Electrodeposion of the Interior Surface of 2.0 Inch I.D. Tubes 36 Ft. in Length and Cell Geometry Designed to Generate Plating Waveforms

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Techniques are described for copper plating the inside of 2.0 Inch low carbon steel tubes in lengths up to 36 feet. Uniform plating thickness throughout the tube has been achieved while varying: (1) average thickness of the copper deposit, (2) type of copper cyanide electrolyte, and (3) repetitive use of copper anode. Periodic reverse techniques result in uniform diameter control and retards introduction of surface roughness during copper plating. Plating with rotating cathodes produces more reproducible uniform thickness control than with a stationary anode and cathode. By varying the cathode area of contact of the dielectric member between the anode and cathode and the rotation speed of the cathode various interrupted plating waveforms can be generated.

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INTRODUCTION

The millimeter waveguide system, has been evaluated as a long haul, high density transmission medium consists primarily of hollow tubes used in propagating the transmitted signals. Previous research has proved that transmission losses are minimized when the internal geometry of individual waveguide sections varies little from that of a right circular cylinder. The Western Electric Research Center, Forsgate Laboratory has processed the helix and dielectric millimeter waveguides for use in a thirty mile experimental installation at a Bell Telephone Laboratories facility(1). The internal diameter of steel waveguide tubes must be copper plated in order to provide an adequate path for the propagated signals. Since plating involves geometrical changes, it is obvious that it has a direct bearing on the transmission efficiency of the above system. The plating methods discussed in this report were developed especially for the dielectric millimeter waveguide system. Other processing steps have been developed to assure good adhesion of the plated copper to the polyethylene dielectric liner(2).

PRINCIPAL OBJECTIVES

The members of the research staff at ERC charged with the development of applicable plating techniques and facilities were required to find ways of depositing a thin layer of copper on the 2 inch bores of low carbon steel tubes exceeding 16 foot lengths such that:

1. the average diameter is maintained at 2.0079 + 0.0004 inch,
2. the maximum diameter variation after plating is less than $\pm$ 0.0006 inch,
3. the technical feasibility of plating long lengths is determined,
4. the finish of the plated surface be less than 35 micro inches RMS,
5. the copper layer be continuous, nonporous and have high conductivity,
6. the deposited copper have excellent adhesion,
7. pre-plating chemical cleaning cause no deterioration of the precision steel tubes,
8. the process be suitable for future production with low cost and high reliability,

Vendors were investigated. Most did not have the technical ability to do the work, and the two which might have been able to plate the tubes quoted unacceptable prices. It was also considered highly desirable to develop an in-house plating technique to determine process capability and effects.

DESCRIPTION OF PLATING EQUIPMENT

The basic components of a facility for electrodeposition of copper on the interior surface of circular steel tubes include a round copper rod inserted into the tube, plating solution (in this case CuCN) pumped through the resulting annulus and a rectifier connected to the copper rod and the steel tube such that the former becomes the anode and the latter the cathode.

Vertical Plating

Initially, plating 18 foot lengths of dielectric millimeter waveguide tubes was done as shown in Figure 1 with the basic components incorporated into a temporary wooden structure. A 1 inch O.D., 5/8 inch I.D. copper anode was inserted into the steel tube and both secured to brackets and to modified commercially available polypropylene Y-branches. The branches in turn were connected to the plating tank with vinyl hoses and PVCD pipes. In order to centralize the anode with respect to the cathode, the loaded fixture was hoisted up to a vertical position before plating. The CuCN solution was pumped through the tube from the bottom up at approximately 25 gpm.

Horizontal Plating

While the vertically plated tubes showed rather good diameter control, several problems arising from this operation detracted from its original attractiveness. Maintaining the critical concentricity between anode and cathode became more difficult with increasing tube length. Handling and the fact that the Forsgate Laboratory ceiling height limited the tube length to 18 feet were other important considerations. It was known that, with activation polarization deposition, rotation of the tube being plated would result in both improved circularity and more uniform axial plating regardless of the position of the tube. In view of this, it was decided to evaluate the feasibility of designing and building a facility for plating the dielectric millimeter waveguide tubes in a near-horizontal position. The complete device for rotation of the tube while holding the anode and Teflon spacer is shown in Figure 2. The method of electrically contacting the rotating tube for plating is shown in Figure 3.

DETERMINATION OF PLATING THICKNESS AND TOLERANCES

The maximum allowable spread, AT as a percentage of plating thickness, is given by the expression:

$$\frac{D t - D E}{AT} = t \quad X \; 100 \qquad (1)$$

where:

Dt theoretical diameter range,

DE diameter range due to engineering tolerances,

t thickness of copper required to yield the average theoretical diameter.

A calculation based on Faradays Law and the dimensions of imported tubes demonstrates the required thickness to be an averages of 0.0012 ±0.00005 inches and the maximum allowed for this tube is 8.3%. Equation 1 states that for a given thickness and theoretical diameter range, the closer the steel tube approximates a right circular cylinder the greater is the allowable tolerance for the plating process. The Western Electric Engineering Research Center, Forsgate Laboratory has worked with an outside tube mill in the development of manufacturing processes which yield acceptable straightness and cross-sectional tolerances(1)

With the new high precision tubes, the limits for the plating thickness are increased. For example, data on diameter variations from a lot of 60 unplated. tubes indicates that the maximum allowable spread in thickness is about 28%. The average thickness in this case is 0.55 mills on the side.

The copper anode with its Teflon spacer is inserted into the cleaned, wet tube and both are loaded into the plating machine. The end connections are fastened and the hot copper cyanide plating solution is pumped through the rotating tube at a flow rate of 25 g/min.. When the tube reaches the operating temperature of the plating solution, the rectifier is turned on. Upon completion of the specified plating cycle, the rectifier automatically shuts off, the electrolyte is drained, and the tube rinsed in cold and hot water. After unloading the plated tube from the plating machine, the anode and spiral are removed. In order to dissolve residual plating salts, the waveguide tube is cleaned with an 8 ounce per gallon sodium cyanide solution for three (3) minutes at room temperature. The tube is then rinsed and dried.

Measurements

A. Diameter Measurement

Figure 4 shows the diameter gauge(1). It is inserted into the tube which is rotated while the diameter variation is observed. Feet (1) support the gauge. Spring loaded components (2) and (3) keep the gauge body in the correct position. Point (4) which is part of right angle attachment (5) senses the variations of the diameter as the tube is turned. The right angle attachment, in turn, actuates the linear variable differential transformer (LVDT) (6). The generated signals are transmitted to an amplifier and recorded on a strip chart recorder. Rod (7) is used to move the gauge in the axial direction.

B. Curvature Measurement

To obtain curvature information, an instrument illustrated in (1) is pulled through the tube. The output of an LVDT, placed in the center, is proportional to the curvature of the illustrated osculating circle. Two diametrically opposed passes through the tube yield the axial curvature by subtracting the outputs. Individual passes yield curvature information of the traced surface. The curvature gauge's sensitivity to surface roughness is a function of the geometry of a specific gauge.

C. Thickness Measurements

Thickness measurements are made involving microscopic observations of cross-sections. Magnetic and eddy current techniques are also used. The Permascope* measures the thickness of a nonmagnetic coating on a magnetic base material. The instrument is similar to a magnetic amplifier in that is measures changes in reluctance of a flux path due to introduction of a nonmagnetic material between the probe tip and base metal. Two hardened probes, concentric to within 10 millionth of an inch, contact the copper plated surface thus forming a closed magnetic circuit between the yoke of the measuring probes and the test specimen. One of the windings of the yoke generates a magnetic flux at 60 Hz while the other measures the reluctance produced by the coating. The amplified voltage from the measuring probe is proportional to a thickness measurement displayed on the scale. The Dermitron* measures the thickness based on the depth of penetration of induced eddy currents in the skin of an electrically conductive system.

RESULTS AND DISCUSSION

When considering the technical feasibility of producing a continuous metallic boundary for the propagation of the transmitted signal, two essential questions must be answered:

1. How does the metal deposition process affect the diameter variation of the metallic boundary?
2. How does the metal deposition affect the curvature of the metallic boundary? This report deals mainly with the first question. The current distribution in electrodeposition is the factor which determines the diameter variations of the copper boundary. Two extreme regions of the polarization curve are defined (4), namely:

1. The activation controlled region usually referred to as the primary current distribution.
2. The mass transport controlled region also referred to as the secondary current distribution. These two regions are normally separated by the limiting current density- which is defined as the current at which the concentration of the primary discharging species of the electrode surface is zero. Below this, the primary current distribution is determined by the voltage drop in solution and factors affecting the activation polarization. In this case the equation of the current overpotential curve is usually in the exponial form(3). Above the limiting current the deposition process is governed by diffusion control. As a result, the secondary current distribution is determined by factors affecting mass transport of the discharging species to the electrode double layer.

   In some electrochemical systems where discharge is from complex species whose polarization overpotential is shifted to the far cathodic regions of the polarization curve, no distinct limiting current is observed(4). This seems to be the case for the copper cyanide system. codeposition of hydrogen can occur without a distinct limiting current. A current polarization curve from the plating electrolyte used in plating millimeter waveguide tubes shows no distinct limiting current. The backside type of a reference elect rode was located six inches from the end of the 17.5 foot long tube. No limiting current was observed under operating conditions.

The occurance of copper deposition from a mixed potential is indicated by:

1. The magnitude of both the overpotential and the reversible potential.
2. No limiting current is observed even though the current efficiency is about 90%.
3. Polarization is stirring dependent.

A. Cathodic Deposition Using Direct Current

Figures 5 and 6 compare thickness uniformity and cathode efficiency of imported and domestic tubes. To maintain an effective electrical diameter of 2.0079 inches the imported tubes are plated on the average 1.2 mils on the side whereas the corresponding thickness for domestic tubes is 0.55 mils. Results in both figures indicate a cathode efficiency of about 90%. The deviation from uniform thickness is less than 0.1 mils. Surface roughness varies from 20 to 60 micro inches

Two difficulties are particularly noticeable when comparing the magnetic and eddy current techniques with cross-sectioning techniques, namely;

1. Comparative thickness measurements at the same point are impossible because:
    a. the cross-sectional technique involves a large magnification.
    b. the probes of the magnetic technique measure an average thickness between two points, .158" apart.
    c. the probe of the eddy current technique covers a 3/16" diameter circle.
2. The magnetic and eddy current measurements are affected by pits, voids, and surface roughness. These techniques average imperfections and give one reading whereas the cross-sectioning method distinguishes each one separately.

Figure 7 is representive of the other 90 degrees around the tube and shows a typical comparison of thickness measurements made by microscopic observations, magnetic and eddy current techniques. Five points are included on the graphs from the cross-sectioning technique whereas the average of three points are included for the magnetic and eddy current technique. The maximum thickness variation, even including differences in the techniques, is less than 25%. A periodic variation of about 25% in the thickness is observed in cross-sectioning measurement which is not seen in either magnetic or the eddy current techniques. The eddy current thickness measurements are consistently higher and the magnetic thickness measurements consistently lower than the cross-sectional measurements. These deviations are possibly due to variations in the standard used for calibration since the differences are consistently in one direction. Thus, it was concluded the more conventual magnetic technique could be used with considerable reliability.

Figure 8 shows a plot of diameter measurements taken on two orthogonal planes. Imported and domestic tubes were plated under the same electrochemical conditions. The imported tube was plated to about twice the thickness of the domestic one. Excluding build-up on the ends, the electrodeposition process introduces a maximum of 0.16 mils in the diameter variation in excess of the one already present in the unplaced tube. The diameter control appears to be independent of thickness in the 0.55 to 1.2 mil range. Diameter measurements on a sample of 60 unplaced tubes and 35 plated tubes are reproduced show that the maximum diameter range for unplaced tubes is .00088". For the plated tubes the maximum range is .00096". According to these measurements, the largest diameter variation introduced by the plating process is 0.000080".

Figure 9 shows a plot of thickness measurements and cathode efficiency taken on four equally spaced points around the circumference and along the tube length. Two different electrolytes were used. For this temperature, changing the cation to 100% potassium, eliminating the brightener, and reducing the total concentration of copper by 20% did not affect thickness and cathode efficiency. Deposition from the two electrolytes is indistinguishable. By lowering the number of chemicals and the total concentration of copper, the costs of chemicals, plating bath control and waste disposal are reduced. Figure 17 illustrates a plot of thickness measurements and cathode efficiency taken on one plane on four different tubes. Tubes D-114, D-92, and D-98 show the effects of active anode usage on thickness of copper deposited on the cathode when plating from the mixed sodium-potassium electrolyte. A less uniform deposit is observed with repetitive anode usage. The same effects are seen when plating from either the mixed sodium-potassium or the pure potassium electrolyte (compare D-98 and D-100) in Figure 10. Such effects can be explained in terms of periodic variation in the diffusion layer thickness throughout the tube as the anode dimensions change. When clearances between anode, Teflon spacer, and cathode are small, the spacer assists in establishing a uniform diffusion layer thickness by:

1. Providing a boundary for reproducible fluid flow,
2. Providing some stirring effects which are uniform throughout the length of the tube,
3. Provide for pulse plating (interruption of current).

The active anode dissolves at about 98% efficiency. This results in changing of the boundary conditions for curved channel flow and in decreasing the effectiveness of stirring due to the rotation of the tube past the stationary spacer. Since the efficiency of the deposition is stirring dependent, these changes result in thickness variations. The irreproducible frequency and amplitude of the varying thickness is due to the variable frequency of the spacer winding and to the variation in contact the spacer makes with the cathode Figure 12 shows thickness measurements taken on the O' plane versus the distance from the respective fluid entrance and exit of a tube from a domestic supplier (D-169) and one from a foreign source (D-157). This increase in thickness at the ends of the tube is attributed to the fact that the anode is uninsulated. Because of this, current lines of force due to polarization of the anode beyond the ends of the cathode terminate on the cathode. In order to eliminate this undesirable increase of plating thickness, approximately 5% of the tube length is cut off on the ends.

The distribution of Forspate Laboratory copper plated tubes put into three classifications governed by diameter and curvature measurements The plating process introduces little additional diameter variation beyond that already present in the steel tube. Most of the tubes classified are based on curvature because:

1. The curvature of unplated, imported tubes is generally greater than on those of domestic origin.
2. Surface roughness introduced by D.C. plating is more pronounced on imported tubes because:
   a. Plating is twice as thick and surface roughness increases with increasing thickness
   b. The bores of the imported, unplated tubes are rougher than those on the domestic tubes.
   c. It was not possible to thoroughly clean the imported tubes since a mixture of phthalic anhydride and a corrosion protection oil was present. This mixture formed an insoluble, non-conductive and non-reacting film which could not be cleaned by normal chemical procedures. It was only possible to detect the presence of the film after plating. The film which produced poor adhesion and rough deposit affected the curvature measurement.

Comparison of Plating with Fixed Cathode Versus Rotating Cathode

Figure 12 shows a plot of thickness measurements taken for four equally spaced points around the circumference versus tube length. One tube was plated with both anode and cathode held stationary in the vertical position. The other was plated with a rotating cathode with a helical Teflon spacer and anode held stationary. The results, based on several experiments with both types of plating facilities, indicate that rotating the cathode yields a more uniform and reproducible thickness. This observation is made with respect to the present waveguide plating technology. The apparent nonuniform irreproducible thickness variation observed using the vertical set up seems to stem from the inability to maintain the anode concentric with the tube over the 18 foot length. Figure 13 shows the same measurements plotted in terms of diameter variation for two orthogonal planes versus tube length. The plot shows that plating with stationary annuli introduces greater diameter variation.

Use of Periodic Reverse Techniques

It is well known to modern electroplaters that, under certain electrochemical conditions, the use of periodic reverse techniques can reduce the surface roughness of the substrate (7,8) and, at least, retard the rate of surface deterioration(9).

Periodic reverse has been used at Forsgate Laboratory to:

a. Plate 18 foot long tubes with excellent diameter and curvature control without introduction of the hyperfine structure in the unfiltered curvature trace seen on D.C. plated tubes.
b. Adjust the diameter of copper plated tubes to meet required tolerances.
c. Show that no curvature deterioration occurs during the time required for the plating process.

Deposition of Copper Using Periodic Reverse Techniques

Figure 15 shows the filtered (top) and unfiltered (bottom) curvature traces of a plane on a domestic tube, D-215. Other planes show the same results. Figure 16 shows the same trace after plating at 17 amp/ft 2 for 20 minutes with the CuCN-NaCN-KOH bath at 155°F. These two figures illustrate the change in the short range curvature using direct current plating techniques. Figure 23 illustrates the corresponding effect when using periodic reverse techniques. Tube (D-298) was first plated at 17 A/ft^2 for 6.3 minutes and then with periodic reverse for 21 minutes. The periodic reverse cycle was 15 sec. cathodic at 17 A/ft^2 and 5 sec. anodic at 13 A/ft^2. The resultant curvature trace is shown in Figure 17. A comparison between Figures 16 and 17 show that no hyperfine structure is observed in the unfiltered trace taken on the periodic reverse plated tube. Figures 18 and 19 illustrate the same effect when using periodic reverse techniques on tube D-299. The 132 minute cycle was cathodic at 17 A/ft^2 for 8 sec. and anodic at 13 A/ft^2 for 8 sec.

2. Plating Tubes to Required Diameters

Periodic reverse techniques have been used to reduce the plating thickness (increase the diameter-) without changing the curvature or introducing hyperfine structures in the unfiltered curvature trace. Figure 26 shows both the filtered and unfiltered curvature traces for tube D-299 which was deplated using periodic reverse techniques. The 24-minute depleting cycle was anodic 15 sec. at 13 A/ft^2 and cathodic 5 sec. at 17 A/ft^2.

This shows that an overplated tube can be deplated to the specified dimensions without changes in curvature. Ten tubes were reclaimed using periodic reverse techniques. The mechanical measurements before and after the process qualifies these tubes for installation.

Anodic Dissolution of Copper Anodes Using Direct Current Techniques

1. Efficiency of Anodic Dissolution

The efficiency of anode dissolution is determined by weight loss and dimensional changes.

a. Weight Loss

Data were compiled from five anodes used in plating millimeter waveguide tubes on the horizontal plating facility. According to Faraday's law for copper dissolution of cuprous cyanide, the total amount of copper dissolved will be given by the current passed, time the current passed and several conversion constants such as the molecular weight.

Percent Efficiency = 100 - Measured Weight Loss/Predicted Weight Loss

The percent efficiency for five anodes used in plating waveguide tubes is 98% and independent of the hardness in the range from B-60 to F-29 Rockwell.

b. Dimensional Change

The diameter changes for anodes mentioned above were taken over the total length of the uninsulated electrode. The average diameter change yields final diameter of 0.684 $\pm$ 0.010 inches. The weight change based on dimension is 29.6 pounds. Based on the amount of current at 100% efficiency, 29 pounds should be dissolved. The percent efficiency, E, is equal to:

$$E = 100 \times 29.6/29.0$$
$$= 102 +/- 3\%$$

Uniformity of Anode Dissolution

Three copper anodes were quantitatively studied, one is unprocessed, the middle and top area uninsulated anodes used in processing waveguide tubes. The first one was processed to its fullest capacity when used without an internal supporting member. Figure 20 shows the variation of anode diameter for two orthogonal planes as a function of anode length. Excellent uniformity and efficiency is shown when end effects are excluded. Figure 21 shows that the anodic dissolution process is not mass transport control dependent with respect to diffusion of anodic product from the electrode surface.

The current density was 60 amp/ft^2 excluding any shielding results from the spacer between the anode and cathode.

The Maximum Utilization of Copper Anodes

An equation has been developed to determine the maximum number of tubes that can be plated with an active anode. Converting all the tubes plated into the same quantity of copper, the average number of tubes per anode is 91. The maximum number of anodes is 109. Therefore, the percent utilization is given by:

$$\% \text{ utilization} = \underline{91}/109 \times 100 = 83.4\%$$

Plating 36 Foot Long Tubes Using Direct Current Techniques

In order to show that processing costs per unit length of installed waveguides could be reduced, it was desirable to demonstrate the feasibility of plating longer lengths of tubes. Thirty-six foot tubes were plated horizontally using the principles previously described for the 18 foot tubes. Figure 22 shows a plot of thickness measured on four perpendicular planes around the circumference and along the 36 foot length. The tube was rotated at 30 rpm and plated with the mixed sodium-potassium copper cyanide plating solution recirculated at approximately 25 gpm. Uniform current distribution is illustrated for the cathodic deposition of copper. The anode was blackened due to oxidation. The intensity of the discoloration increases from the entrance to the exit (with respect to fluid flow) illustrating mass transport influences on the diffusion of anodic products. The diameter and curvature measurements of two 36 foot long tubes are classified in the excellent group.

Conclusions

Under controlled conditions the following conclusions can be stated:

1. The average diameter of copper plated tubes can be maintained at 2.0079 inches with a variation much less than $\pm$ .0004 inches. (For 51 mm diameter waveguide.)
2. The copper plating process previously described adds less than 10% of the thickness of copper to the diameter variation already present in the steel tubes.
3. Where there are no pits or voids in the steel substrate, the surface roughness along the tube axis after plating varies from 20 to 60 p inches.
4. Electrical measurements of copper plated tubes have been made using two different methods: one involves a shuttle pulse test and the second involves the injection of a spurious mode into the waveguide. Both measurements indicate the copper losses in the range of frequencies from 100-110 GHz exceed the theoretical copper losses by only 15-20%.
5. For well controlled cleaning and plating conditions, copper has excellent adhesion to the steel as indicated by bend tests (greater than 180 degrees bend) and microscopic observations of cross-sections. In addition, the results of this study have shown:
   1. Plating with a rotating cathode produces a more reproducible, -uniform thickness than plating with a stationary anode and cathode.
   2. Uniform plating thickness using rotating cathodes was observed while varying:
      a. thickness of the deposit,
      b. type of copper cyanide solution,
      c. continuous use of the same copper anodes.
   3. Use of periodic reverse yields uniform diameter control while retarding the deterioration of the surface finish.
   4. Plating with rotating cathodes in lengths up to 36 feet yield thickness variations of less than $\pm$ 5% using a new anode.
   5. Pulse Plated waveforms of the interruption of current type can be generated using cell geometry. The properties of the waveform depend on the anode, cathode and insulator distances and how many of the insulator(s) proceed past the cathode, the speed of the cathode pass the anode.

Acknowledgments

D J. Fineberg and W. E. Rapp made significant contributions without which the project would not have been so successful in such a short time frame. R.G. Baker is to be acknowledged for contributions to the success of this project.

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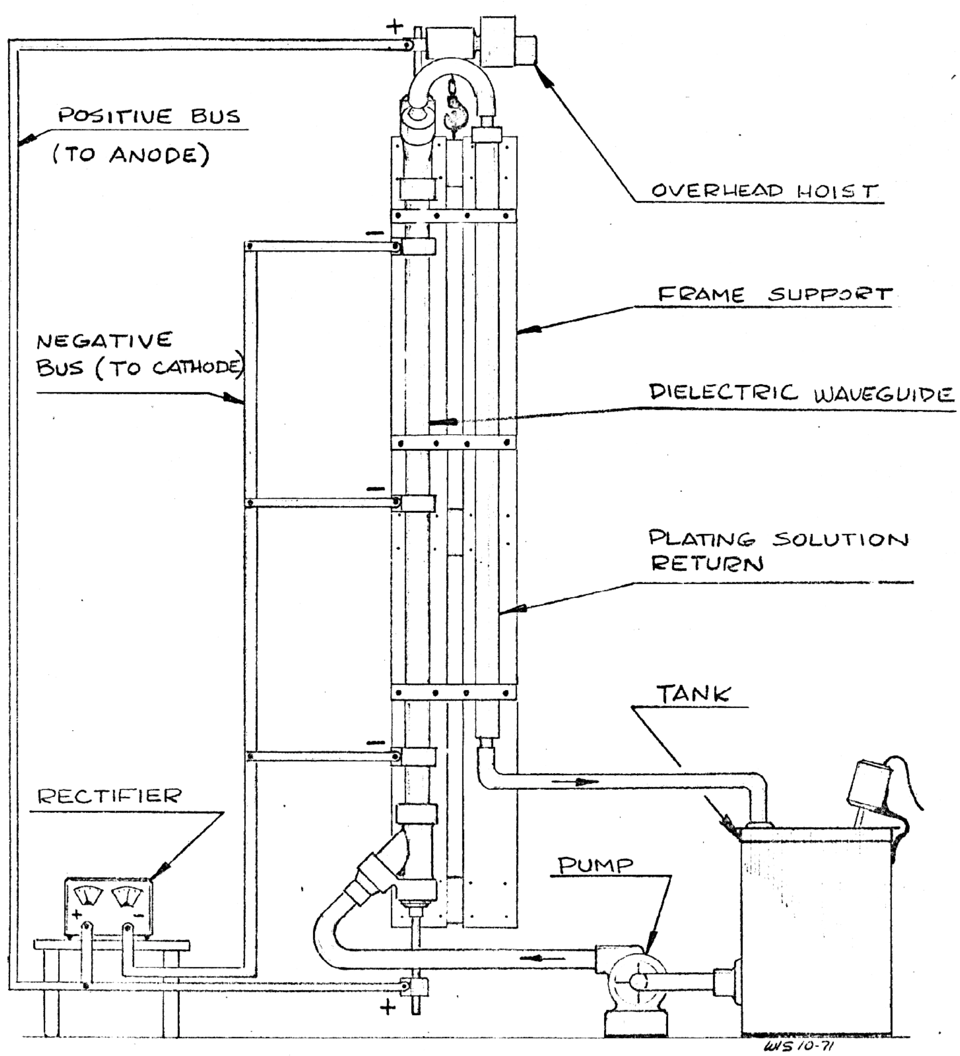
Figure 1

VERTICAL PLATING RACK



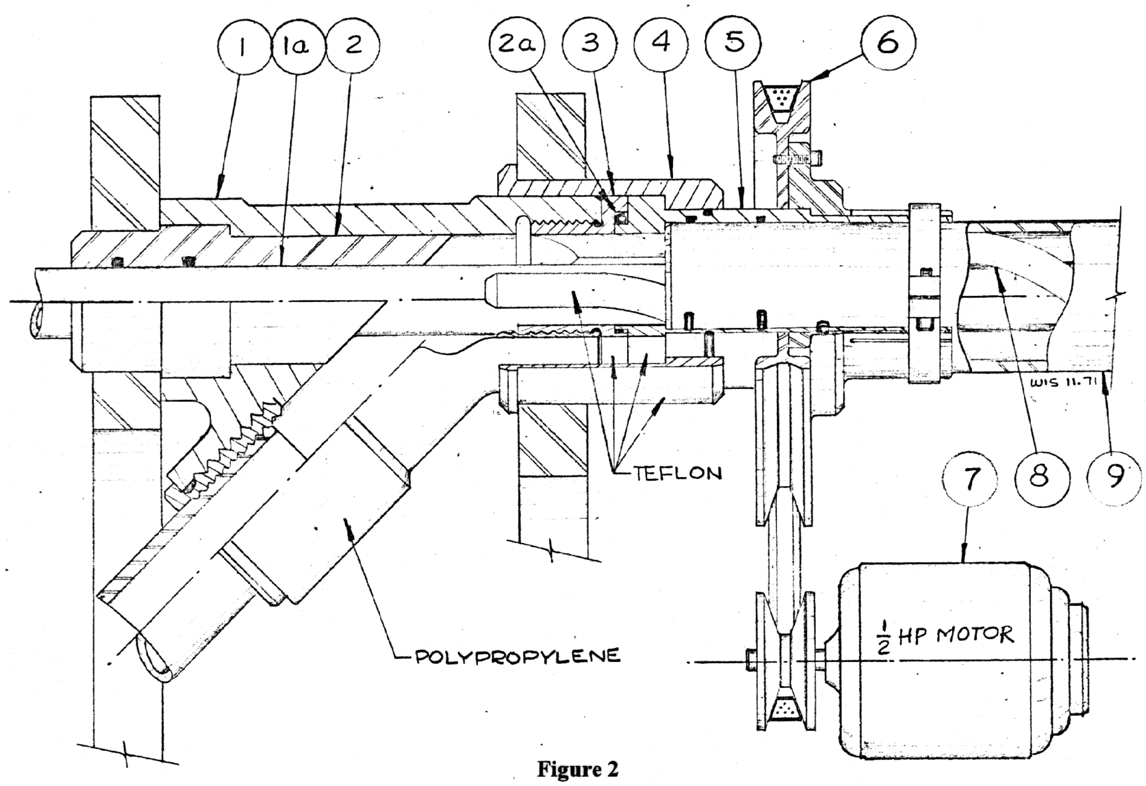


Figure 2

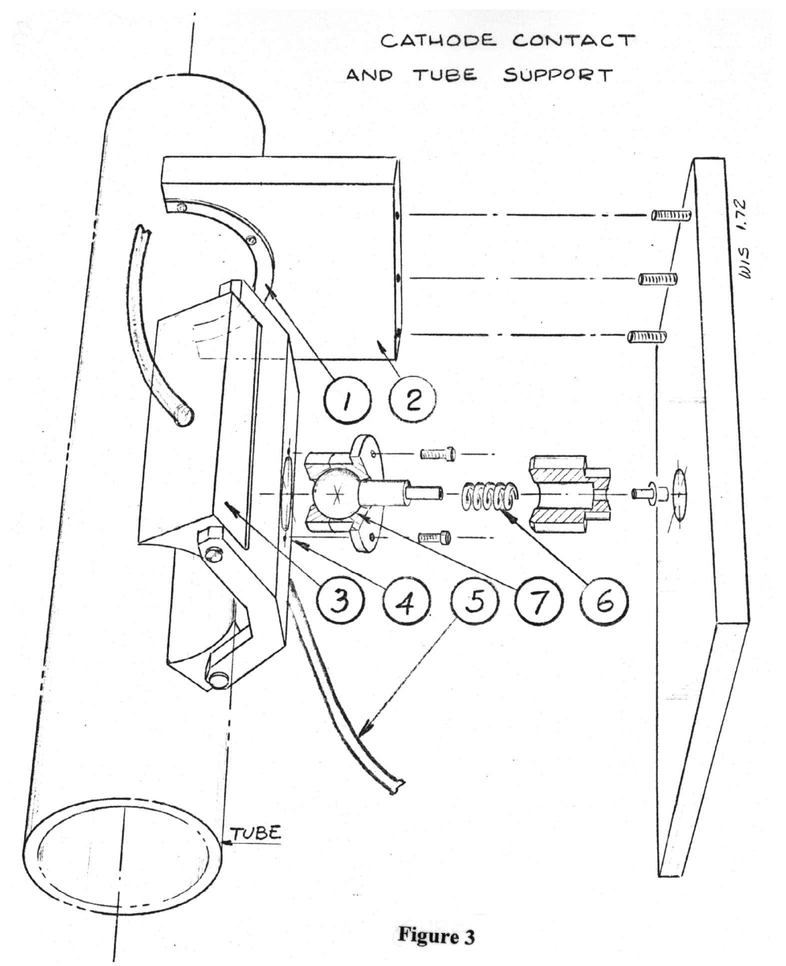


Figure 3

DIAMETER GAGE

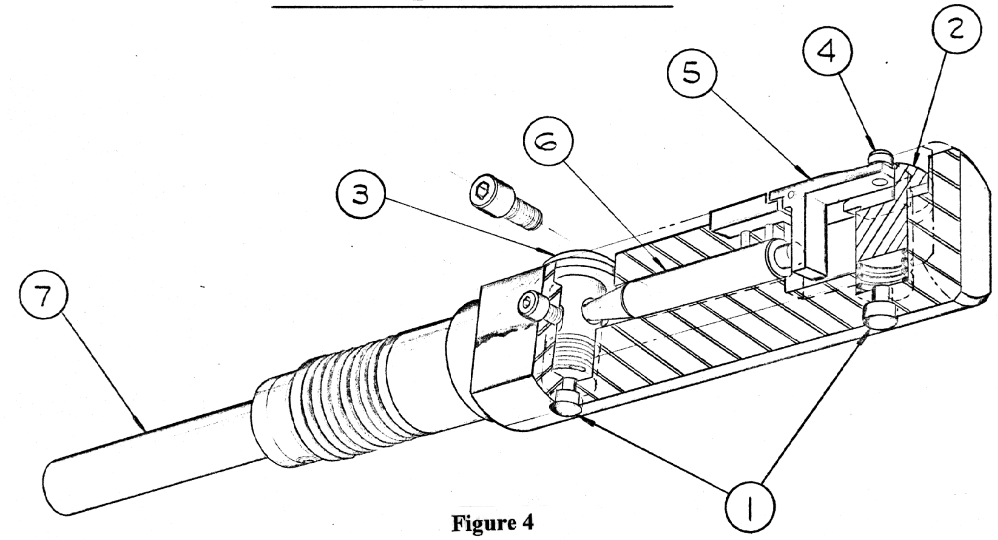


Figure 4

MILLIMETER WAVEGUIDE – DIELECTRIC
HORIZONTAL PLATING
THICKNESS – VARIATION
FOUR PLANES

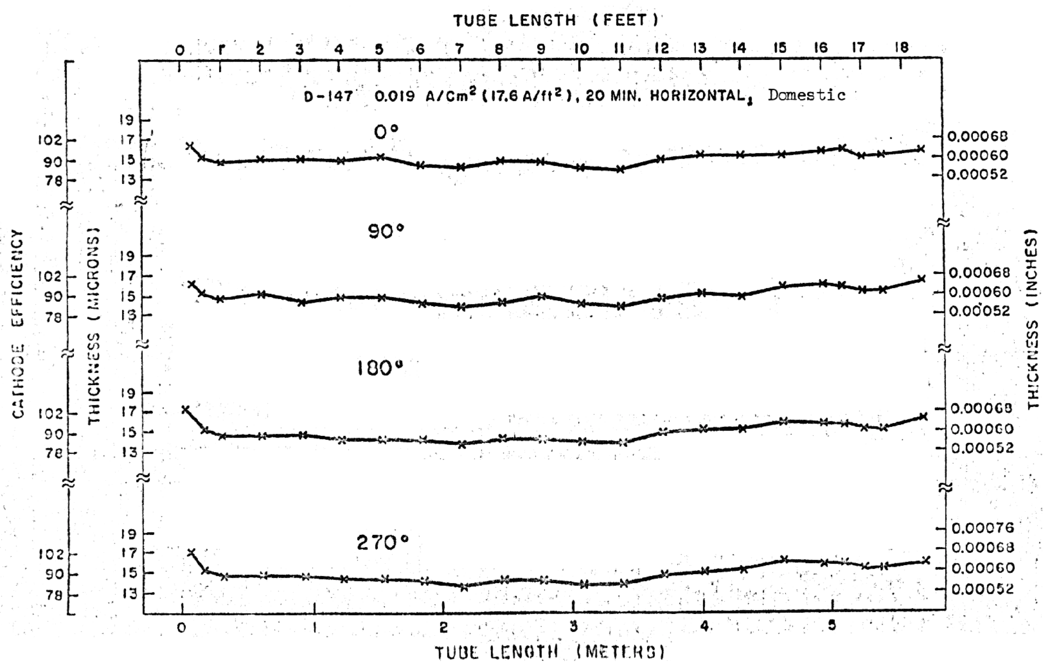


Figure 5

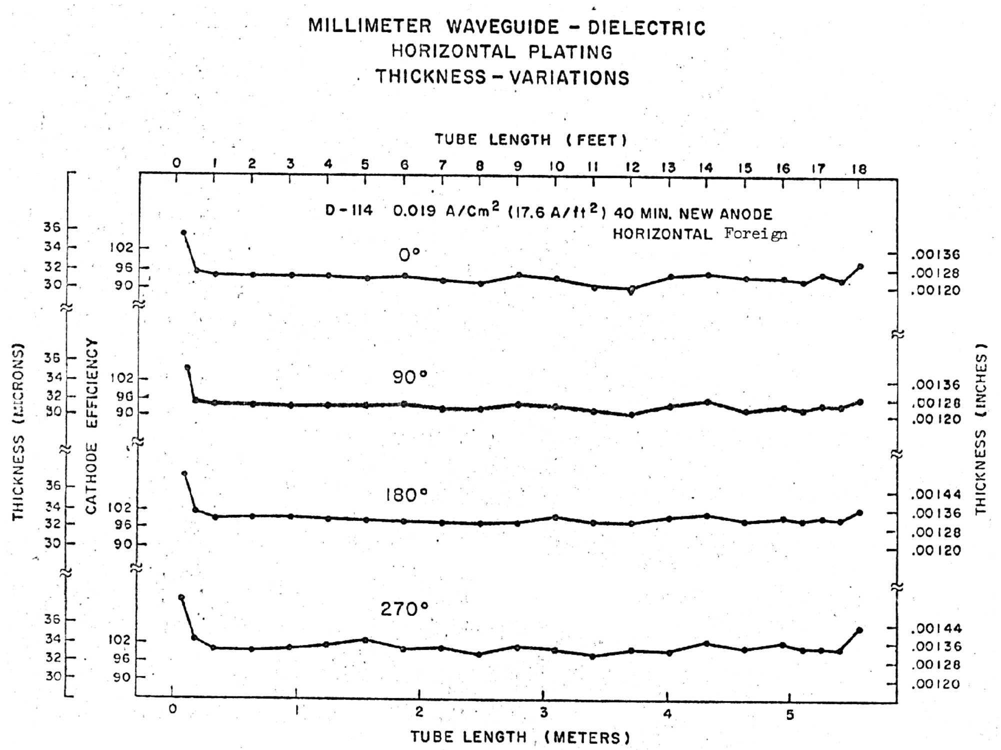


Figure 6

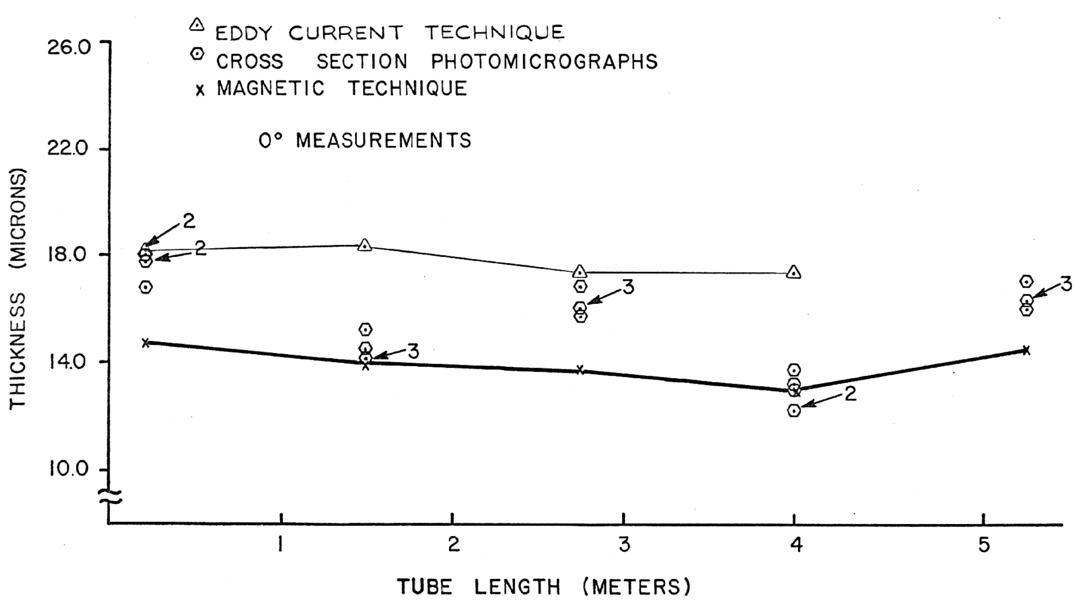


Figure 7

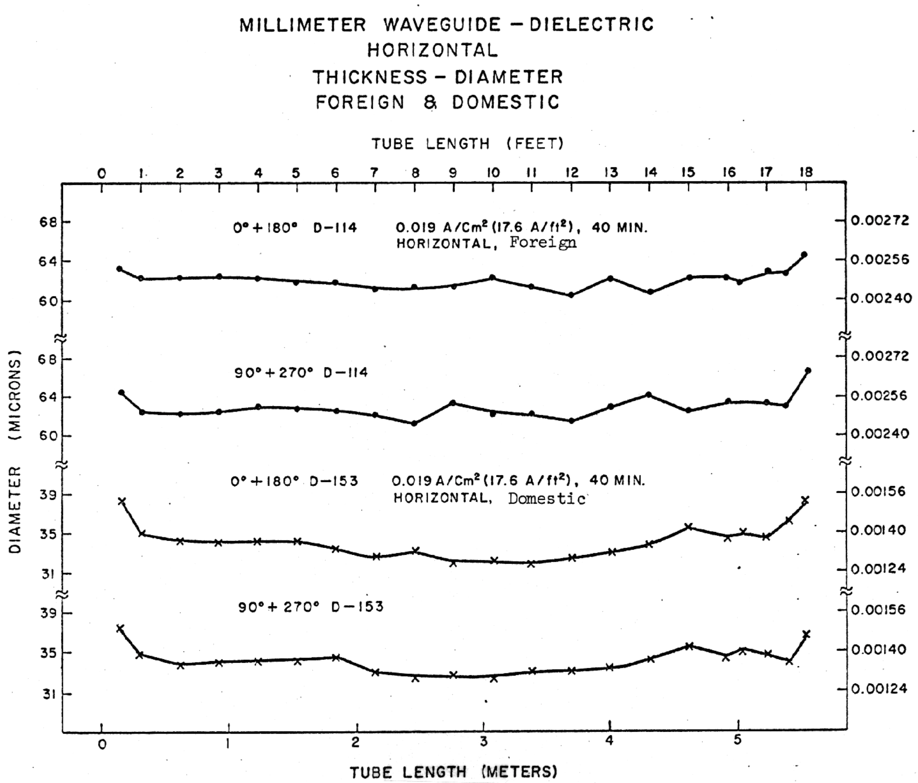


Figure 8

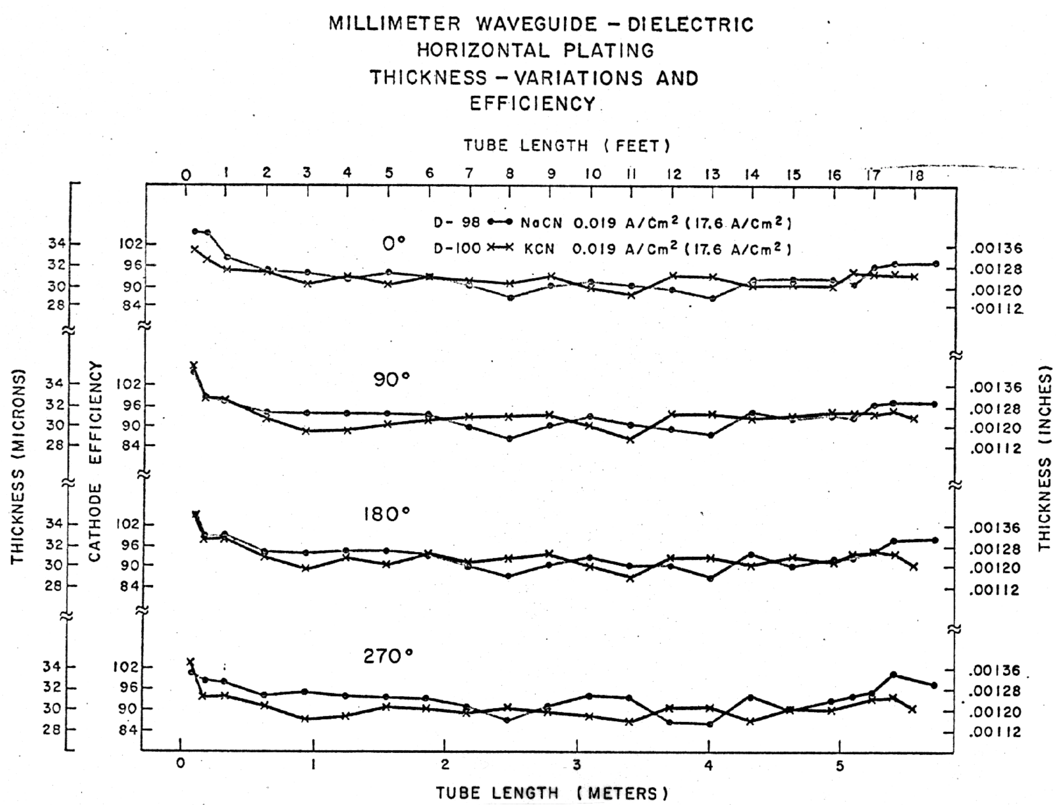


Figure 9

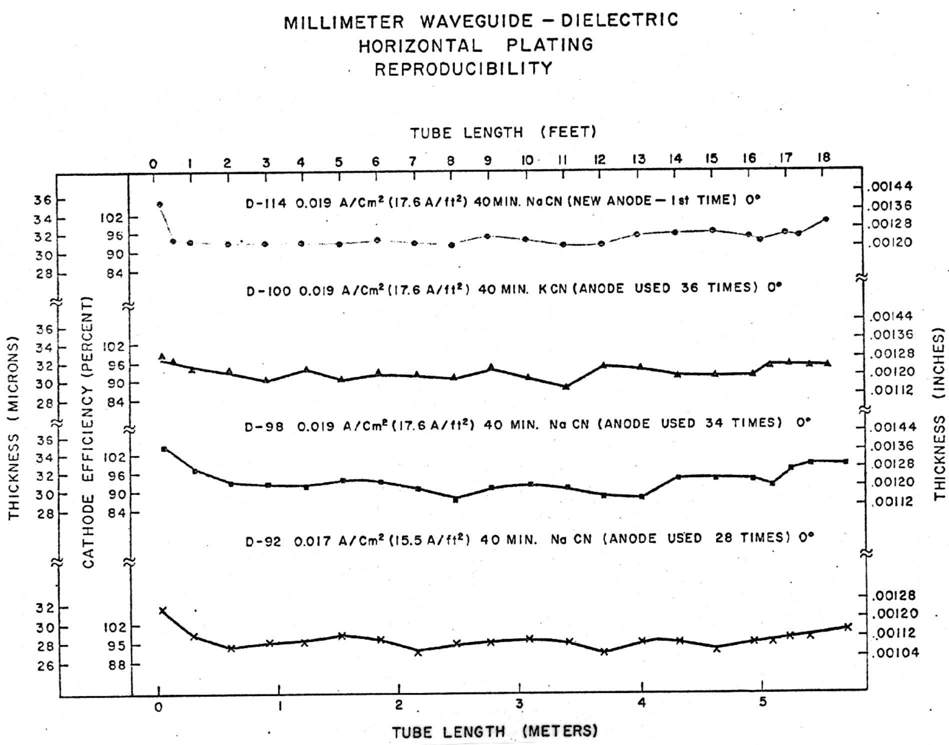
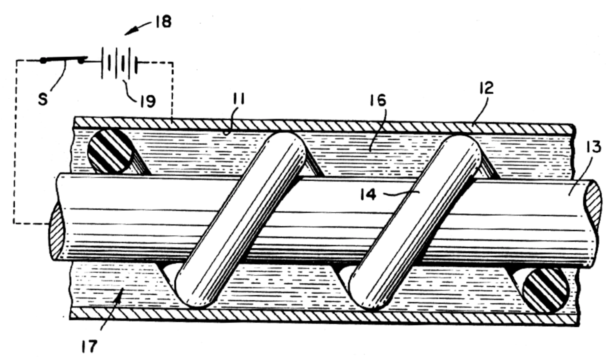


Figure 10



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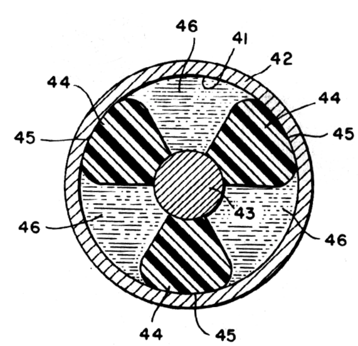

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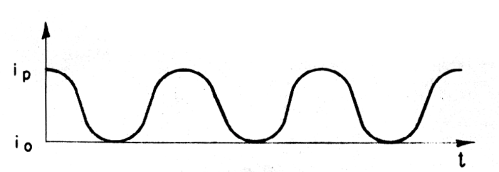

C

Figure 11

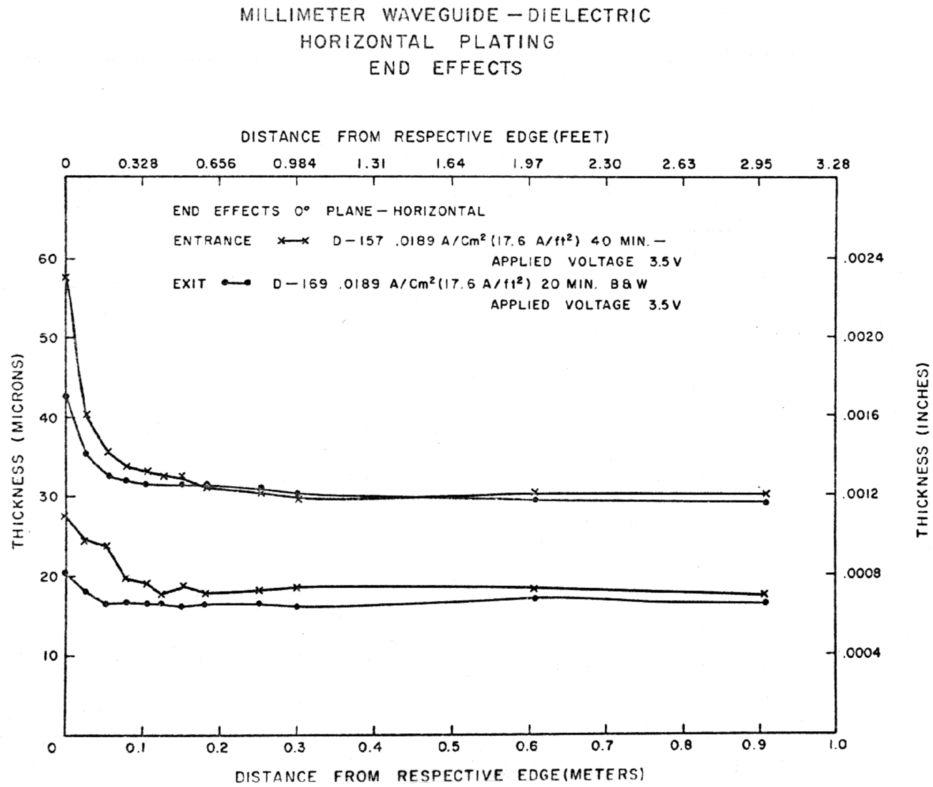

Figure 12

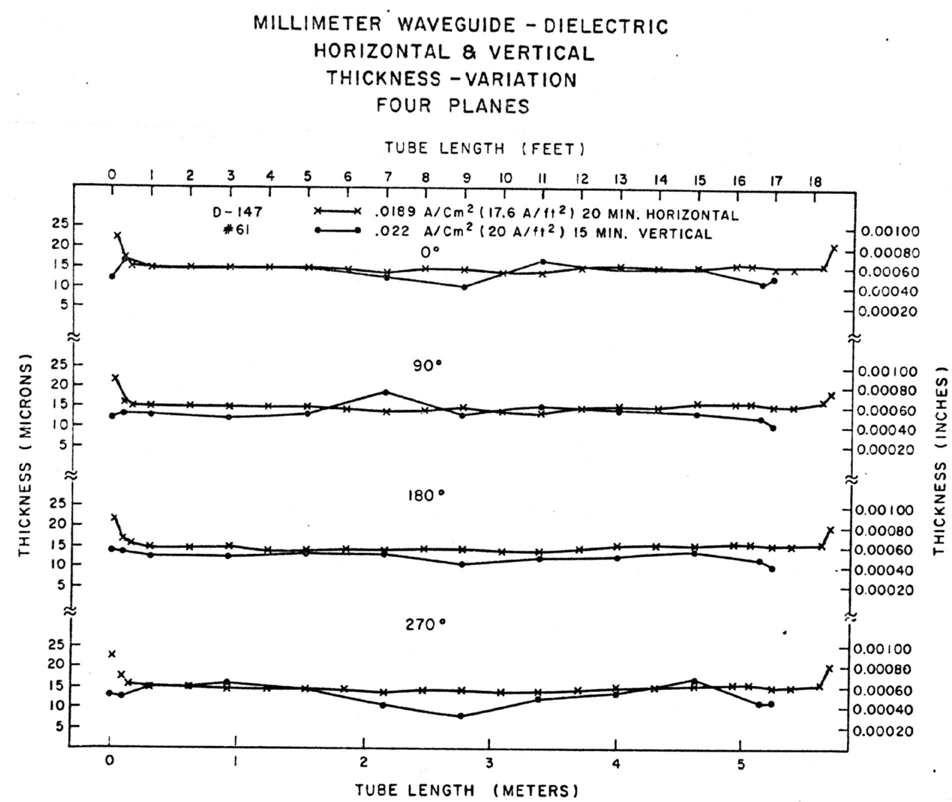

Figure 13

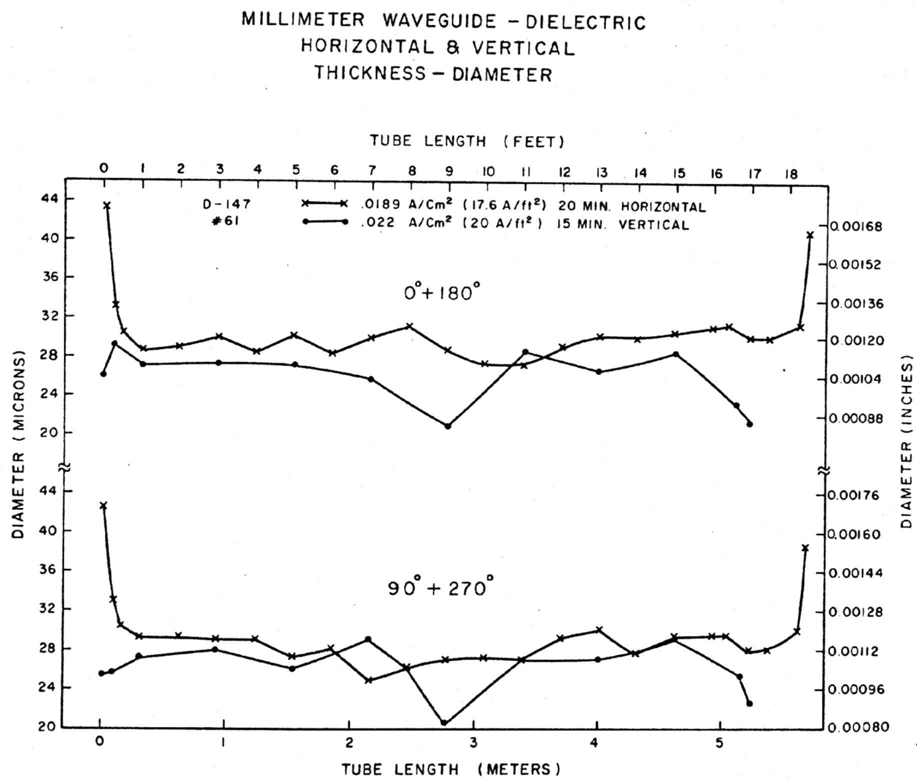

Figure 14

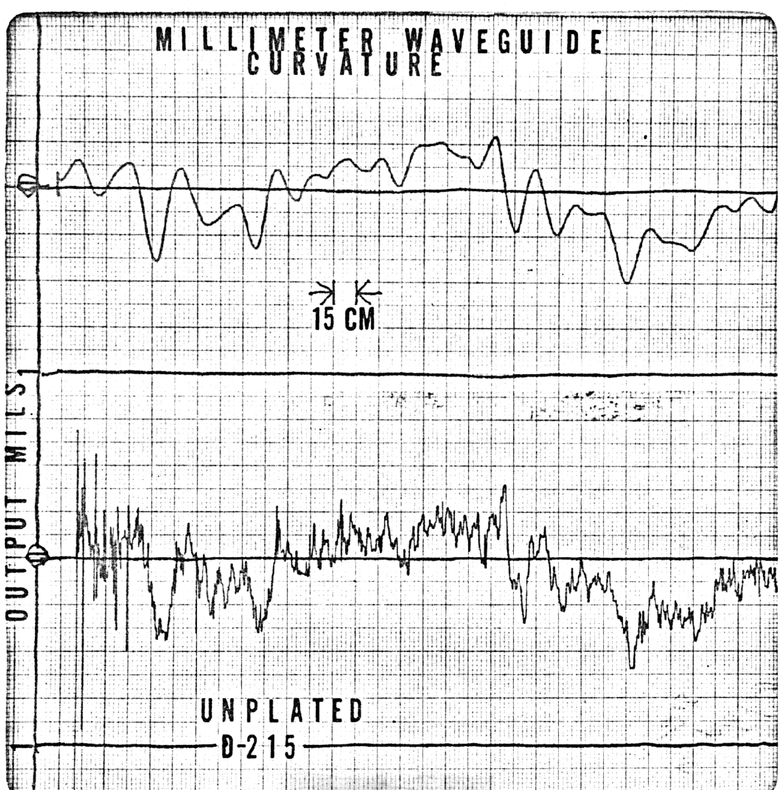

Figure 15

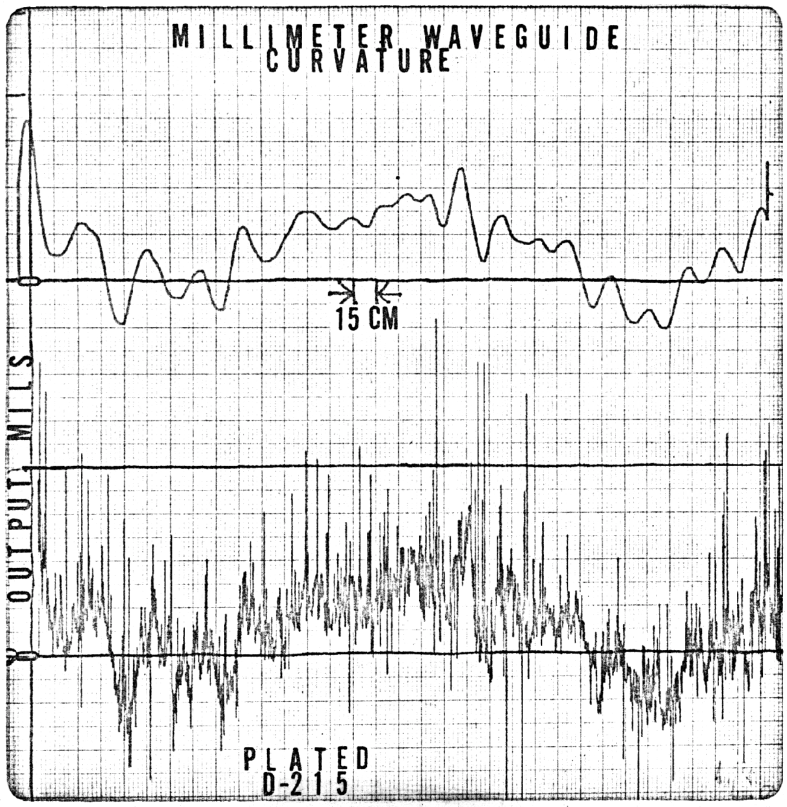

Figure 16

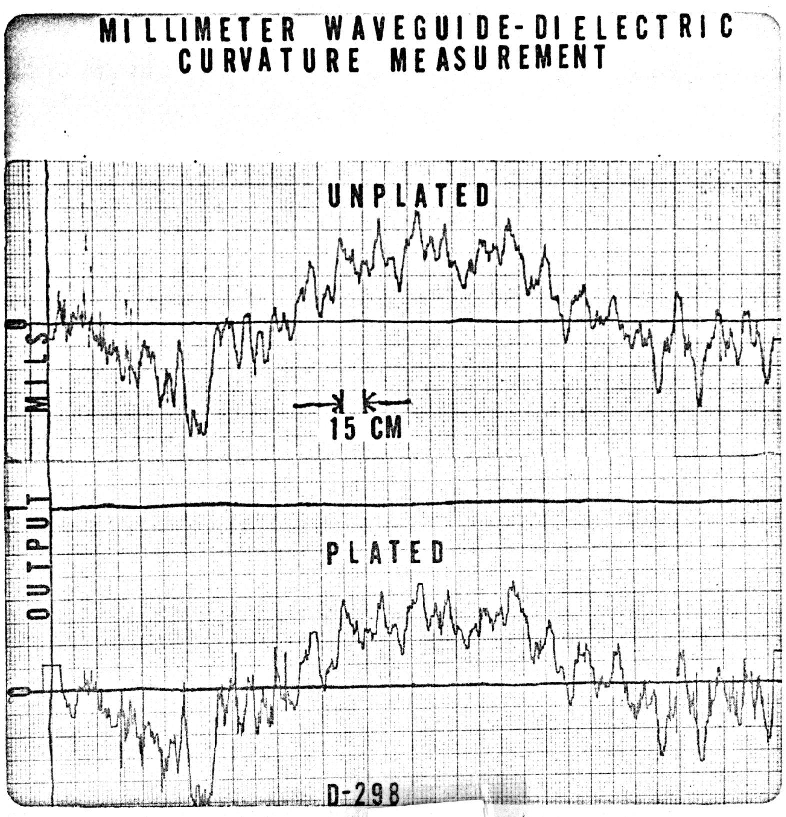

Figure 17

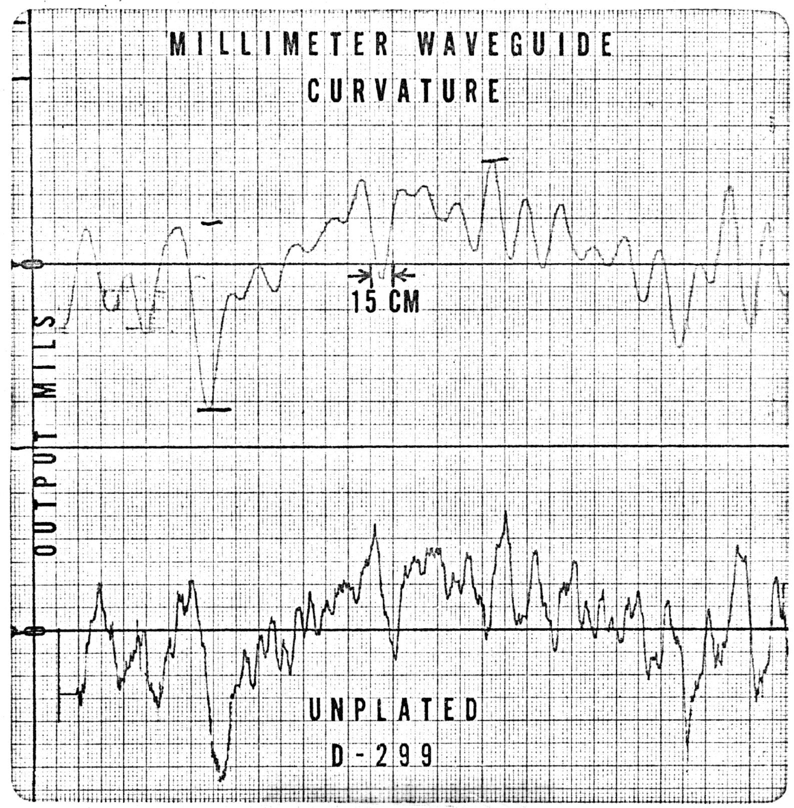

Figure 18

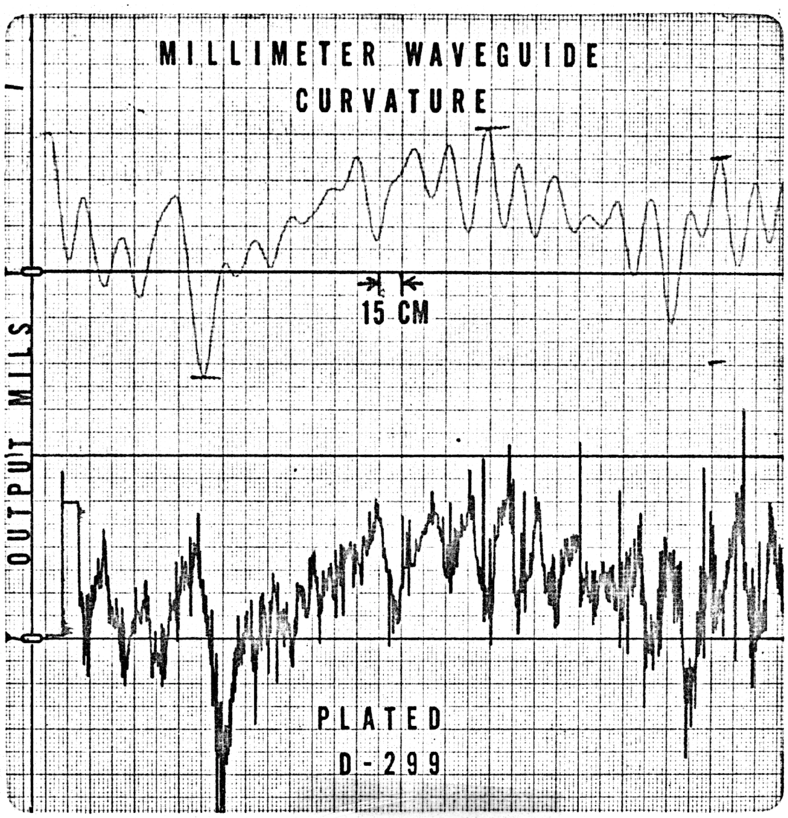

Figure 19

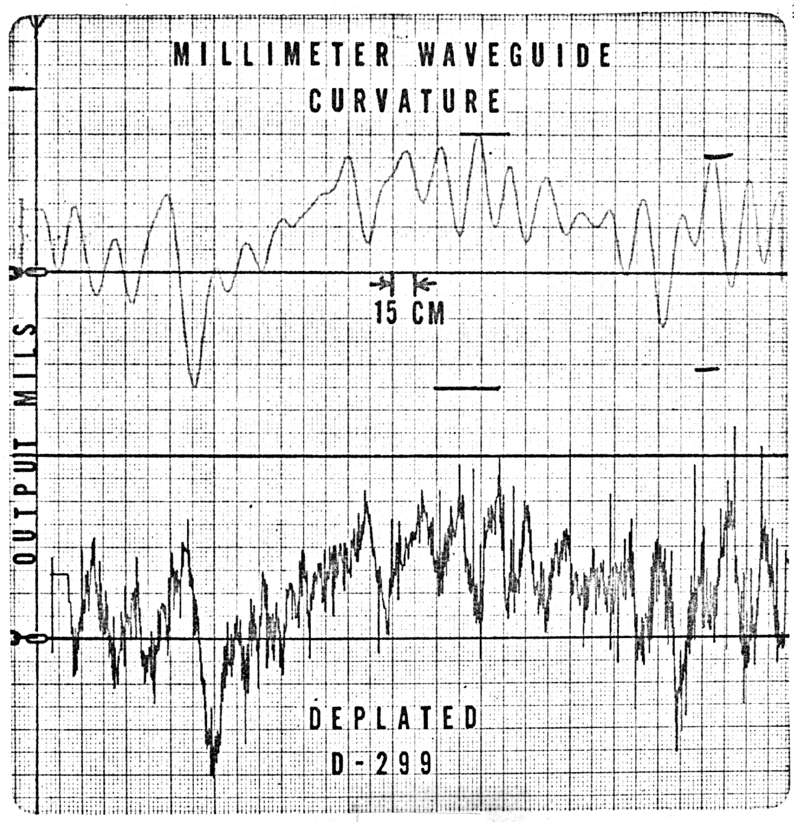

Figure 20

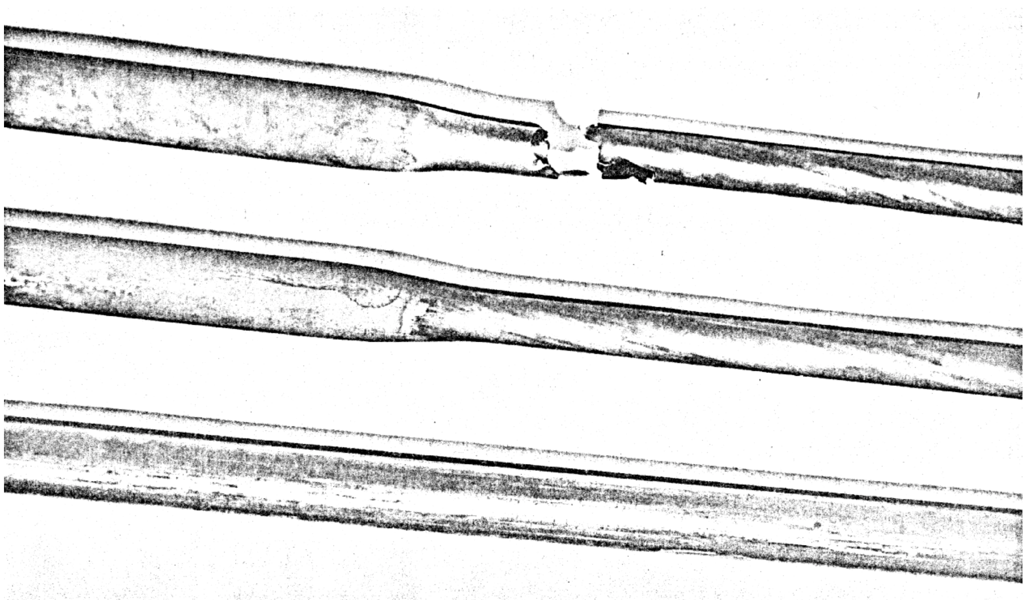

Figure 21

MILLIMETER WAVEGUIDE - DIELECTRIC
DISSOLUTION OF COPPER ANODE
PLATED 49 WAVEGUIDES

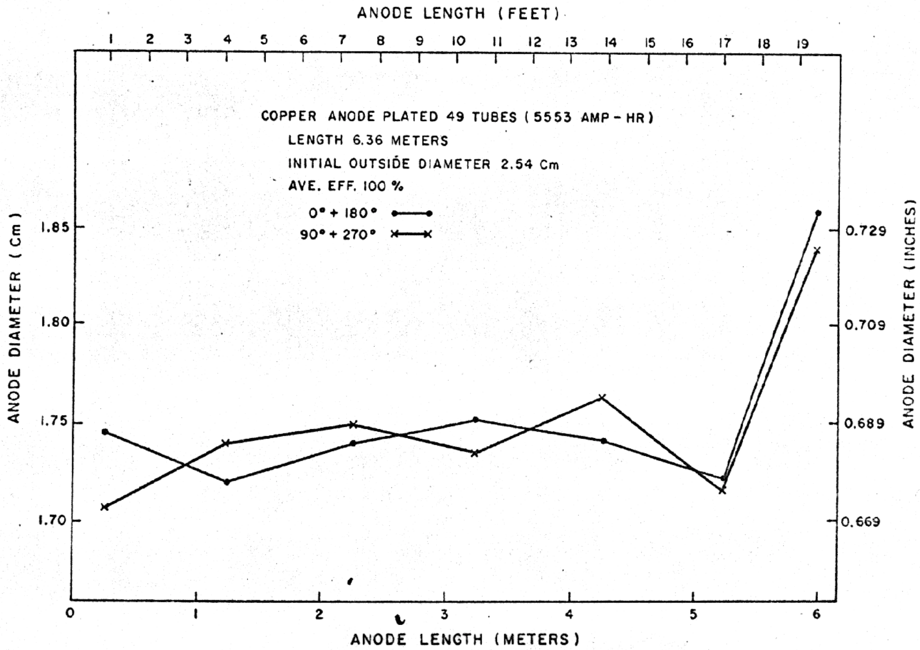

Figure 22

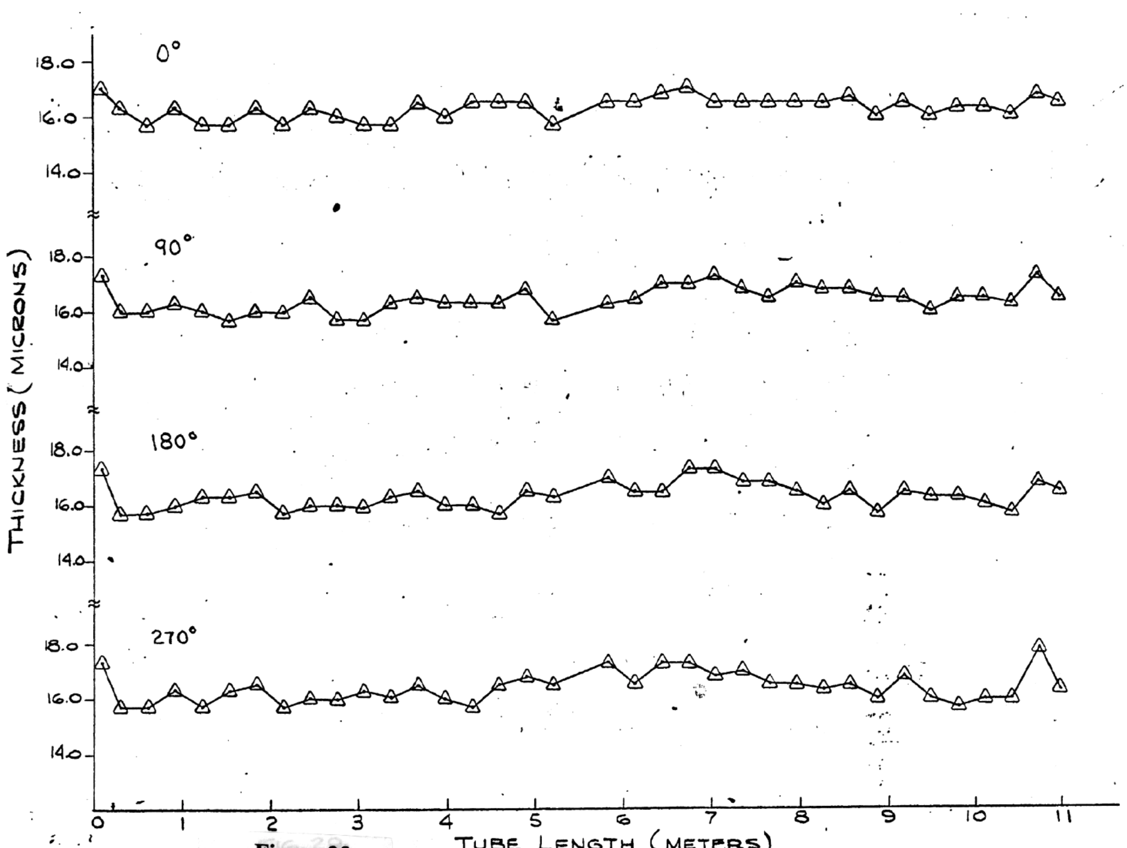

Figure 23