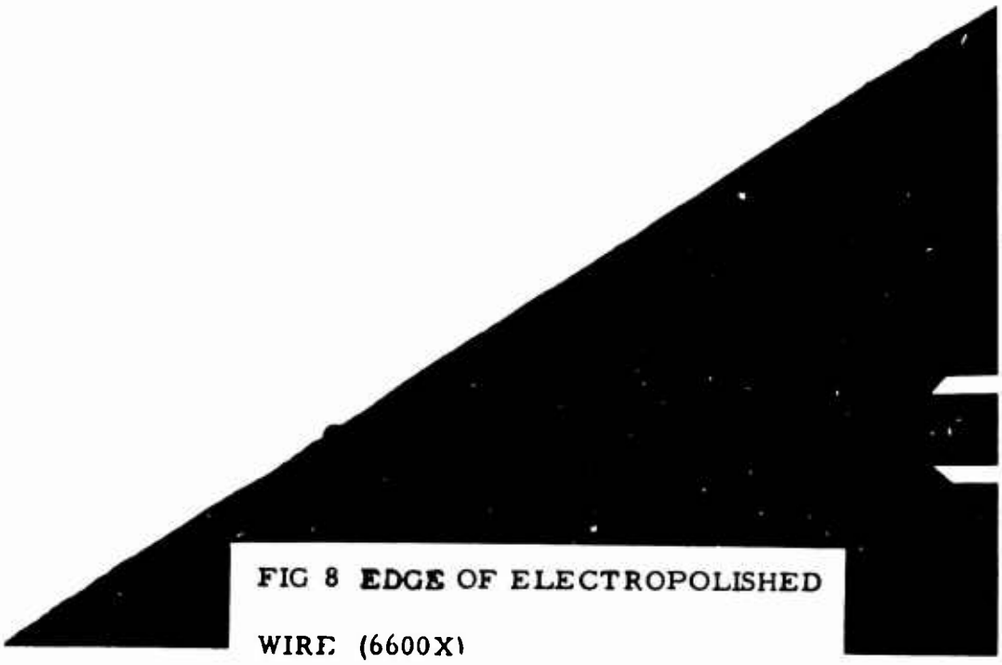


FIG 8 EDGE OF ELECTROPOLISHED WIRE (6600X)

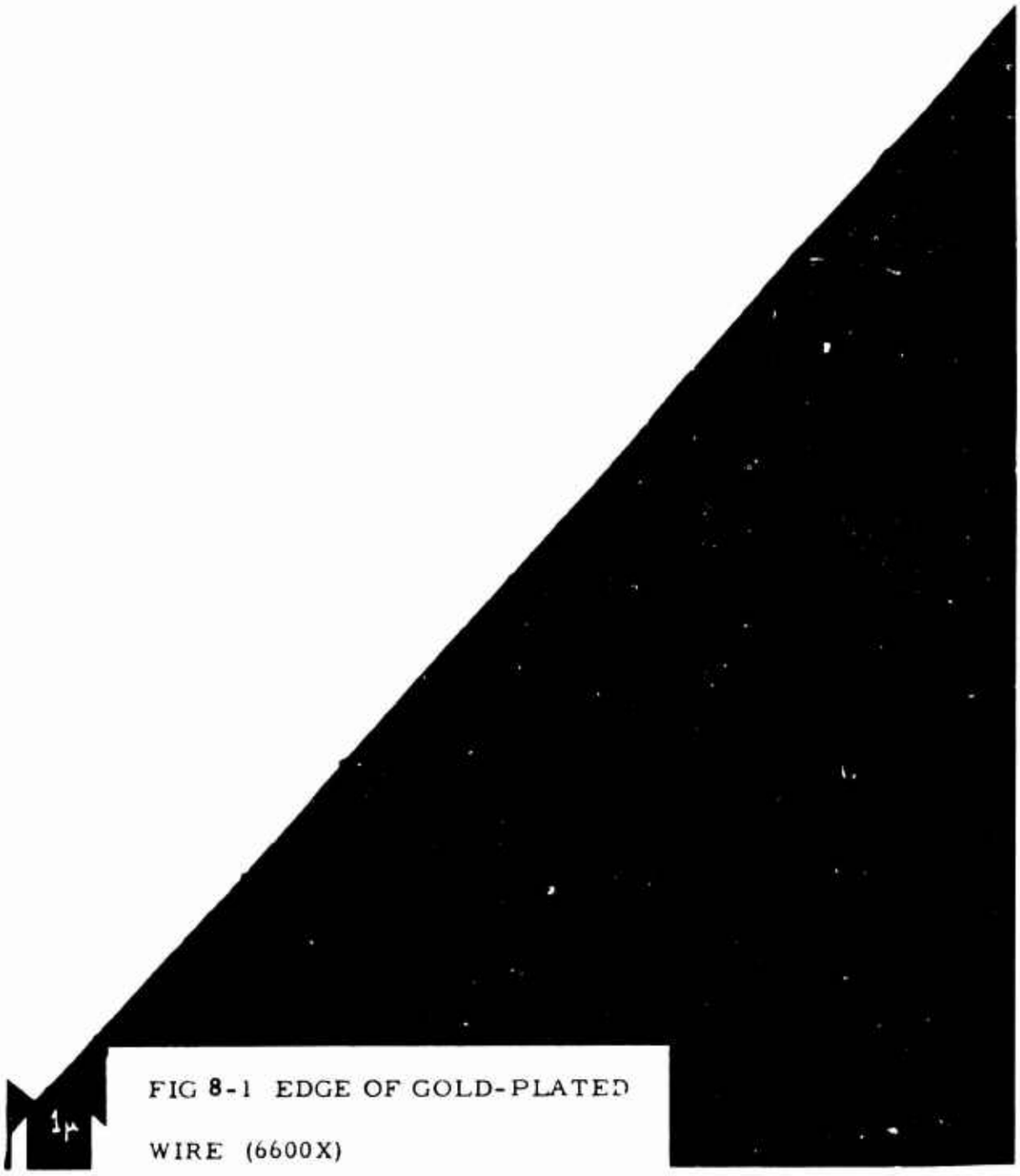


FIG 8-1 EDGE OF GOLD-PLATED WIRE (6600X)

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The fabrication of instrumentation necessary for measurement of switching time is described briefly.

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Permalloy						
Nickel-Iron Alloys						
Permalloy Films						
Plated Wire						
Magnetic Film						
Cylindrical Magnetic Films						
Memory Element						

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