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Contact Whiskers For Millimeter Wave Diodes

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Greenbelt, Maryland 20771

CONTACT WHISKERS FOR MILLIMETER WAVE DIODES

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May 1978

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CONTENTS (Cont'd.)

ABSTRACT

Several techniques are investigated for making short conical tips on wires (whiskers) used for contacting millimeter-wave Schottky diodes. One procedure, using a phosphoric and chromic acid etching solution (PCE), is found to give good results on 12μm phosphor-bronze wires. Full cone angles of 60°-80° are consistently obtained, compared with the 15°-20° angles obtained with the widely used sodium hydroxide etch. Methods are also described for cleaning, increasing the tip diameter (i.e. blunting), gold plating, and testing the contact resistance of the whisker.

The effects of the whisker tip shape on the electrical resistance, inductance, and capacitance of the whiskers are studied, and examples given for typical sets of parameters.

§1. Introduction and General Discussion

§1.0 Introduction

The fabrication of pointed wires for contacting Schottky diodes is probably the least controlled step in the fabrication of millimeter-wave mixers and detectors, and has always involved considerable luck. In working towards higher frequencies, the need for smaller and better controlled whisker points led to our undertaking the theoretical and experimental work reported here.

§1.1 Electrical Characteristics

Section 2 of this report discusses the electrical characteristics of conically pointed contact whiskers. If the contact area between the whisker and diode is extremely small, excessive contact resistance may be introduced in series with the diode. On the other hand, if the tip of the contact whisker is comparable with or wider than the diode's anode, excess capacitance may result in shunt with the diode. Whisker points which are very long and thin are mechanically delicate, while stubby whisker points can withstand more physical abuse (e.g. repeated contactings) and have the additional advantage that the whisker can be bent much closer to its tip, allowing a shorter overall whisker.

§1.2 Whisker Material

Our experiments have been almost exclusively with commercially available* 12μm diameter phosphor-bronze wire (Alloy C: 92% Cu, 8% Sn, stress relieved).

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*Sigmund Cohn Co., Mt. Vernon, N. Y.

Earlier work using pure gold and tungsten wires gave unsatisfactory results: Gold whiskers, being extremely soft, are easily damaged and appear to have insufficient elasticity to ensure a secure contact with a diode. Tungsten on the other hand is very hard and has a tendency to punch through the metallized anode of a diode, forming a tungsten-GaAs point-contact diode in parallel with the original Schottky diode, which has a disastrous effect on mixer performance. This has been noticed particularly in cryogenic mixers where differential expansion with repeated cooling gradually works the whisker through the anode metallization of the diode. Phosphor-bronze wire largely overcomes both these problems, but must be gold plated to ensure a good contact (see Section 3.11 on contact resistance). A gold-nickel alloy, Nioro**, has reportedly given excellent results[1] when etched with a potassium cyanide solution, and requires no plating. We have not tested this material, nor have we used any cyanide etching solutions, as it is considered desirable to avoid using highly poisonous materials.

§1.3 Whisker Pointing

In Section 3 details are given of the whisker fabrication procedure we have found most satisfactory so far. In arriving at this procedure a number of different approaches were tried, all using electrolytic etching of the phosphor-bronze whisker wire in some aqueous electrolyte. Sodium and potassium hydroxide solutions (~10% by weight) had been in use in our laboratory for several years and had both given good results where relatively long whiskers were acceptable (full cone angle $< \sim 15^\circ$). An AC voltage (~3V RMS) was used, with a platinum cathode, followed by pulse blunting to increase the tip radius (see §3.8), then gold plating. Similar results were obtained using DC (~2V). However, despite many experiments, we could find no way to produce large cone-angles using this electrolyte.

** 82% Au, 18% Ni, Western Gold & Platinum Co., Belmont, CA.

Microscopic examination of the etching process suggested that the surface tension and contact angle between the surface of the liquid and the whisker were critical in determining the shape of the final point. In particular it appeared that the surface tension, acting along the surface at the contact point, supported the liquid raised in the meniscus above the mean surface level. As the metal was etched away the wire diameter was reduced, thereby reducing the total force available to support the meniscus at the whisker - see Fig. 1.1.

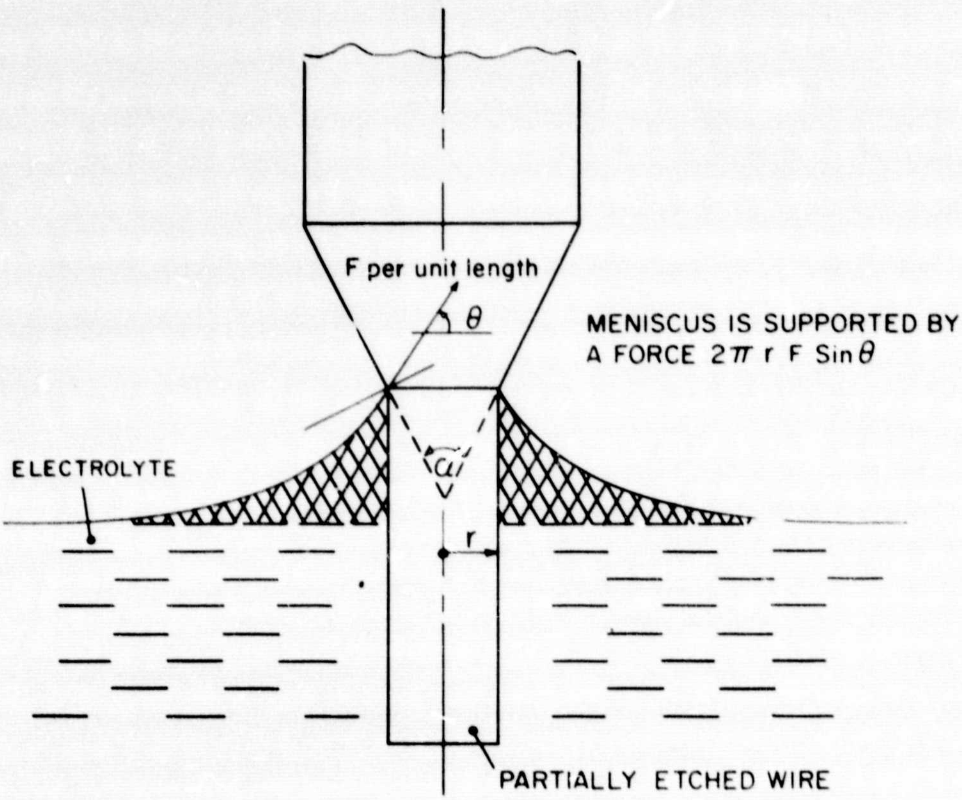

Fig. 1.1 Shaded meniscus liquid is supported by the force $2\pi r F \sin\theta$.

Consequently, as the wire diameter was reduced, the meniscus fell, giving rise to an almost conical point. *

As a first attempt at making blunter cone angles, we tried to change the contact angle between the electrolyte and the whisker by floating a non-conducting fluid (turpentine) on top of the electrolyte (10% NaOH). This resulted in a reverse meniscus where the whisker penetrated the interface between fluids. Some acceptable whiskers with (full) cone angles $\leq 45°$ were made in this way, but results were erratic and the yield small, probably due to gas bubbles being trapped at the fluid interface. **

The next approach to whisker pointing was to use an electro-polishing solution as electrolyte. Successful results have been reported [4] using an electrolyte containing sulphuric, chromic, and phosphoric acids [5] (14%: 0.5%: 49% by weight, the remainder water). However we were not able to get reproducible results with this. We believe that the temperature and proportions of the constituents are quite critical, as they are when the solution is used in more conventional electropolishing applications. Successful electropolishing of copper has been achieved using 85% phosphoric acid in which a quantity of copper has been electrolytically dissolved. This too was found unsatisfactory for pointing phosphor-bronze whiskers.

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* A totally different theory of electrolytic wire pointing is given by Martin et. al. [2] for tungsten wires. They attribute the pointing action to the convection of ions near the metal surface. We feel that this is not a dominant effect under the etching conditions we normally use, in which copious evolution of bubbles occurs.

** Lidholm [3] describes a method of adjusting the surface tension of a NaOH etching electrolyte by the addition of a small amount of methanol. We have not tried this approach as his report was received after completion of this project. The whiskers shown in his photographs have cone angles of $15° - 25°$, i.e. smaller than our target of $60° - 90°$.

A third electropolishing solution, which we shall call PCE, consisting of phosphoric and chromic acids, and water (80%: 5%: 15%, by weight), was tried and found to give clean points with (full) cone angles of 60° - 80° when pointing phosphor-bronze whiskers. The exact mechanism of this process is not clear, but microscopic observation suggests that it depends on (i) having a suitable meniscus shape (i. e. contact angle), (ii) the rate of bubble evolution, and (iii) the formation of a transparent sheath, probably tin oxide or hydroxide, as the wire is etched away. This sheath seems to dissolve gradually in the electrolyte as etching progresses, and usually falls away from the wire just before etching is completed. This whisker pointing process is described in detail in Section 3.

Figure 1. 2 shows some typical whiskers made using the various procedures described above. These whiskers have not been "blunted", nor gold plated, but are as they appear after etching and cleaning in dilute hydrochloric acid.

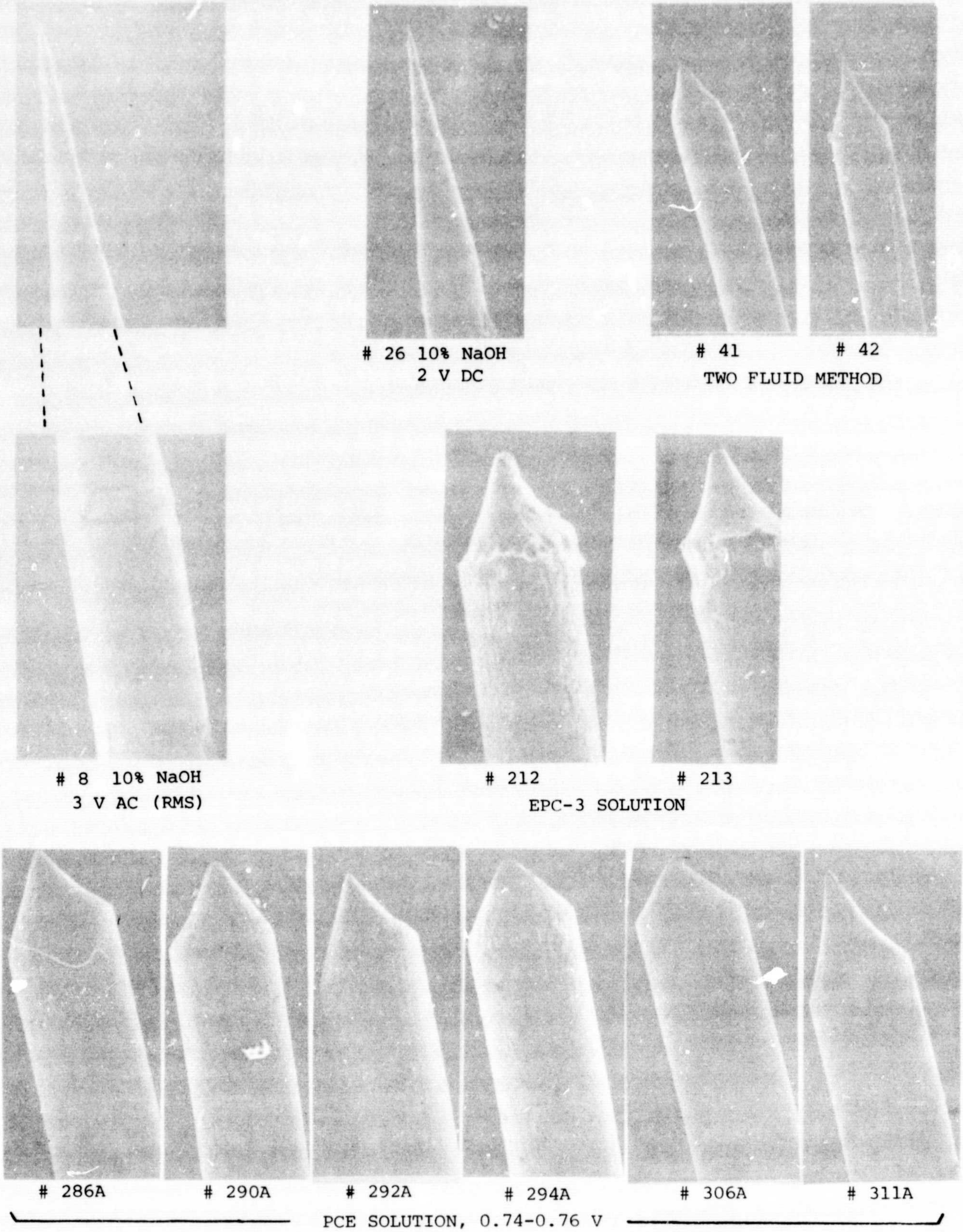

FIG. 1·2 Typical whiskers made by the various processes. SEM photographs are at various magnifications. All wires are 0.0005" (12.5 μm) diameter.

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§2. Electrical Considerations

§2.1 Whisker Resistance

§2.1.1 DC Resistance

Assuming uniform current flow in the whisker, and radial current flow in the tip, the DC resistance is (see Fig. 2.1(a))

$$R_{DC} = \underbrace{\frac{\rho L}{\pi r_1^2}}_{\substack{\text{uniform length } L \\ R_{DC,\,L}}} + \underbrace{\frac{\rho \tan \frac{\alpha}{2}}{2\pi \left(1 - \cos \frac{\alpha}{2}\right)} \left[\frac{1}{r_0} - \frac{1}{r_1}\right]}_{\substack{\text{tip} \\ R_{DC,\,tip}}}$$

The DC tip resistance of a phosphor-bronze whisker is plotted in Fig. 2.1(b) as a function of α, r_0, and r_1. The DC resistance per 0.001" of the straight part of the whisker is plotted in Fig. 2.1(c) as a function of wire radius r_1.

§2.1.2 RF Whisker Resistance

At high frequencies the current in the whisker can be considered to flow with uniform density in a skin of depth δ on the surface of the whisker, where

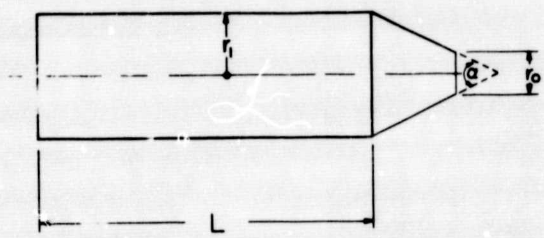

FIG. 2.1(a)

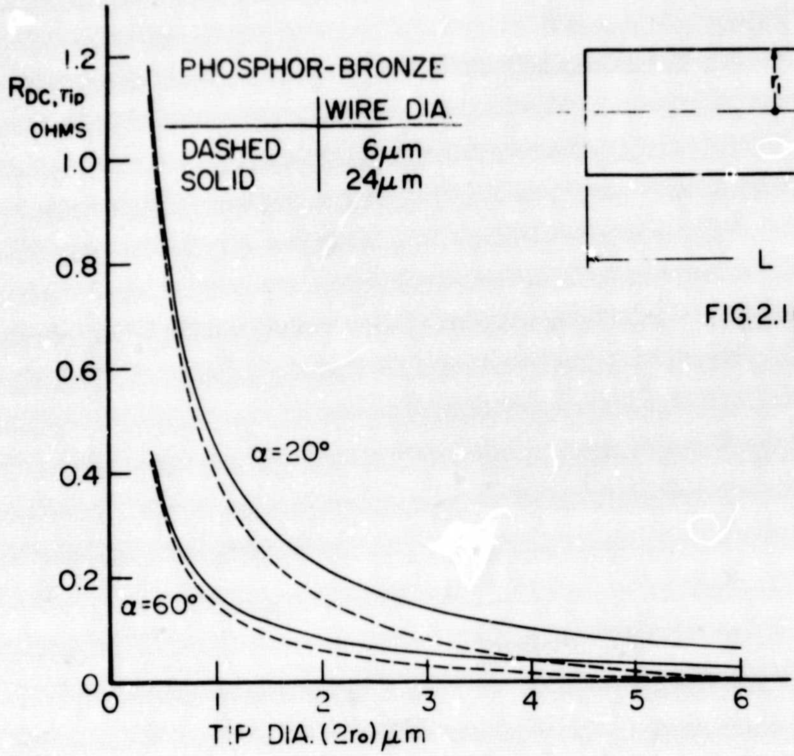

FIG. 2.1(b)

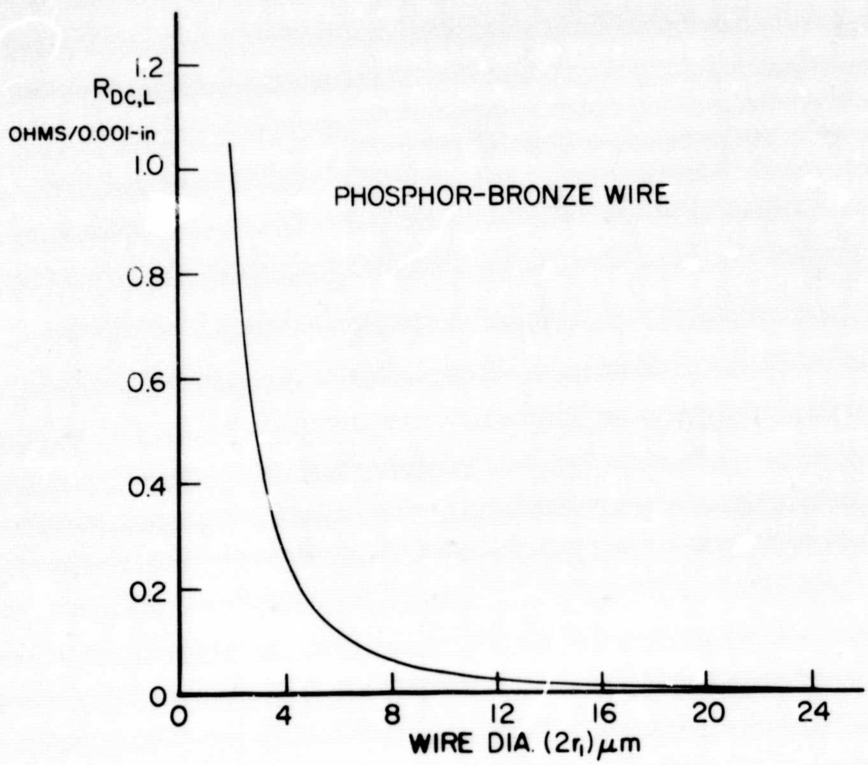

FIG. 2.1(c)

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$$\delta = \sqrt{\frac{2\rho}{\omega\mu}}$$

$$= 1590 \sqrt{\frac{\rho(\text{ohm-cm})}{f(\text{GHz})}} \ \mu\text{m} \quad \text{(for non-magnetic conductors)} .$$

For gold at 100 GHz, $\delta \simeq 0.25\ \mu$m. Hence if a whisker is plated with gold to a depth $>>0.25\mu$m, all the RF current will flow in the gold.

For a whisker of length L, with a tip radius $r_0 >> \delta$, the RF resistance is

$$R_{RF} = \underbrace{\frac{\rho L}{2\pi r_1 \delta}}_{\substack{\text{uniform length L} \\ R_{RF,\ L}}} + \underbrace{\frac{\rho}{2\pi\delta \sin(\alpha/2)} \ln \frac{r_1}{r_0}}_{\substack{\text{tip} \\ R_{RF,\ tip}}}$$

The RF tip resistance at 100 GHz of a gold plated whisker is plotted in Fig. 2.2(a) as a function of α_1, r_0, and r_1. The RF resistance per mil of the uniform part of the whisker is shown in Fig. 2.2(b) as a function of wire radius r_1. These graphs can be used for other frequencies by multiplying the resistance values by $\sqrt{\frac{f(\text{GHz})}{100}}$.

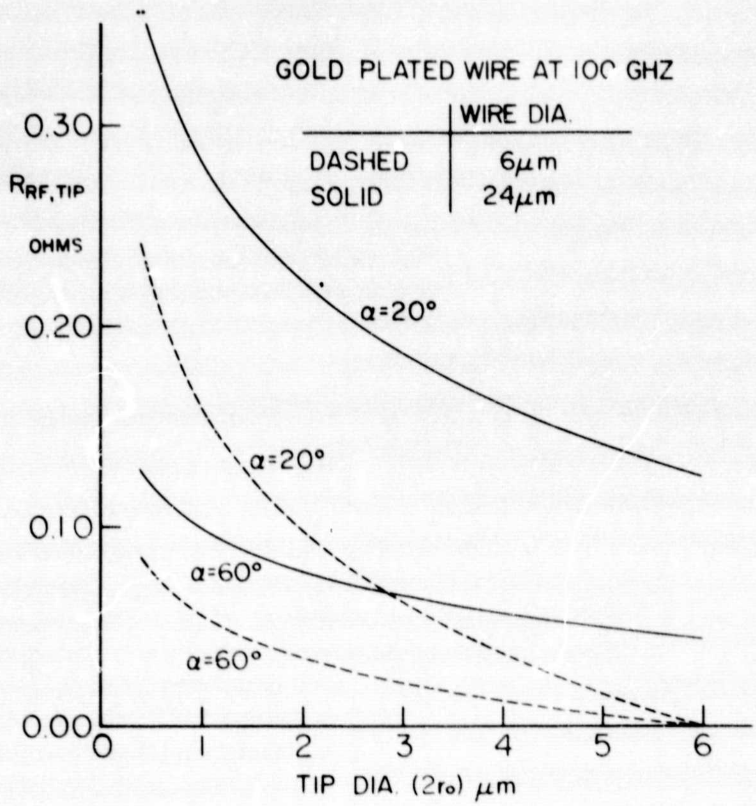


FIG. 2.2(a)

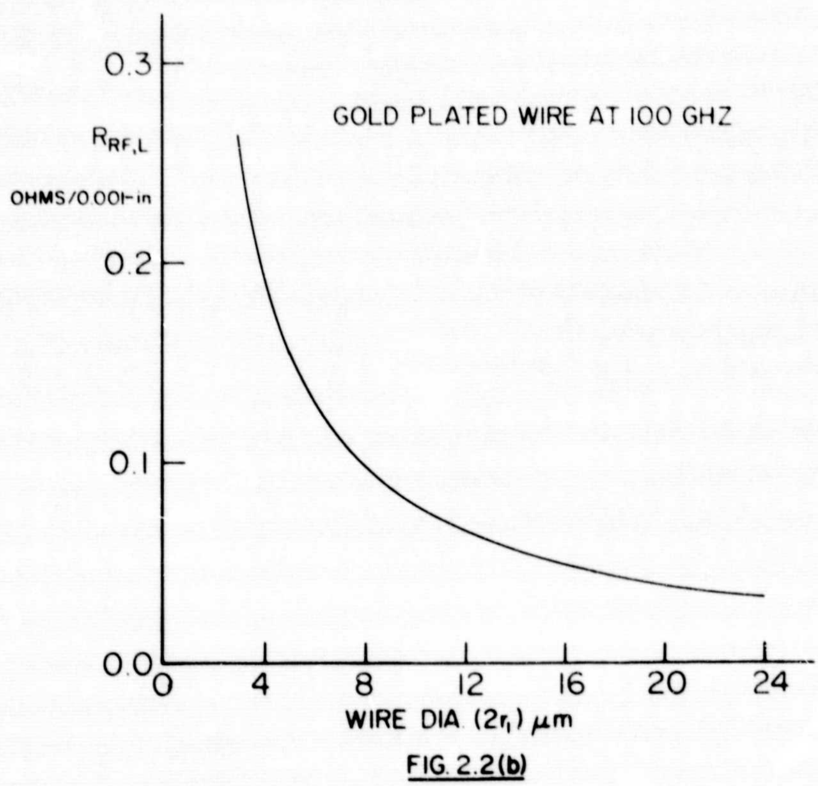


FIG. 2.2(b)

§2.2 Whisker Tip Inductance

The equivalent circuit of a diode contact whisker is a complicated function of the geometry and dimensions of the diode mounting structure.[6] However for the purpose of comparing whiskers with different tip shapes it is possible to consider only the inductance due to magnetic energy stored in the vicinity of the whisker tip, provided the tip dimensions are much smaller than a wavelength. We shall use the term tip inductance (ΔL_{tip}) to denote the excess inductance of the pointed wire over that of a uniform wire of the same length. The tip inductance is then the inductance due to the magnetic energy stored in the shaded region in Fig. 2.3(a), plus a usually small correction for the difference in skin inductance between the uniform wire and the conical point. Hence, with reference to Fig. 2.3(a),

$$\Delta L_{tip} = L_{shaded\ region} + \left[L_{skin,\ point} - L_{skin,\ uniform} \right]$$

where

$$L_{shaded\ region} = \frac{\mu_0}{2\pi} \cot(\alpha/2) \{ r_1 - r_0 - r_0 \ln(r_1/r_0) \} \ \mathrm{H}$$

$$L_{skin,\ point} = \frac{\delta\mu}{4\pi \sin(\alpha/2)} \cdot \ln \frac{r_1}{r_0} \quad \mathrm{H} \qquad (\propto \omega^{-\frac{1}{2}})$$

$$L_{skin,\ uniform} = \frac{\delta\mu}{4\pi \sin(\alpha/2)} \cdot \left(\frac{r_0}{r_1} - 1 \right) \cos(\alpha/2) \quad \mathrm{H} \qquad (\propto \omega^{-\frac{1}{2}})$$

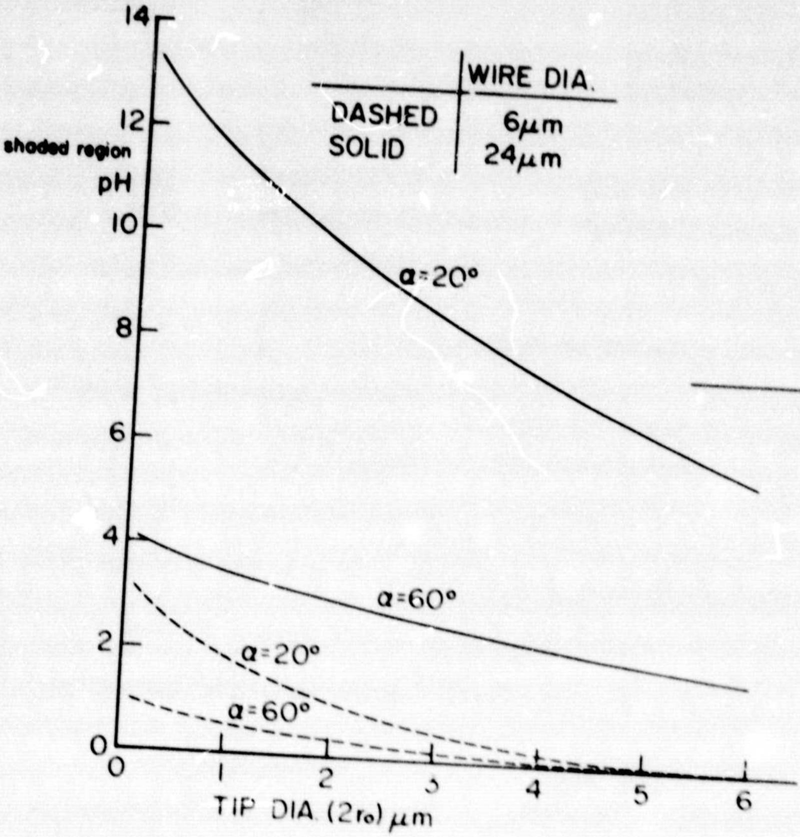


FIG. 2.3(b)

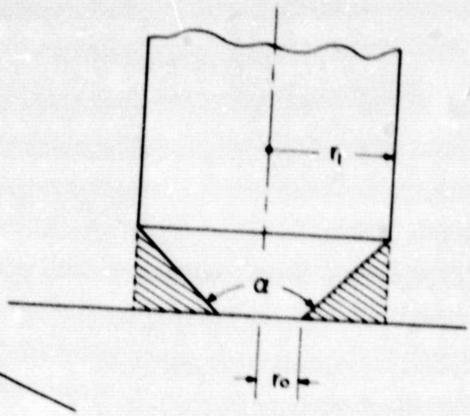


FIG. 2.3(a)

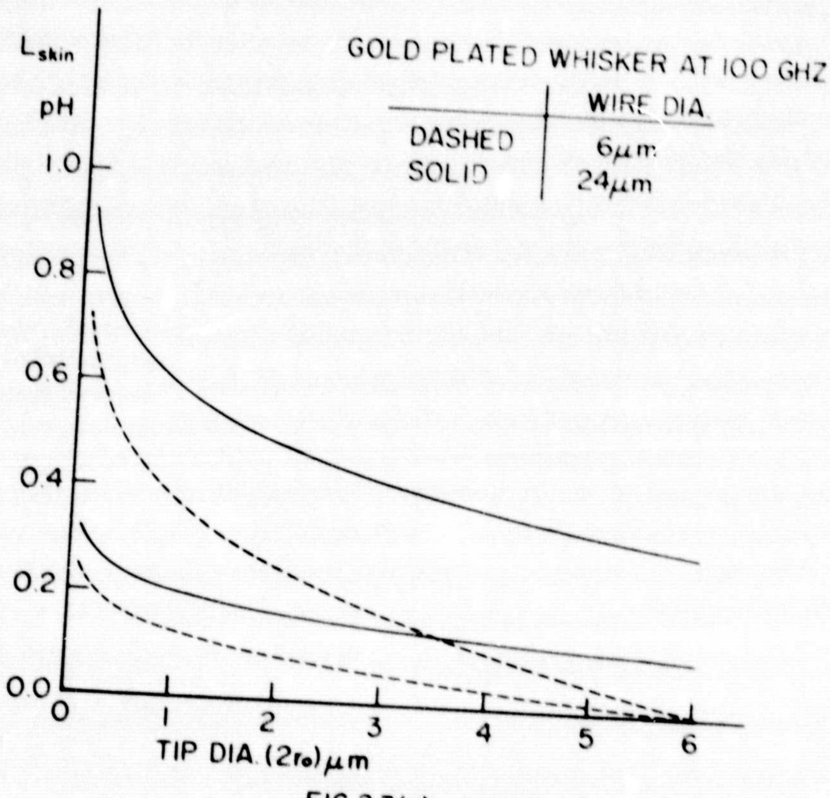


FIG. 2.3(c)

The tip inductance is shown in Fig. 2.3(b) & (c), separated into its constant and $1/\sqrt{f}$ - dependent components, for gold plated whiskers at 100 GHz.

§2.3 Whisker Capacitance

The capacitance between the tip of the contact whisker and the surface of the diode chip appears in parallel with the junction capacitance of the diode, and hence plays a role in determining the microwave performance of a mixer or detector.

There appears to be no simple analytical solution [7] for the electric field between a ground plane and a conducting surface of revolution whose axis is perpendicular to the ground plane. For an ellipsoidal surface an exact solution exists [8], but this is not a good approximation to our pointed wire. For a cylinder with closed ends, techniques for computer solution have been proposed by Smythe [7] and Marin [9], but these are not simple to implement.

To compare the whisker-to-chip capacitances of whiskers with various point shapes and diameters we resort here to a convenient approximate analysis, with the following assumptions: (see Fig. 2.4(a))

(i) The electric field between the conical sides of the whisker tip and the surface of the diode chip is toroidal.

(ii) The electric field between the uniform section of the whisker wire and the surface of the chip is also toroidal.

(iii) The electric fields at distances greater than ~0.005" from the tip will be independent of the tip geometry for all practical cases.

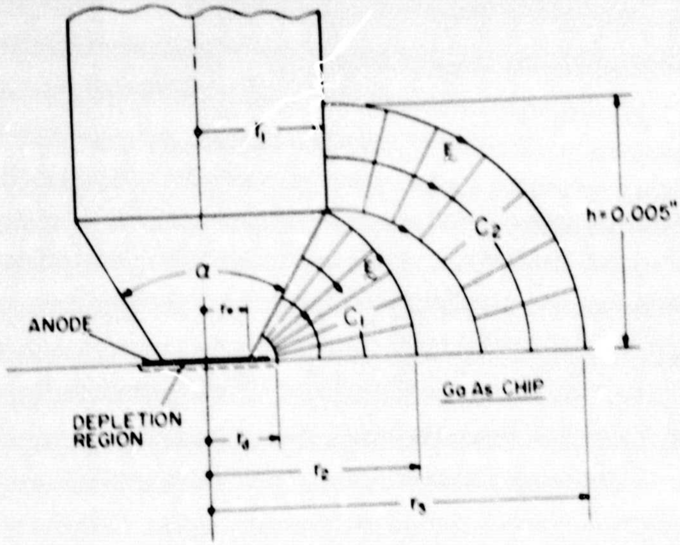


FIG 2.4(a)

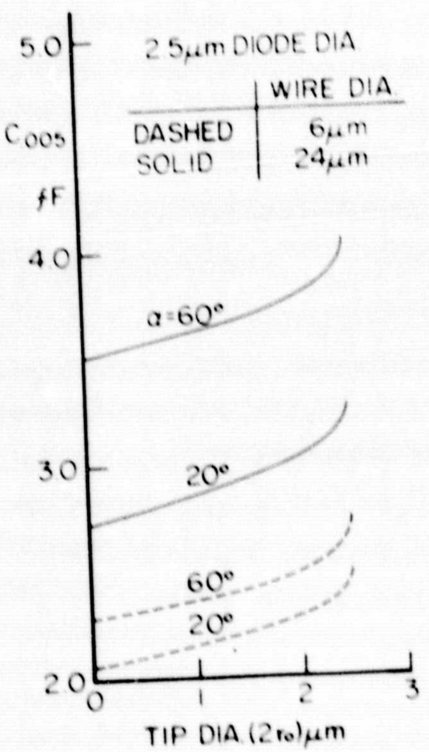


FIG 2.4(b)

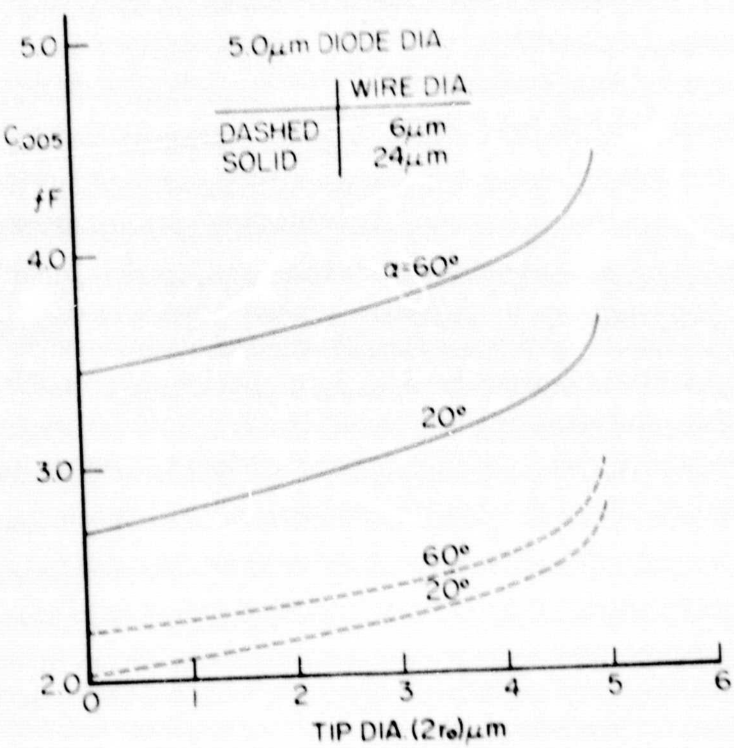


FIG 2.4(c)

For comparison purposes the approximate whisker-to-chip capacitance, $C_{.005}$, of the last 0.005" of the whisker will be calculated for a number of different whisker geometries and diode diameters. With reference to Fig. 2.4(a), we have: $C_{.005} \simeq C_1 + C_2$, where C_1 and C_2 depend on the diode diameter, contact diameter, wire diameter, and tip cone-angle. Typical values of $C_{.005}$ are shown in Fig. 2.4 for 2.5 and 5μm diameter diodes.

If the whisker has too large a tip diameter, or is pushed too hard onto the diode, so that it is in contact with the chip outside the perimeter of the anode metallization (see Fig. 2.5(a)), there will be an additional capacitance C_3 in parallel with C_1 and C_2. C_3 depends on the thickness of the SiO_2 layer on the semiconductor chip, and a typical value of 0.3μm has been assumed in calculating the results in Fig. 2.5(b).

The graphs in Figs. 2.4 and 2.5 were computed using the following equations:

$$C_{.005} = C_1 + C_2$$

$$C_1 = 2\pi \epsilon_o \sqrt{B^2 - A^2} \Bigg/ \ln \left| \frac{\sqrt{B^2 - A^2}\ \tan\phi + (A+B)}{\sqrt{B^2 - A^2}\ \tan\phi - (A+B)} \right| \quad \text{for } B > A$$

or

$$C_1 = \pi \epsilon_o \sqrt{A^2 - B^2} \Bigg/ \text{arc tan} \frac{\sqrt{A^2 - B^2}}{A+B} \tan\phi \quad \text{for } A > B$$

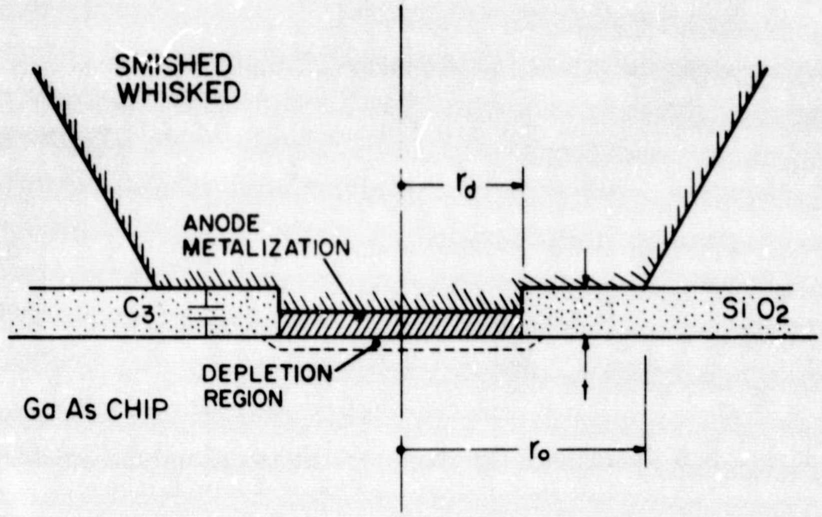


FIG. 2.5 (a)

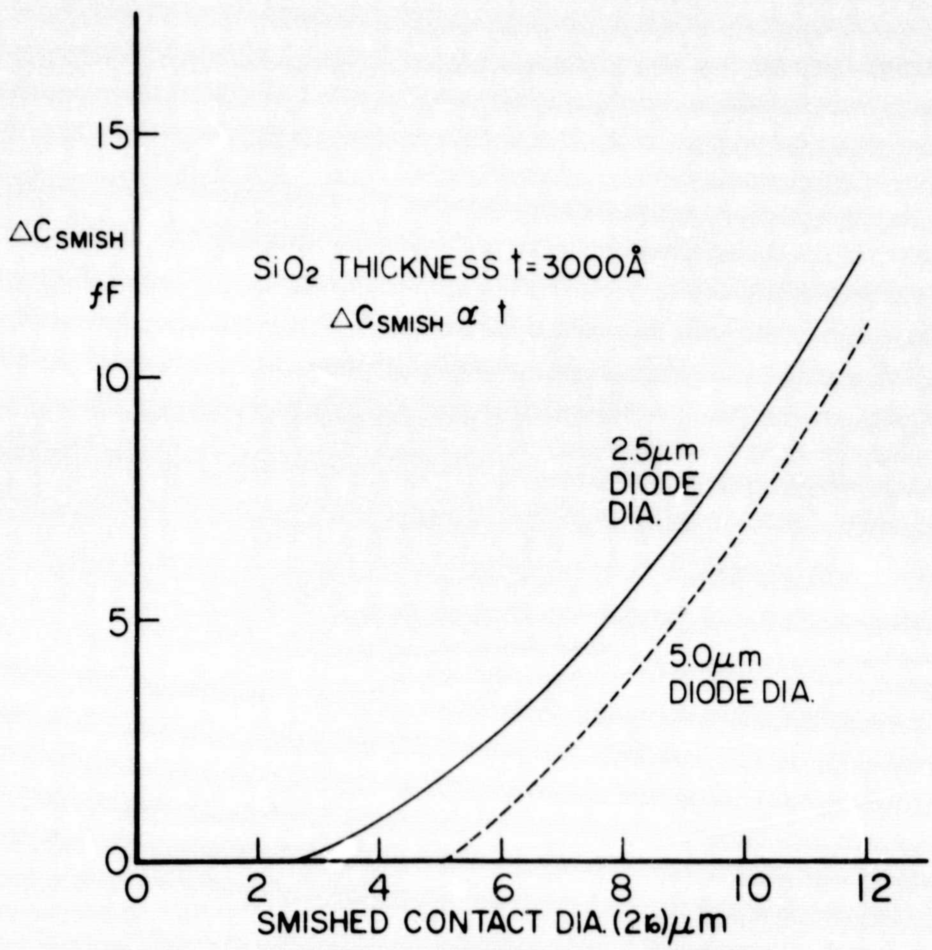


FIG. 2.5 (b)

where $$A = r_0 \ln \left(\frac{r_2 - r_0}{r_d - r_o} \right)$$

$$B = r_2 - r_d$$

$$\phi = (\pi - \alpha)/4$$

$$C_2 = 2\pi \epsilon_o \sqrt{B^2 - A^2} \Bigg/ \ln \left| \frac{\sqrt{B^2 - A^2} + (A+B)}{\sqrt{B^2 - A^2} - (A+B)} \right| \quad \text{for } B > A$$

or

$$C_2 = \pi \epsilon_o \sqrt{A^2 - B^2} \Bigg/ \text{arc tan} \; \frac{\sqrt{A^2 - B^2}}{A+B} \quad \text{for } A > B$$

where $$A = r_1 \ln \left(\frac{h \tan(\alpha/2)}{r_1 - r_o} \right)$$

$$B = h - (r_1 - r_o) \cot(\alpha/2)$$

$$C_3 = \pi \epsilon_r \epsilon_o (r_o^2 - r_d^2)/t \quad \text{for} \quad r_o > r_d$$

$$= 0 \quad \text{for} \quad r_o \leq r_d$$

where $\epsilon_r \sim 3.8$ for SiO_2

§3. General Fabrication Considerations

In this section the main steps in pointing, plating, and testing phosphor-bronze contact whiskers are discussed in detail. The complete step-by-step fabrication procedure is given in §4.

Some differences of opinion exist on whether a contact whisker should be pointed before or after it is bent to its final spring shape. For the very short whiskers used in mixers above 100 GHz we have found it more efficient to point before bending. Although this means that care must be taken not to damage the point while bending the whisker - which is not difficult if a well designed bending jig is used - it has the following advantages over the bend-before-point approach: (i) if a good point is not obtained, the wire can be pointed again; (ii) the distance from the whisker tip to the first bend can be better controlled; and (iii) there is no problem arising from the etching solution tending to climb around the first bend. Throughout this section and section 4 it is assumed that a whisker ~1/4-inch long is attached to the end of a 0.03-inch diameter nickel post for ease of handling. After pointing and plating the whisker would normally be bent into the desired shape, using a bending jig, and then soldered to the final whisker post to be used in the diode mount.

§3.1 Cleaning

Phosphor-bronze wire that has been stored for any length of time will have a certain amount of surface tarnish. To enable the tip of the wire to etch to a symmetrical final shape it is important to remove this tarnish along with any other contaminants that are present. The most successful cleaning agent we have found for removal of tarnish from phosphor-bronze wire is a 50% (by volume) solution of hydrochloric acid in distilled water at room temperature. The wire is not damaged, pitted or measurably reduced in diameter after immersion, with agitation, for one minute in this solution.

Immediately prior to the HCl dip particles of dirt, flux residue, oil and grease etc., are removed by ultrasonically cleaning the wire in distilled water for one minute, then in methanol for one minute.

Following the acid dip, the wires are ultrasonically cleaned again in water and then in acetone for one minute each. There are no other chemicals used for cleaning the wires. Each wire should be etched to a point immediately after cleaning to avoid recontamination.

After etching, the whisker assembly should be cleaned with the water, the HCl dip, water again, and finally acetone, to remove encrustations of by-products of the etching process that remain on the surface of the wire near the tip. One minute in each bath, with ultrasonic agitation, is sufficient.

§3.2 Straightness

It is important that the tip of the wire enter the etchant normal to the surface so that the point is symmetrically formed. Phosphor-bronze wire as it comes off the reel is seldom perfectly straight. With the length of wire used ($\sim$ 1/4-inch) the ends may be up to ten degrees out of line with each other. Adjustment should be made, after soldering the wire to the post, by bending the wire at its base until the far end of the wire is parallel to the post. The far end of the wire will not then lie on the axis of the nickel post but this is not important since, before etching, the wire can be centered in the etching cell.

§3.3 Storage

During handling and storage the wire may electrostatically attract and hold chemically active dust particles which are difficult to remove. Once the wires have been mounted on posts and cleaned it is best to transfer them immediately to an inert dust free atmosphere. If this is not done the wires may eventually become too contaminated for proper etching to take place. We find that a metal storage rack in a jar containing anhydrous calcium sulphate desiccant crystals is satisfactory: the metal rack appears better than a plastic one as the whiskers are less inclined to acquire a static charge which would attract dust.

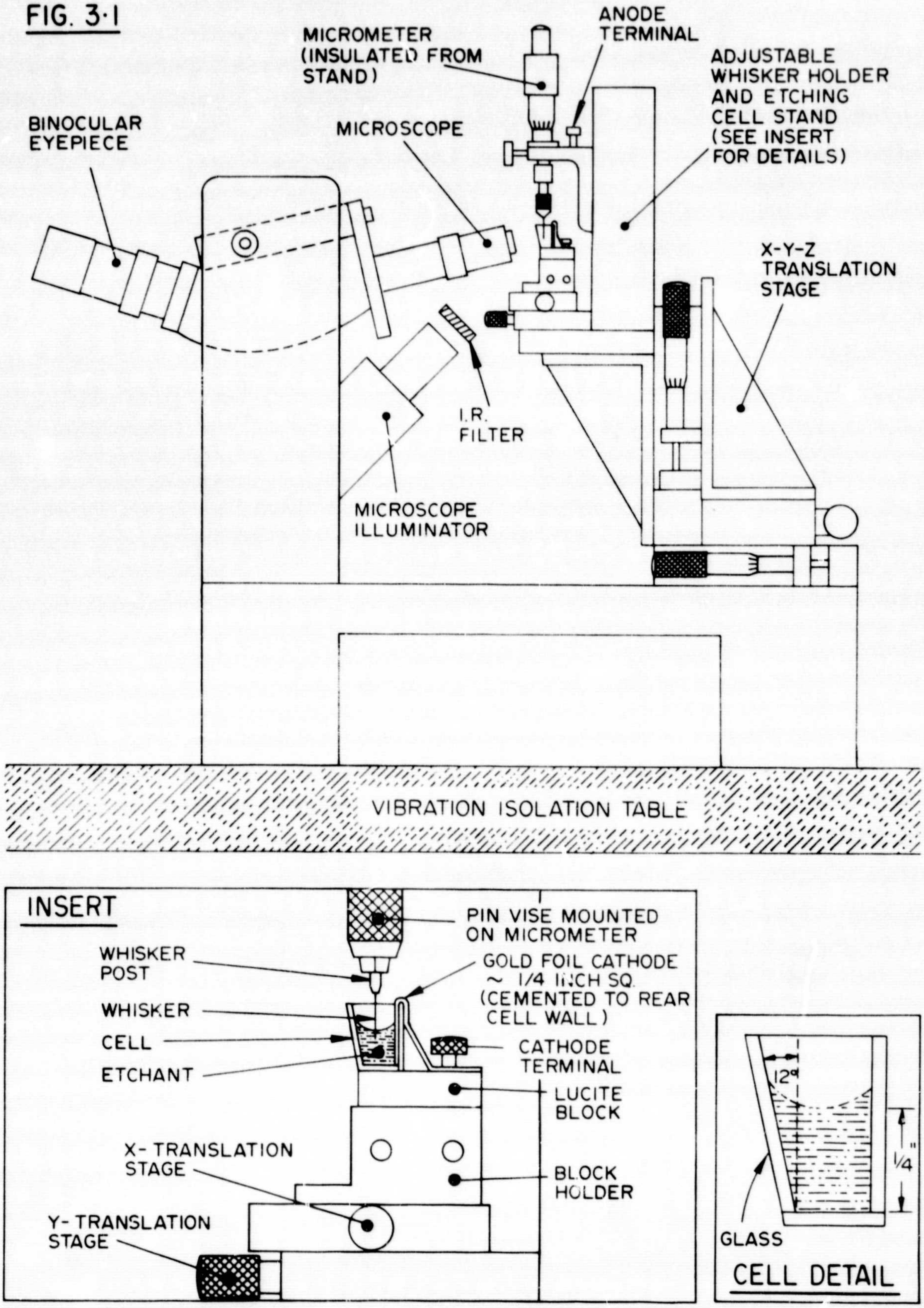
§3.4 Etchant Vibrations

It is important to have a completely steady air/etchant interface since this is where the final shaping of the whisker tip is done. Low frequency building vibrations can cause waves on the surface of a large etching cell, even if a pneumatic vibration isolation table is used. It has been found that a relatively small etching cell damps out waves sufficiently: the dimensions of the cell should be $\sim 1/4$ inch on each side - even a 1/2 inch cell has been found too large. The main disadvantage of the small cell is that the meniscus at the cell walls gives a horizontal surface only at the center of the cell, and the whisker must be positioned at this point if a uniform tip is to be achieved.

§3.5 Whisker Pointing Apparatus

The apparatus used for pointing whiskers is shown in Fig. 3.1. Its essential features are: (i) A glass cell for the electrolyte - see §3.6 - with adjustments which allow the whisker to be located at the correct depth and near the center of the cell. (ii) A microscope which allows the whisker to be viewed from below the surface of the

FIG. 3·1


MICROMETER (INSULATED FROM STAND)
ANODE TERMINAL
ADJUSTABLE WHISKER HOLDER AND ETCHING CELL STAND (SEE INSERT FOR DETAILS)
BINOCULAR EYEPIECE
MICROSCOPE
X-Y-Z TRANSLATION STAGE
I.R. FILTER
MICROSCOPE ILLUMINATOR
VIBRATION ISOLATION TABLE
INSERT
PIN VISE MOUNTED ON MICROMETER
WHISKER POST
GOLD FOIL CATHODE ~ 1/4 INCH SQ. (CEMENTED TO REAR CELL WALL)
WHISKER
CELL
ETCHANT
CATHODE TERMINAL
LUCITE BLOCK
X-TRANSLATION STAGE
BLOCK HOLDER
Y-TRANSLATION STAGE
12°
1/4"
GLASS
CELL DETAIL

electrolyte. A 15 mm working distance lens* is used with 20X eyepieces. (iii) X-Y-Z adjustments which enable the whisker and cell to be brought into the field of the microscope, and focused. (iv) An illuminator with an infrared filter to prevent heating of the electrolyte. (v) A power supply with a setting accuracy of ~1 mV.

§3.6 The Cell Used for PCE Solution

As mentioned in §3.4 a 1/4 inch cubic cell was found to be nearly optimum for suppressing surface ripples. The cell is fabricated from pieces of microscope slide glued together using epoxy adhesive.**

The choice of cathode material for use with PCE solution was found to be quite critical. A platinum cathode had been used successfully for pointing fine wires (tungsten, phosphor-bronze, niobium) in a sodium hydroxide electrolyte, and accordingly platinum foil was initially tried with the PCE solution. In this case however we found that after using the same piece of platinum for a while an increased voltage was required to produce a pointed whisker; the more the cathode was used, the higher the voltage. Eventually more than twice the initial voltage was needed to etch the whisker. If the cathode was scrubbed clean, the excess voltage was temporarily reduced. Stainless steel exhibited the same behavior, but with a gold cathode the difficulty disappeared. This effect is probably a result of hydrogen adsorption on the surfaces of the platinum and steel cathodes. The required voltage using a gold cathode is ~750 millivolts, and is quite repeatable provided fresh PCE solution is used every day. Cell temperature has some influence on the required voltage; however at normal room temperatures the effect is small enough to be ignored. (Heat from the illuminator is removed by the IR filter - Fig. 3.1).

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*Nikon type 77254 U20, N.A. 0.33, 15 mm working distance.

**Sears & Roebuck Co., Craftsman #9 8059.

§3.7 Whisker Etching

The shape of the etched whisker can be quite effectively controlled by the applied voltage, and to a lesser extent by the length of wire immersed in the etchant. "Zero" immersed length has been found most conducive to forming a 60-90 degree cone angle: this is achieved by lowering a wetted wire until it just contacts the surface of the liquid, whereupon the meniscus rises 0.0010" to 0.0015" up the wire (when using 0.0005" diameter wire). A voltage of ~0.750V is applied to the wire after immersion, and etching is allowed to continue until the meniscus falls away from the point. The voltage applied to the whisker has a strong effect on the cone angle and the tip diameter. Variations of only a few tens of millivolts can make the difference between cone angles as blunt as 145 degrees and as sharp as 30 degrees.

Observing the etching action through the microscope after the voltage has been applied, a mass of small bubbles is seen surrounding the wire tip. Usually the view of the tip is obscured by the bubbles. The total etching time should be ~90 sec., during which the etching current can be recorded, providing useful indication of whether the process is proceeding normally. The current vs. time graph should proceed smoothly down from the initial high current, and break abruptly at completion of etching - see Fig. 3.2. A short total etch time results in a narrow cone angle, while a long etch time forms a wide angle, or even a completely blunt tip.

The detailed mechanism of etching is not understood, but from numerous observations it appears that a transparent sheath forms around the wire during etching, and is continuously dissolved and reformed. This sheath can be easily observed in the microscope when the applied voltage is too low, appearing as a transparent cylinder that remains attached to the end of the wire. If the voltage is too low this sheath

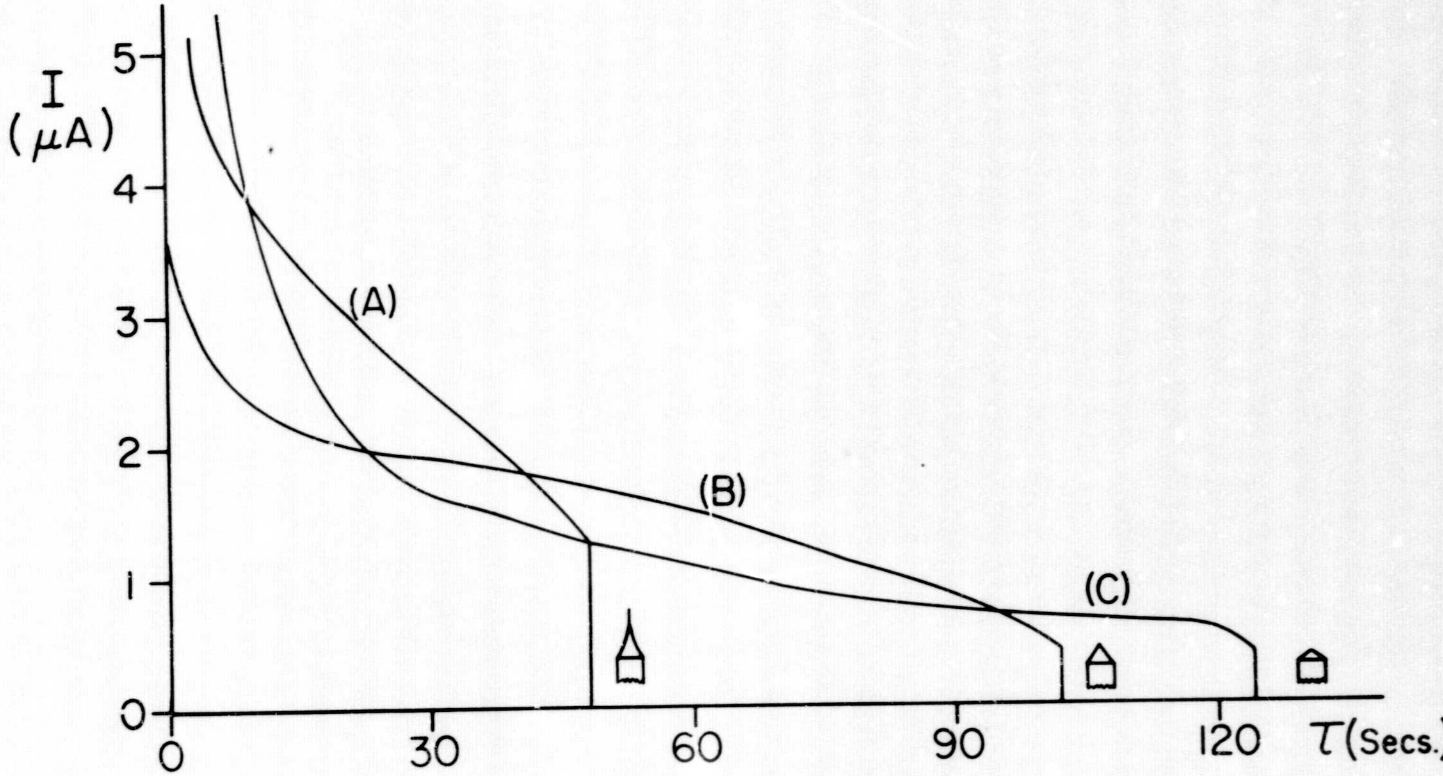

Fig. 3.2

prevents the correct pointing action on the wire because the meniscus formed by the etchant surface remains attached to the cylinder long after etching should have ceased. Under these circumstances etching continues inside the cylinder, eroding the tip until no cone is left, and the wire once again becomes flat tipped. An intermediate condition (one that is to be avoided also, since the result is usually an asymmetrical cone) is that the cylinder breaks off from the wire in one or more pieces instead of either staying on, or being continuously dissolved.

After etching is complete and the voltage switched off, the finished whisker may be lowered into the etchant again for inspection through the microscope. The shape can be seen, and a rough idea of the tip diameter can be obtained with some experience. If the shape is not satisfactory the whisker may be re-etched. If the whisker is well-formed but its tip diameter is too small, it may be blunted using the pulse blunting technique (§3.8). If a scanning electron microscope is available the whisker should be inspected in the SEM before and after blunting. Optical microscopes would have to be operated beyond their resolution limit in order to measure accurately a whisker point made to contact a 2 micron diameter diode.

§3.8 Pulse Blunting

It has been found that by immersing the tip of the whisker in a sodium hydroxide solution (10% by weight) and applying relatively high positive voltage pulses to the whisker, metal is etched chiefly from the actual whisker point. The simplest method of applying the pulses to the whisker is to allow a capacitor to discharge through a push-button switch, using the circuit shown in Fig. 3.3. A platinum cathode is used.

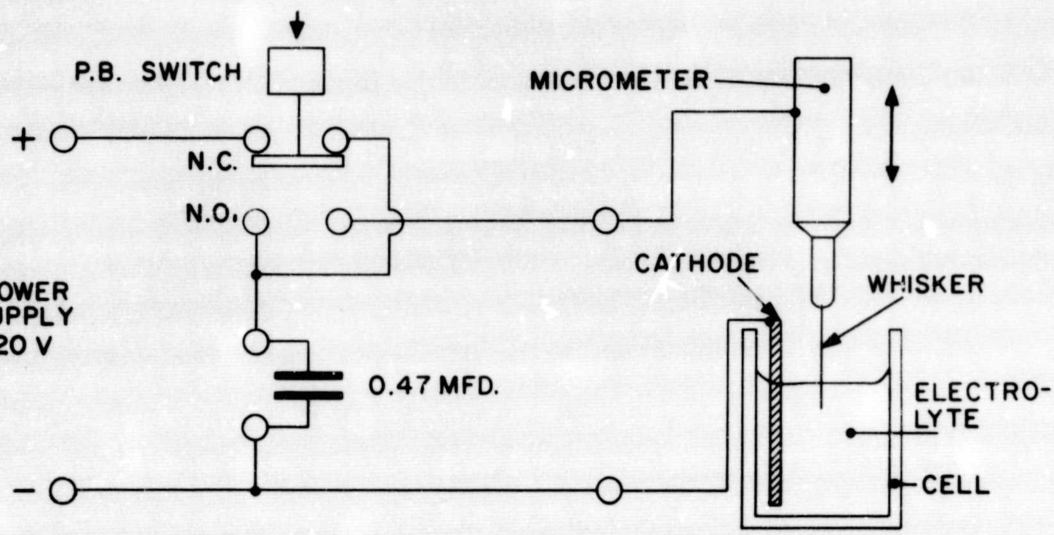

Fig. 3.3

Experiments were made to determine a set of satisfactory operating conditions. By fixing the capacitor at 0.47 μF it was found possible, with 0.003" of whisker immersed, to achieve a controllable rate of blunting. 10 to 20 pulses with the capacitor charged up to the supply voltage of 20V produces a tip diameter of ~1.5μ from a very sharp tip.

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The etching apparatus of Fig. 3.1 was used for pulse blunting. This enabled the tip to be observed between pulses. An SEM was used for final measurements after blunting.

§3.9 Cleaning Before Gold Plating

To achieve uniform gold plating it is essential to clean the whisker thoroughly. We have tried several chemical cleaning solutions, and also cathodic electrocleaning, but without satisfactory results. However, good plating has been achieved following a short period of sputter-etching by argon ions.

A dc sputtering system is used*, with the whisker positioned vertically on the cathode. Other whisker orientations were tried (horizontal, and partially recessed into the cathode) but these suffered from excessive back-sputtering of cathode material onto the whisker. With the whisker and post standing point uppermost on the cathode, most of the etching is accomplished in the middle** of the whisker and on the nickel post. It is found that enough cleaning action takes place at the tip when the amount of material removed from the middle of the wire amounts to ~7% of the original diameter. The cathode to anode spacing is set at ~2 inches. This is greater than the spacing normally used in this instrument, but results in more stable operation at the low current necessary to avoid overheating the whisker. A current of ~3 mA was found to be the maximum current permissible*** during sputter etching. The argon pressure, 10-20

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*Technics Inc., Alexandria, VA, Model "Hummer II".

**It is thought that the tapering action may be caused by splashing of the stream of ions incident on the tip of the whisker, thereby deflecting ions from the part of the wire just beneath the tip. At the center of the wire the ions impinge at an effective angle again, while at the bottom of the wire they are travelling more nearly parallel to the wire, and therefore cause less etching.

***Excessive current melts the solder (96° C MP) holding the whisker on its post, or warps the wire.

microns, is adjusted to maintain $\sim$3 mA at fixed voltage. The whiskers are etched in batches of 5 under these conditions for 5 minutes.

Inspection of the sputter-cleaned whiskers in our ISI Mini-SEM was found to recontaminate the surface. After examination at high magnification, the area scanned by the electron beam was clearly visible at lower magnification as a slightly darkened region. A suggested cause of this is that diffusion-pump oil vapor is decomposed on the whisker by the energy of the electron beam. Consequently we transfer the cleaned whisker directly to the plating bath without intermediate inspection.

§3.10 Gold Plating

Satisfactory gold plating is achieved using a commercial plating solution (Selrex: Antronex-N) at room temperature, with the current density recommended by the manufacturer (3mA/cm^2). Ultrasonic agitation is used during plating: this appears to give a more uniform plate -- probably by releasing bubbles evolved on the metal surface which would otherwise block plating in their vicinity. The area of plating is determined by immersing the whole of the whisker and a measured length of the nickel whisker-post before applying the current. Typically the 0.030 inch diameter posts are immersed to a depth of 0.10 inch, which, for 3 ASF, requires a current of 0.2 mA. After 3 mins. plating time the calculated plating thickness is $\sim$1600Å.

During the plating operation cleanliness is extremely important. We use fresh gold plating solution each day, even though the old solution has obviously not been exhausted. Contamination from the atmosphere by dust and organic matter is likely to give poor results with old solutions.

§3.11 Test of Plating Quality: the Push Test

After the whisker has been gold plated, the quality of the gold plating at the tip can be tested, without damage to the whisker, by measuring the force needed to make a good electrical contact with a clean gold surface. This may be done as shown schematically in Fig. 3.4 by bringing the whisker tip into contact with a thin piece of gold foil, and measuring the contact resistance vs. the deflection of the foil. The foil we use is a piece of beryllium doped gold (10 ppm Beryllium) ~0.5 inch free length and 0.040 inch wide by 0.0012 inch thick, which gives a spring constant, measured at its free end, of 0.7 mg per 0.001 inch of deflection. No permanent tip deformation has been observed for whiskers with cone angles of 70° - 90° with contact forces up to ~4 mg.

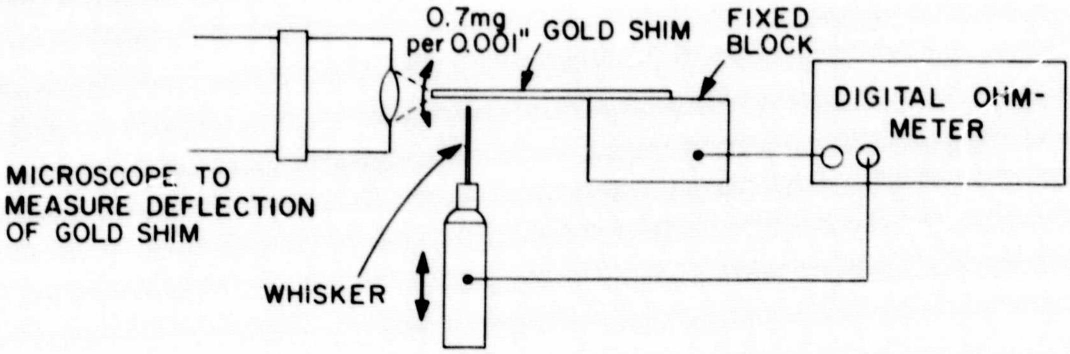

Fig. 3.4

A typical resistance curve is shown in Fig. 3.5 for plated whiskers. We have found that for all whiskers there is ~0.5Ω excess resistance, even at 4 mg. contact force, that can not be attributed to lead resistance. Calculations show that ~0.5Ω can be accounted for by current crowding at the point of contact if the effective diameter of the contact area is ~0.1 micron. The fact that no tip damage is observed under the SEM after the push test indicates a contact diameter <0.1 micron. (SEM resolution is ~500Å).

It should be noted that the contact force between whisker and diode when a diode is contacted in the usual way, is sufficient to produce considerable deformation of the whisker tip, and is therefore much greater than the forces used in this simple "push-test".

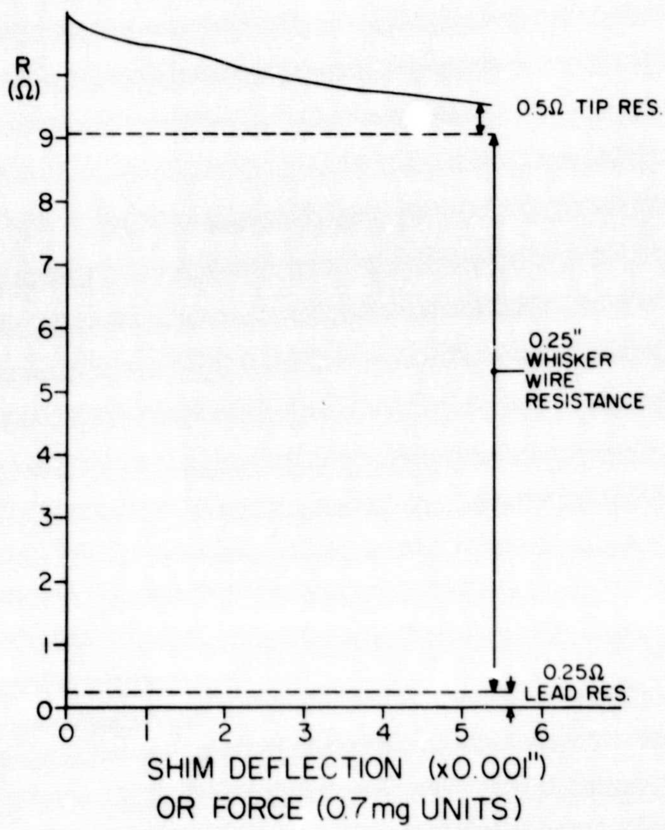

Fig. 3.5

§4. Step-By-Step Fabrication Procedure

This section lists, in order, the essential steps in making phosphor-bronze contact whiskers. The compositions of the various solutions are given in the appropriate sections.

§4.1 Solder ~0.25 inch lengths of 0.0005 inch diameter phosphor-bronze* wire axially to the ends of ~0.5 inch long posts made from 0.030 inch diameter nickel wire. The solder used is Alpha B20E2 (M.P. 96°C) with Supersafe #30 organic flux.** Trim the ends of the wires flat with a small guillotine.

§4.2 Clean these assemblies of all residual flux (Supersafe #30 is water soluble) by immersion in distilled water, with ultrasonic agitation, for one minute, then in methyl alcohol, with ultrasonic agitation, for one minute. Store these assemblies as explained in §3.3.

§4.3 Immediately before pointing the whisker, dip the whisker in a 50% solution by volume of hydrochloric acid and distilled water (use a fume hood) for one minute with agitation. Rinse off the acid with distilled water while still under the fume hood. Clean in distilled water, with ultrasonic agitation, for 30 seconds, then in acetone, with ultrasonic agitation, for 30 seconds.

§4.4 Mount the whisker in the pointing apparatus (§3.7). Fill the 1/4" gold-cathode cell with PCE solution, made up from 100 ml phosphoric acid, 12.5 gm chromium trioxide

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*C-Bronze 92% Cu, 8% Sn, stress relieved, from Sigmund Cohn, Mt. Vernon, N. Y.

**Superior Mfg. Co., Cleveland, Ohio.

and 39.5 ml water. Wet the end 0.005 inch of the wire in the PCE solution, withdraw, and adjust for "zero" immersion depth as explained in §3.7. Etch the wire until the meniscus falls away, as explained in §3.7, observing progress through the microscope, and record the etching current vs. time using a chart recorder.

§4.5 Wash the assembly in distilled water, with ultrasonic agitation, for 30 sec., immerse in 50% hydrochloric acid (use fume hood) for one minute to remove etching by-products, then rinse off the acid in distilled water while still under the fume hood. Clean, with ultrasonic agitation, in distilled water for one minute, and then in acetone, with ultrasonic agitation, for one minute.

§4.6 If an SEM is available check the whisker for quality at this stage. If necessary blunt the point using the pulse-blunting procedure described in §3.8, using a cell with a platinum cathode and 10% (by weight) solution of sodium hydroxide. Reclean as in §4.5 before reinspection.

§4.7 Sputter etch the whisker assembly for 5 minutes as detailed in §3.9. Gold plate immediately - do not examine in SEM.

§4.8 Gold plate to the required thickness using Selrex: Autronex-N gold plating solution as detailed in §3.10.

§4.9 Wash the gold plated assembly in distilled water, then in acetone, using ultrasonic agitation, for 30 seconds each.

§4.10 Test the contact resistance as detailed in §3.11.

§4.11 Store the finished whisker in a dessicator until required for use.

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