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A TECHNOLOGY OF TUNNEL DEVICES FOR MILLIMETER-WAVE APPLICATIONS

by L. E. Dickens

Prepared by
JOHNS HOPKINS UNIVERSITY
Baltimore, Md.
for Goddard Space Flight Center

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FIGURE PAGE

I. INTRODUCTION

The purpose of the work herein reported is the development of tunnel diode fabrication techniques and circuits for millimeter-wave receivers with improved sensitivity.

This report will be concerned primarily with the details of the experimentally and in many cases empirically determined procedures used in the fabrication of the tunnel devices which have thus far proved quite effective up to 70 gc.

The tunnel devices herein considered are all tunnel diodes, but are classified as back-diodes, amplifier and mixer diodes, and oscillator diodes by the type diode (GaAs, GaSb, Ge), the peak current I_p and the peak current to valley current ratio (P/V).

The incentive for this project was supplied by the excellent groundwork done by R. Trambarulo and C. Burrus of BTL[1-8].

Part III describes in detail the method used to dope the semiconductor to the level of degeneracy required by the point contact diodes. A remelt process is used to effect the necessary mixing. This process is a variation of the original melting and mixing process described by Torrey and Whitmer[9].

Part IV presents the details of the fabrication of the complete devices, with a discussion of the various epoxy resins and combinations of resins used to encapsulate and hence ruggedize the point contact assembly.

Part V gives the procedure for electrically forming the junction between the point contact wire and the appropriate semiconductor material. Also given in this section is a discussion of the equipment used to monitor the metal to semiconductor contact and the subsequent junction forming.

Part VI briefly outlines some of the results thus far obtained with the diodes constructed according to these procedures, and shows some of the diode holders and other circuitry in which the diodes are tested.

II. PROJECT STAFF

The engineers contributing their efforts to this project and material for this report are:

Dr. L. E. Dickens
Mr. C. H. Chen
Mr. A. J. Fredriksen.

III. MATERIALS PREPARATION

The fabrication of point contact tunnel diodes requires the use of semiconductor material which is more highly doped than any commercially available. Because of this fact, this project has investigated the methods of redoping and recrystallizing semiconductor material from a simple melt. An RF heater is used not only for its good localizing of the heat, but also because the mixing action obtained by the induced currents should yield a more homogeneous product.

To obtain the necessary high frequency characteristics of the tunnel diodes, the semiconductor must be degenerately doped. For germanium and gallium arsenide to be degenerately doped requires dopant concentrations of the order of 10^{20} to 10^{21} atoms/cc. For Ge and GaAs, this corresponds approximately to the solid solubility level. That is, for example with Ge, 3 (atomic) % is required to yield 10^{20} donor concentration and this is also the solid solubility[10], a larger precentage of As in the Ge, upon freezing from the melt, would cause occlusions of the excess As to form rather than the host crystal accepting all the As either substitutionally or intersticially. Large single crystals of heavily doped semiconductor material are not needed, since in the construction of the diodes, only cylinders 0.020" in diameter by 0.020" in length are used. Heavily doped polycrystalline material with individual grain sizes of the order of 0.020" and larger is made by melting together an amount of semiconductor material with the appropriate amount of dopant in a sealed and evacuated quartz tube. The semiconductor and dopant are melted and mixed in the quartz ampoule using an RF induction furnace. The details of the process are given in what follows.

The optimum dopant concentrations for p-type gallium arsenide and n-type gallium arsenide, gallium antimonide and germanium were determined and the recipes are given in the following table.

Table I

p-type	GaAs/Zn	GaAs	95.7%	1.0000 g.
		Zn	2.41%	25.2 mg.
		As	1.89%	19.8 mg.
n-type	GaAs/Se	GaAs	95.59%	1.0000 g.
		Se	2.52%	26.3 mg.
		As	1.89%	19.7 mg.
n-type	GaSb/Te	GaSb	99.77%	1.0000 g.
		Te	0.23%	2.5 mg.
n-type	Ge/As	Ge	97.24%	1.0000 g.
		As	2.76%	28.4 mg.

Fused quartz is used to make the ampoule used to remelt the semiconductor and dopant because of its high temperature yield point (1,600°C) and because of its chemical inertness; that is, it does not react with the molten semiconductor and contributes a negligible amount of contamination impurities. What follows is a step by step procedure used in encapsulating the semiconductor and dopant in a quartz ampoule.

A section of standard thickness quartz tubing roughly nine inches long and with an inner diameter of 6 mm is clamped in a vertical position from a ringstand with the bottom of the tubing about six inches above the bench top. A hydrogen-oxygen flame is then applied to the tubing about one inch above the bottom. As the quartz softens, the pressure of the flame makes an indentation which is continually amplified by rotating the quartz or moving the flame around the work. Eventually the bottom section drops away. The tube bottom is rounded off and fire polished. Usually a white oxide coating forms on the quartz tubing during the preceding operation. This may be removed by rotating the work while at the same time a soft, hydrogen rich (low oxygen content) flame is moved slowly from the bottom to the top of the work. The tube, now sealed at one end, is now cleaned with acetone, alcohol, and distilled water and allowed to dry. When dry, the semiconductor and dopant are introduced into the tube and a small 1.5 inch long section of solid quartz rod, cleaned in the same way as the tubing, is inserted into the end of the quartz tube on top of the semiconductor material.

The quartz tubing containing the semiconductor is now attached to the vacuum system via a ball and socket joint; the ball joint being attached to the quartz tube by a short length of rubber vacuum hose. The end of the quartz rod adjacent to the semiconductor is placed four centimeters from the sealed end of the quartz tubing. The ampoule is opened to the vacuum system very slowly to avoid having any of the dopant material drawn off by the vacuum. After the pressure within the ampoule has reached a value about 0.01 microns, the ampoule is sealed by heating the quartz tube around the area of the rod with a hydrogen-oxygen flame. As the tubing (adjacent to the section of rod) softens, it is drawn into contact with the rod because of the pressure difference. By slowly rotating the work and moving the flame along the quartz tubing adjacent to the rod, the two are joined. To avoid punctures, care should be taken so as not to heat the quartz tubing at places other than those adjacent to the rod. The semiconductor is now ready to be remelted.

The ampoule containing the semiconductor and dopant is surrounded with a graphite sleeve and the sleeve and ampoule placed within the coil of an induction furnace. Some care must be exercised in the mounting of the graphite sleeve inside the coil; the two cannot touch, otherwise arcing and shorting of the coil results. The extra length of quartz tubing attached to the ampoule is useful for mounting the ampoule in the furnace coils.

The induction furnace power is turned on and adjusted for maximum power coupling into the graphite sleeve and semiconductor material. The temperature of the ampoule is monitored with an optical pyrometer focused on the graphite sleeve. For at least two minutes the temperature is maintained at 50 degrees centigrade above the melting point. The power is then shut off and the ampoule allowed to cool slowly within the graphite sleeve. Slow cooling increases the individual crystal grain size in the polycrystalline semiconductor that results. Figures 1-4 are photographs of Ge, GaAs, and GaSb material redoped by this process.

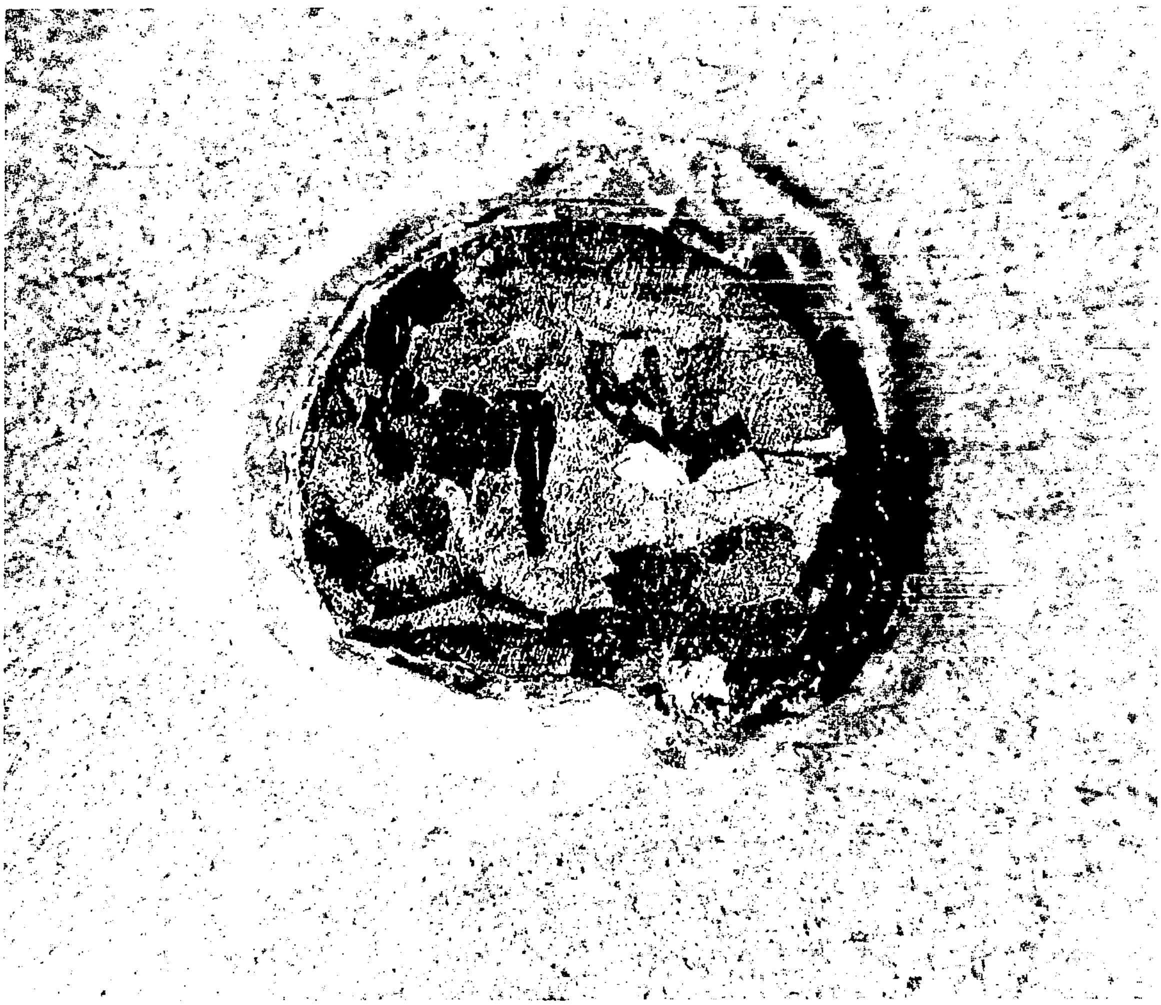


Figure 1. Germanium - Doped by Remelt; Sliced and Given Alkaline-Peroxide Etch

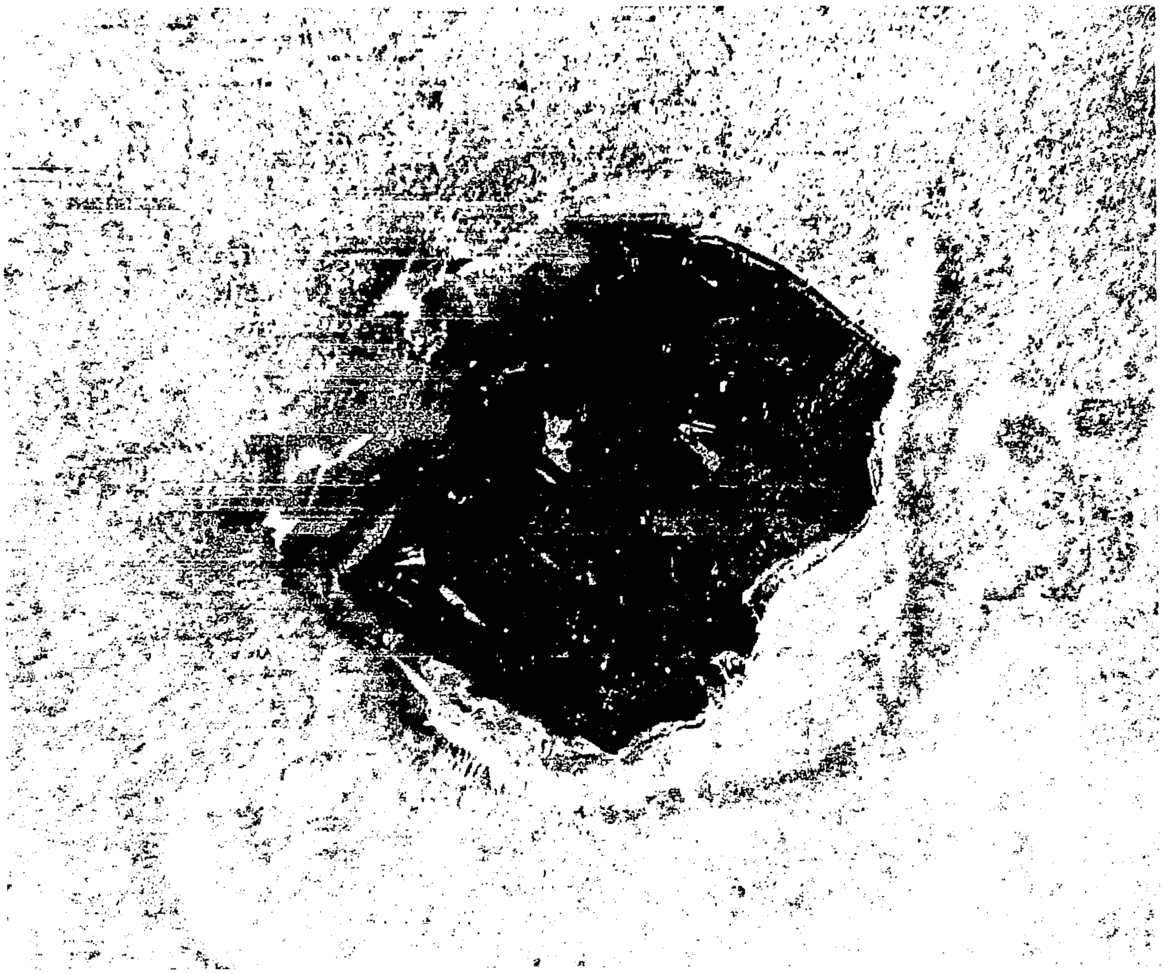


Figure 2. p-Type Gallium-Arsenide

Figure 3. n-Type Gallium-Arsenide

Figure 4. n-Type Gallium-Antimonide

IV. DEVICE FABRICATION

A. SEMICONDUCTOR PREPARATION

To begin the fabrication of any of the tunnel devices, the semiconductor appropriate to the particular task is selected. After the slug of material is prepared as described in Part III, it is sliced on the diamond saw into slices about 0.020" thick. Then, using the ultrasonic cutter, cylinders are cut from the slice. The cylinders are 0.020"in diameter and 0.020" thick. Wax is used to mount the semiconductor for slicing and then cutting with the ultrasonic cutter. All traces of this wax must be removed from the surface of the semiconductor if the subsequent plating processes are to be successful. The wax is removed by a minimum of three separate boilings in alcohol. The semiconductor surface is then given a final cleaning by boiling in 5% NaOH (at 95°C) for two minutes and then rinsing in clean, distilled water. The cylinders are not allowed to air dry, but are transferred immediately to the palladium chloride activator in which it remains for one minute at room temperature. The cylinders are again rinsed in distilled water and transferred directly to the electrodeless nickel plating solution. There they remain for two minutes. The temperature of the plating solution is held at 95°C. A recipe for an acceptable plating solution is as follows[11].

Nickel chloride ($NiCl_2 \cdot 6\,H_2O$)	30 g/ℓ
Sodium hypophosphite ($NaH_2PO_2 \cdot H_2O$)	10 "
Ammonium citrate $\left[(NH_4)_2\,HC_6H_5O_7\right]$	65 "
Ammonium chloride (NH_4Cl)	50 "

Filter

Add ammonium hydroxide (NH_4OH) until solution turns from green to blue. Temperature 90-100°C with addition of more NH_4OH as needed to maintain blue color.

B. DIODE ASSEMBLY

The diode assembly takes that form which was first proposed by this project in March 1963 to the NASA sponsors and is now incorporated and which we call the "pin" diode. The diode is fabricated on the end of a 40 mil diameter pin which is about 0.75" long. Epoxy is used to "set" the point after the junction has been formed. For mechanical support, the epoxied junction is surrounded by a ceramic housing. The required metalized ceramic cylinders (0.030" O.D. x 0.012" I.D. x 0.005 - 0.015" thick) have been fabricated to our specifications by the Metalized Ceramics Corporation, Providence, Rhode Island.

The complete structure of the diode is shown in Figure 5.

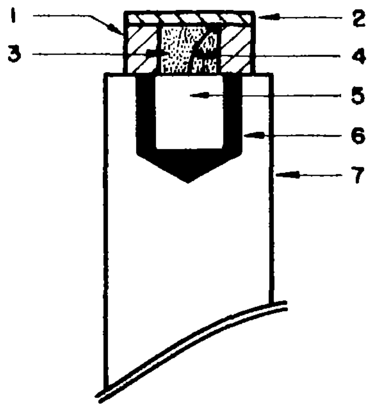


Figure 5. Experimental Structure of Pin Type Millimeter-Wave Diode and Package

In the figure there is identified the following parts.

1. Metalized ceramic (94% Al_2O_3) ring soldered to part 7
2. Metal cap - 1.0 mil thick copper or Hysol silver epoxy
3. Epoxy filler and junction stabilizer
4. Point contact - thin foil ribbon or fine wire
5. Semiconductor
6. Solder
7. Nickel pin

The nickel pins (Item 7 above) are made of Grade A nickel wire, B and S No. 18 (0.040" in diameter) cut to 0.875" lengths. This material is obtained from Diver-Harris Company, and is obtained in straightened 12" sections. The hole to receive the semiconductor cylinder is drilled on a Jeweler's Lathe using a No. 75 drill (0.021" diameter) and drilled 0.030" deep. The inside of the hole is tinned with 60/40 Sn-Pb using ammonium chloride flux. Other fluxes have been tried such as rosin flux, zinc chloride (Ruby flux), and H-200 hydrazine in alcohol from Fairmount Chemical Company, Inc., but the ammonium chloride seems to give the least difficulty in obtaining consistently satisfactory soldering of the semiconductor into the pin. Using the same flux, solder in the nickel plated cylinder of semiconductor. Be sure that the cylinder is seated as deeply as possible in the hole. This helps to minimize the amount of solder remaining in the pin after the soldering operation and helps in succeeding operations. Also it helps to eliminate any flux that might otherwise have a tendency to remain trapped below the cylinder.

The soldering jig shown in Figures 6a and 6b consists of a holder for the pin and a set of titanium contact fingers. These fingers grip the pin on opposite sides of and adjacent to the semiconductor cylinder. The heating occurrs locally by passing a 60 cps current between the fingers and hence through the nickel pin (and around the semiconductor). The spreading resistance at the contact between the titanium fingers and the nickel is high enough compared to the rest of the circuit so that sufficient power is dissipated at the contact points (I^2R power) to easily raise the temperature of the pin tip to soldering temperature. A variac is used to control the current and hence the resulting temperature.

After the tinning operation and again after the cylinder soldering, the flux can be removed and the parts in general cleaned by immersion for a short time first in clean alcohol, ultrasonically agitated, and then in clean acetone, ultrasonically agitated. A good description of the available solvents and wash liquids is contained in Reference 12.

The next step in the diode fabrication process is to grind the end with the semiconductor in such a manner that the end surface is

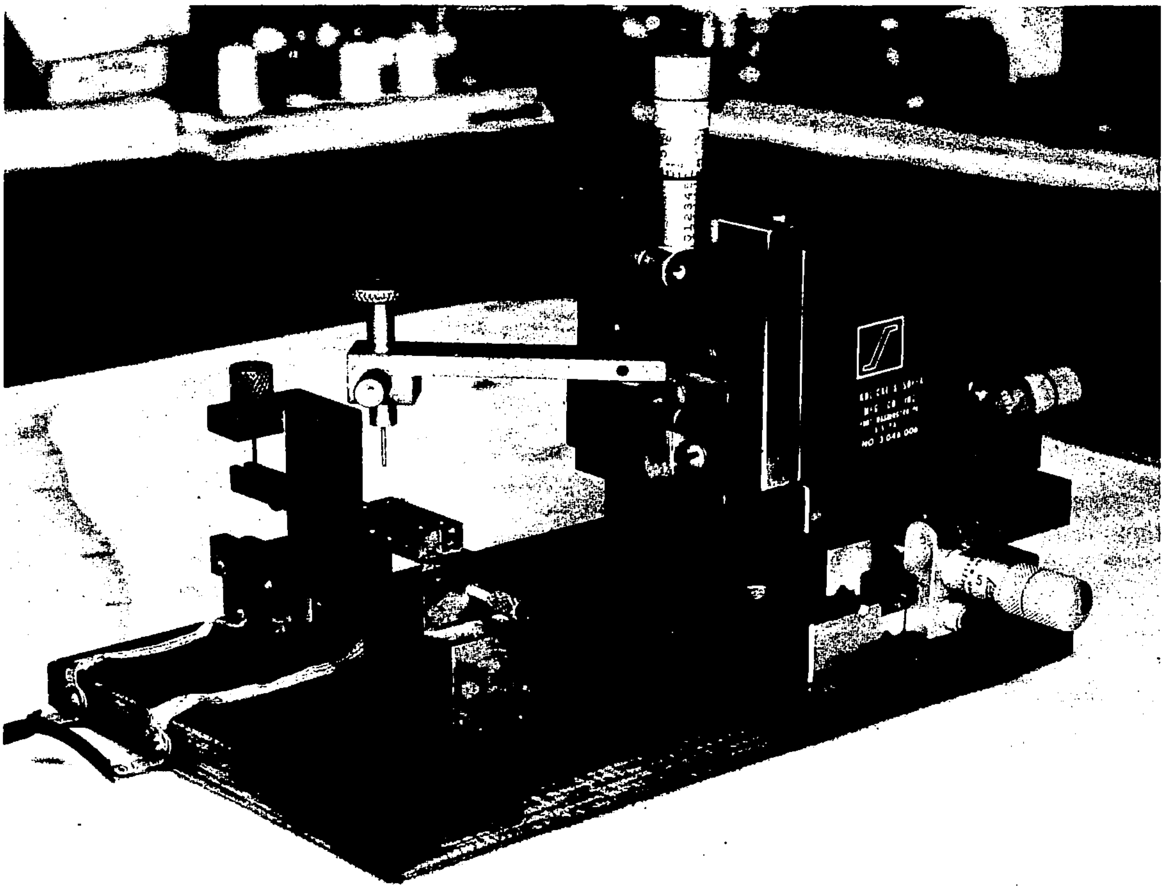


Figure 6a. Soldering Jig

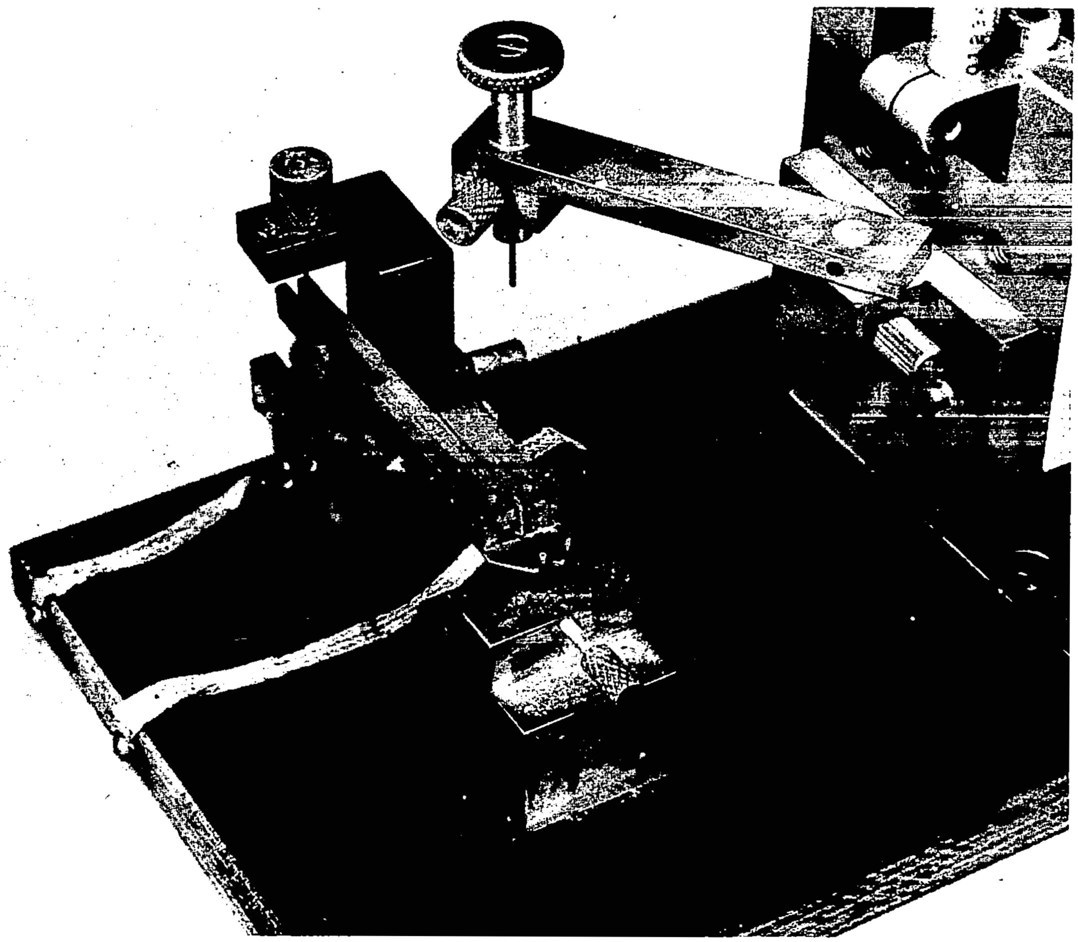


Figure 6b. Soldering Jig with Pin in Place

perfectly smooth, the end nickel, the annulus of solder, and the semiconductor surface are all coplanar and this plane is perfectly perpendicular to the pin axis. Figures 7a and 7b show the diode holder and other apparatus used to grind and polish smooth the ends of the pin and the semiconductor. Number 600A Rubwet silicon carbide paper made by Armour is used (dry) for the general grinding. A piece of hard finished (smooth) cardboard is used for the final polish. A photograph of this surface, after the final polish, is shown in Figure 8, and after etching is shown in Figure 9.

The next step of the process is the mounting of the metallized ceramic ring (Part 1 of Figure 5). The same soldering jig as shown in Figures 6a and 6b is used.

A very small dot of flux (NH_4Cl) is placed on the end of the pin which is mounted in the pin holder of the soldering jig. With fine tweezers, the ceramic ring of the appropriate thickness is placed on the pin end and centered over the semiconductor cylinder. The ceramic rings should be tinned with 60/40 Sn-Pb solder prior to this operation. The pressure foot of the solder jig is lowered into place by adjustment of the 3-d micropositioner (see Figures 10a and 10b). The vertical control should be advanced in such a manner to insure that a slight but positive pressure will be exerted, vertically, throughout the soldering operation. The soldering cycle, which can be controlled by a foot switch, should be timed so that heat will be applied for no longer than three seconds. If the heating current has been properly adjusted, the ceramic ring well tined, and fresh flux is used, the solder from around the semiconductor (Part 6, Figure 5) will flow up and join with that on the ring and then flow out across the tin surface, and 1-2 seconds should be all the time that is required. Experience has shown that when more time is taken to complete this operation, difficulty with the succeeding steps will be encountered. This step of the process is followed by thorough cleaning first in water, then alcohol followed by a dip in acetone; each dip being ultrasonically agitated.

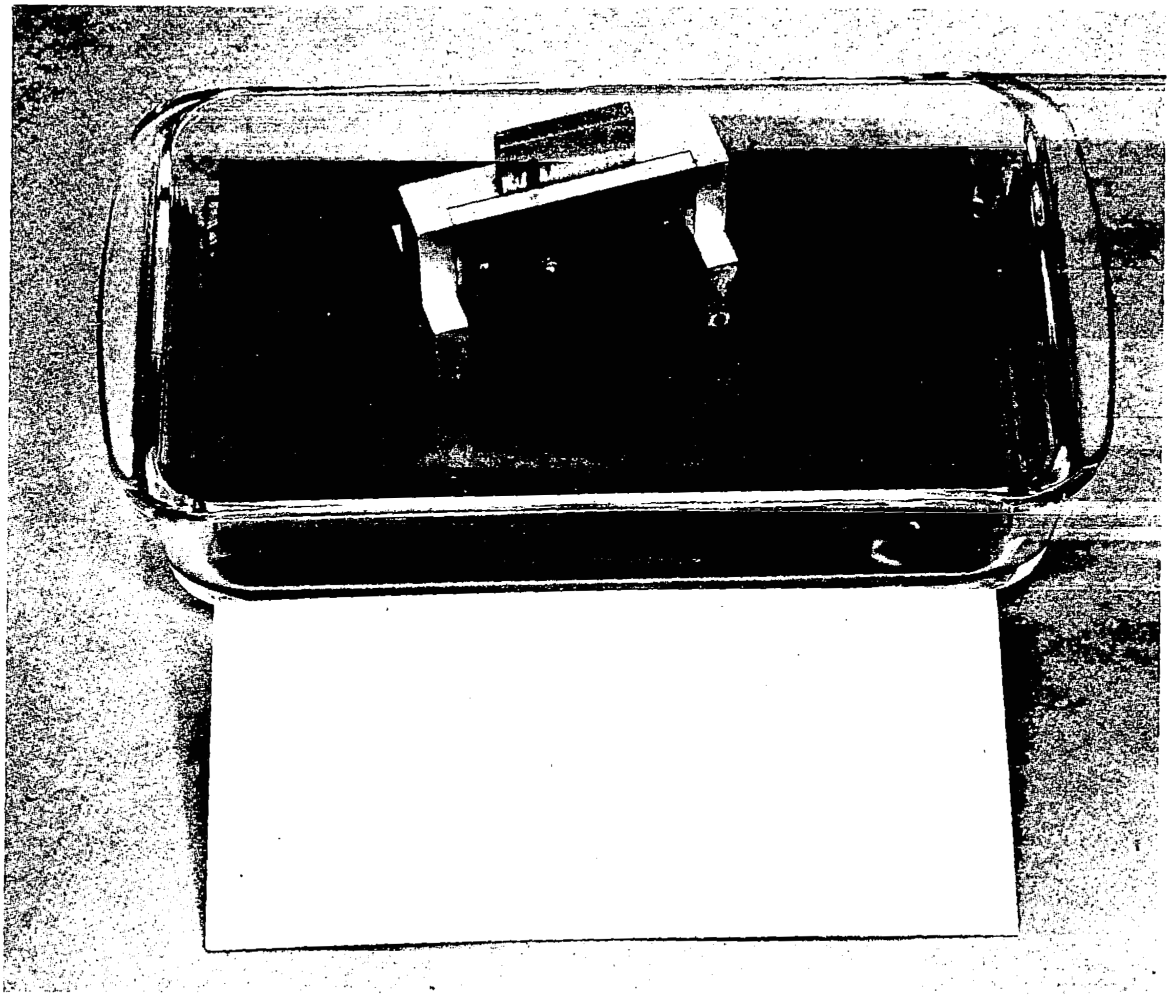


Figure 7a. Apparatus for Grinding and Polishing the Pin Mounted Semiconductor

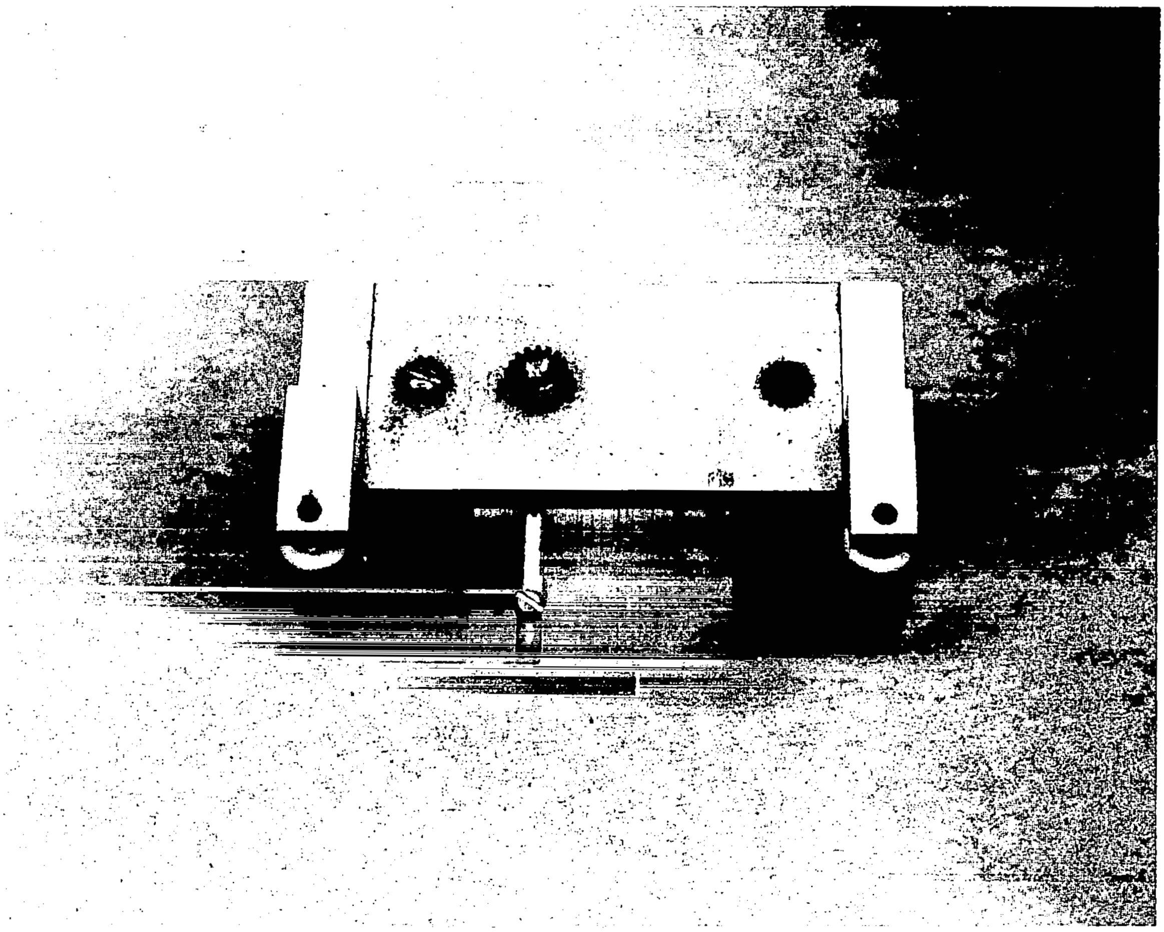


Figure 7b. Diode Holder for Grinding and Polishing

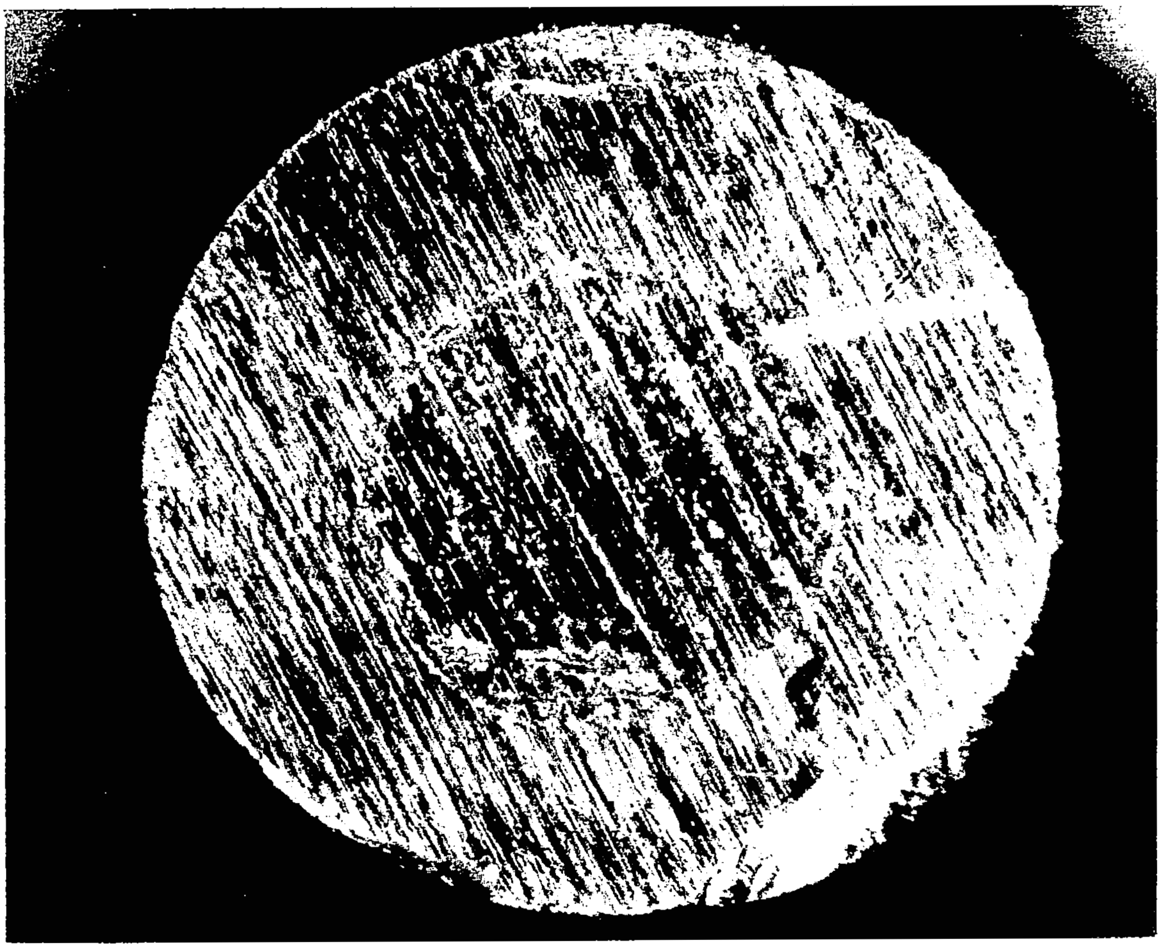


Figure 8. Semiconductor in Nickel Pin, After Grinding but Before Etching

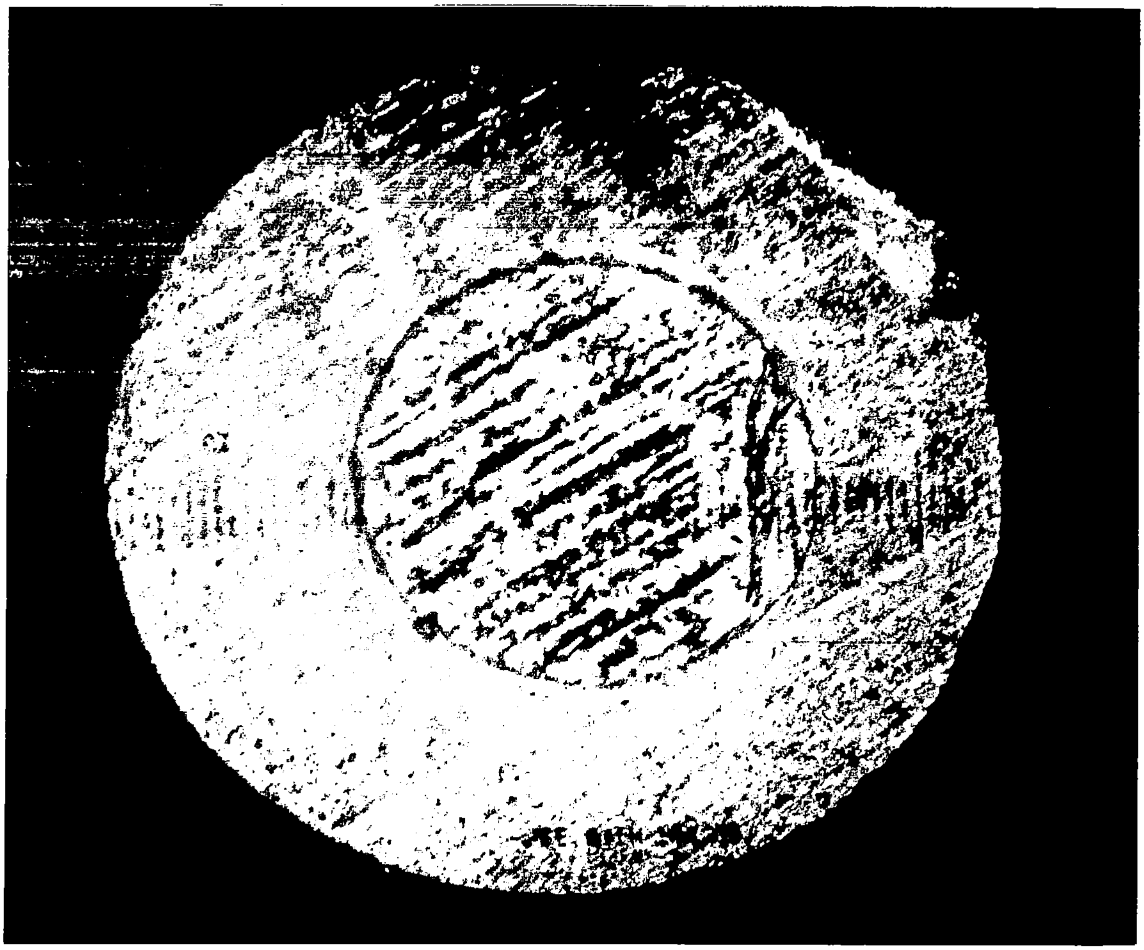


Figure 9. Same as Figure 8 but After Etching

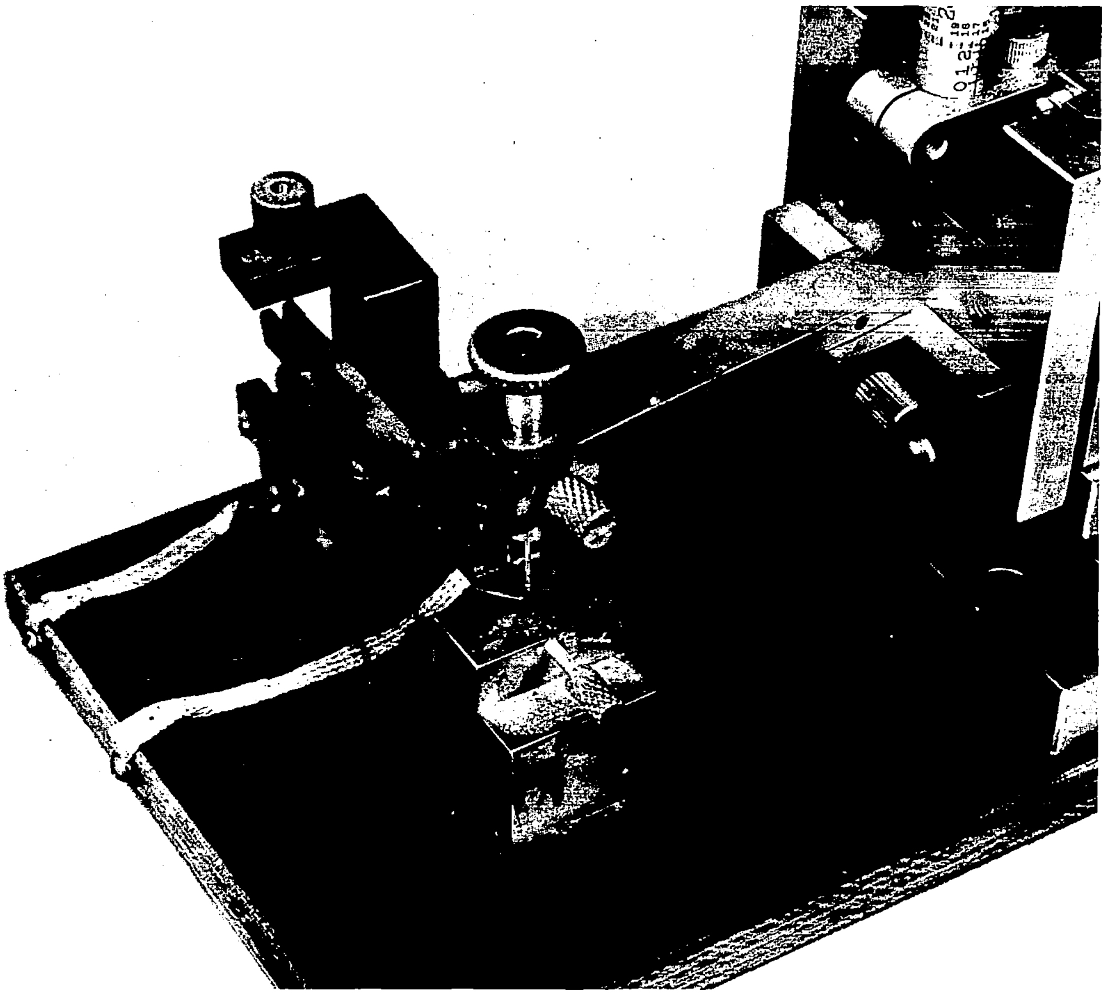


Figure 10a. Soldering Jig with Pressure Foot in Place over Ceramic Ring

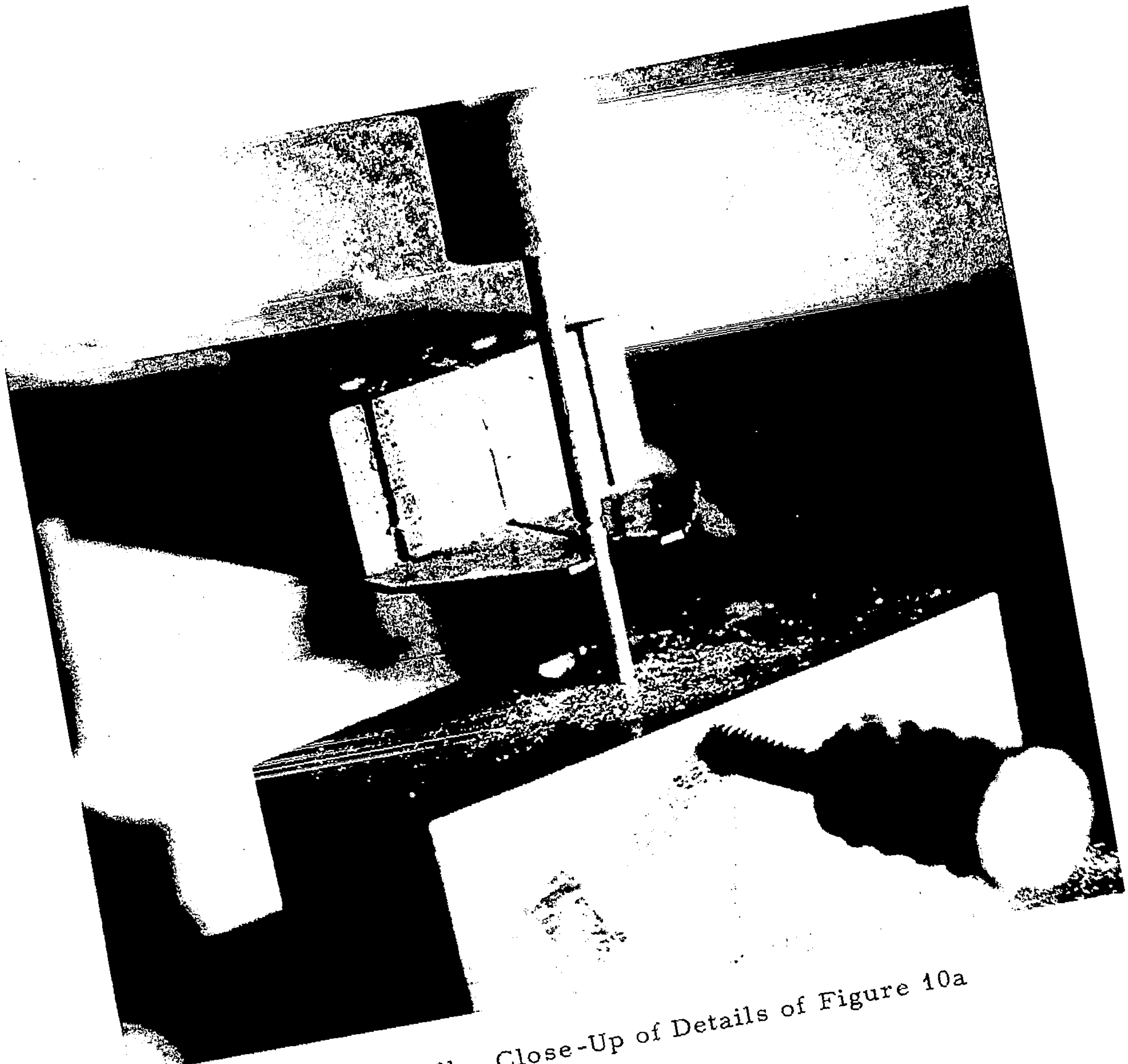


Figure 10b. Close-Up of Details of Figure 10a

Semiconductor Etching

Once the ceramic ring has been soldered in place, the semiconductor surface must be etched to remove 1-2 mils of material. This removes from the surface any contaminants placed there during the previous steps and also removes the minute fractures and other grinding and polishing damage usually found on the semiconductor surface to a depth of from 0.5 to 1.5 mils[13]. There is a tremendous variety of mixtures and combinations of chemicals that are used for either chemical or electrolytic etching. The particular combinations employed are usually determined by the basic material to be etched (Germanium, GaAs, GaSb, etc.). But most chemical etches will consist of one or more components which act as surface oxidant for the semiconductor and another component such as hydrofluric acid or sodium hydroxide which dissolves the oxide as it is formed. Additional substances are often included to control the rate at which the material is removed or the physical nature of the resulting surface.

The following is a listing of recipes for chemical etches (CE) found useful by this project.

CE No. 1 - $1\ HF + 3\ HNO_3$

CE No. 2 - $50\ HNO_3 + 30\ HC_2H_3O_2 + 15\ HF$

CE No. 3 - $1\ HF + 3\ HNO_3 + 4\ H_2O$

CE No. 4 - $5\ HNO_3 + 3\ HF + 3\ HC_2H_3O_2 + Br$ (10 drops per 50 cc)

CE No. 5 - $3\ H_2O_2 + 10\ NaOH$ Hot

CE No. 6 - $1\ HNO_3 + 1\ H_2O_2 + 1\ HF + 4\ H_2O$

For the above listing all of the acids and the peroxide are concentrated solutions, that is, HF - 48%, HNO_3 - 70%, $HC_2H_3O_2$ - 100%, and H_2O_2 - 30%. The sodium hydroxide is a 5% solution.

CE-4 is the "chemical polish - CP4" recommended in Reference 10 for etching germanium. CE-6 is similar to the "superoxal" etch recommended in Reference 10, but with HNO_3 added as a further oxidant. This CE-6 has been found to give very satisfactory etches of the surface of germanium and silicon. In particular, it appears to be a good preferential etch on the germanium. CE-1, 3 are used primarily with GaAs; CE-3 etching slower than CE-1. CE-2 (Reference 14) is also very good for germanium and GaSb. The acetic acid serves as a moderator, slowing down the reaction and making it more controllable.

As CE-1, 2, 3, 4, and 6 are all of strong acids, volatile, and highly corrosive, obvious precautions must be taken in its mixing, storing, and use. As all of these noted etches are strongly reactive with both the nickel used for the pins, and the solder that is used to attach the semiconductor, care must be taken to insure that the semiconductor is not contaminated during the etching process.

To etch the semiconductor after it is mounted in the pin, CE-1, 2, and 6 are sufficiently fast and violent to insure that the nickel or lead and tin from the solder cannot deposit back on the semiconductor surface and cause a contamination problem, only if the etching is done before the ceramic ring is soldered in place. That is, if it is desired to test the semiconductor for junction quality by forming several diodes at various spots on the surface before affixing the ceramic ring, then these chemical acid etches may be used. However, it has been found that, regardless of how good the surface is, soldering in place the ceramic ring will cause some contamination of the semiconductor surface which results in degradation of the final junction. Therefore, after the ceramic ring is in place, CE-5 is used, either to "clean up" a previously etched surface, or to give the initial etch. A clean up etch usually takes about 30 seconds to one minute in the etch. An initial etch usually takes about 2 minutes. This etch has been proven useful for germanium, GaAs, and GaSb. On GaAs this etch is preferential; that is, it will yield a dark matte or a silvery matte finish depending upon the crystal orientation of the surface being etched.

After the final etch with CE-5, rinse with clean H_2O, alcohol and finally acetone, each one ultrasonically agitated.

The pin is now placed in the junction forming jig. The forming jig, with pin in place, is shown in Figures 11a and 11b. A "catwhisker" of the appropriate material is formed and placed in the jig. The catwhisker is made of fine wire (diameter < 1 mil) or very thin metal foil (thickness ≤ 0.5 mil). The shape of this metal contact has a tremendous effect on the amount of pressure that will exist at the contact point between the wire and the semiconductor. By far, the primary difficulty encountered in making acceptable point contact tunnel devices, is in maintaining the pressure light enough so that when the electrical pulse is applied to the junction, the melting and subsequent freezing of the material in the vicinity of the contact can take place without movement of the contact wire with the obvious disruption of the melting and recrystallizing process. Figure 12 shows the shape and dimensions that have been experimentally determined to yield the most satisfactorily consistent results. The dimensions are particularly applicable for wire points of indium-gallium and tin. For stiffer wires such as zinc, aluminum, and copper, these dimensions may be doubled.

The wire point on the brass pin is held on a movable arm on the Forming Jig. Using the three-dimensional positioning adjustments, the wire is lowered through the ceramic ring until a contact with the semiconductor surface is made. This contact must be an absolutely light, grazing contact; the contact pressure must be so small that when a current pulse is passed through the contact and the materials in the vicinity of the contact point are melted, no movement of the wire point above the semiconductor may result. This point cannot be emphasized too strongly. The success or failure of the operator at making point contact tunnel diodes rests in his ability to maintain his instruments and his "touch" sufficiently sensitive that the light pressure may consistently be obtained. Failure to make contact through the wire to the semiconductor generally indicates either a contaminated semiconductor surface or an unclean wire point.

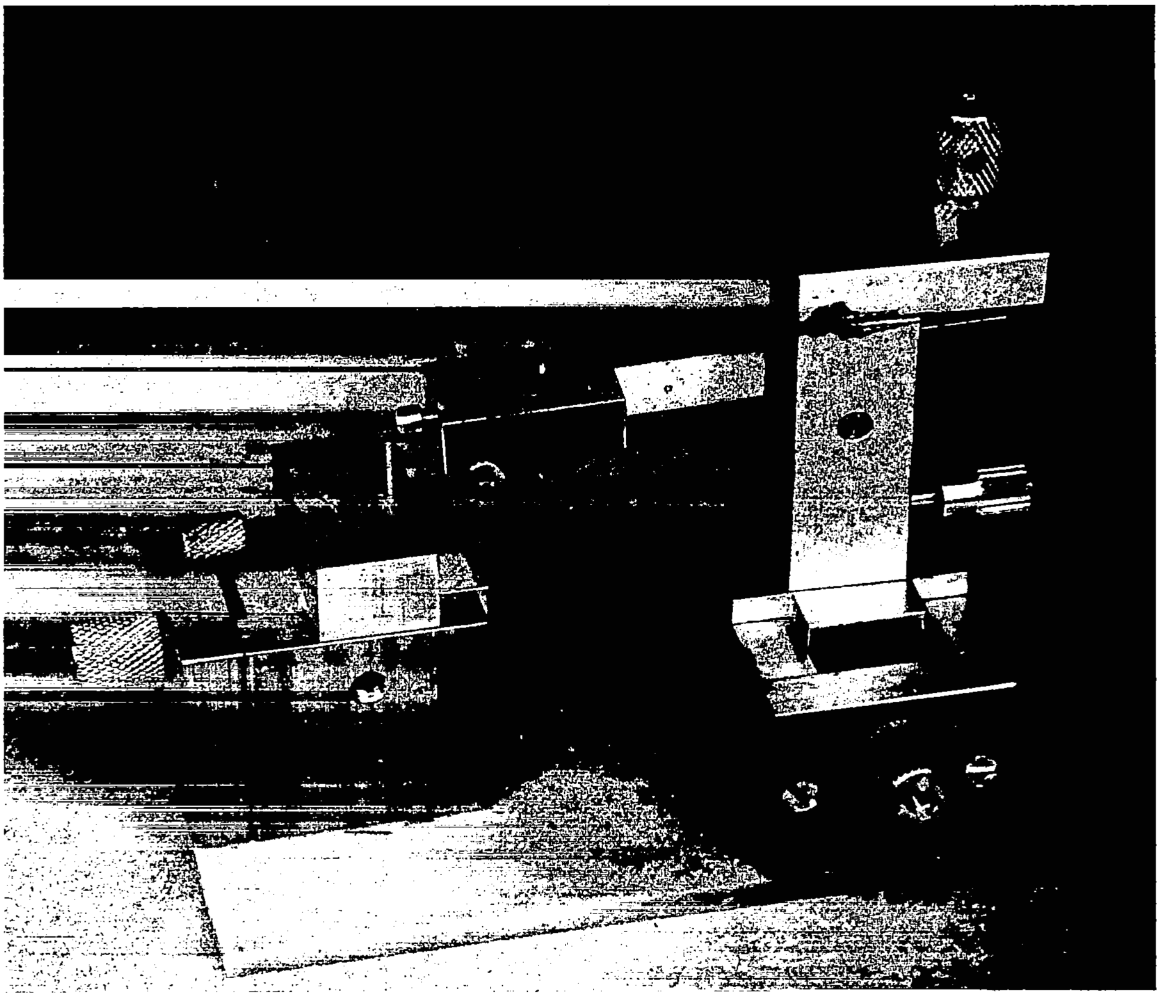


Figure 11a. The Forming Jig with Pin in Place

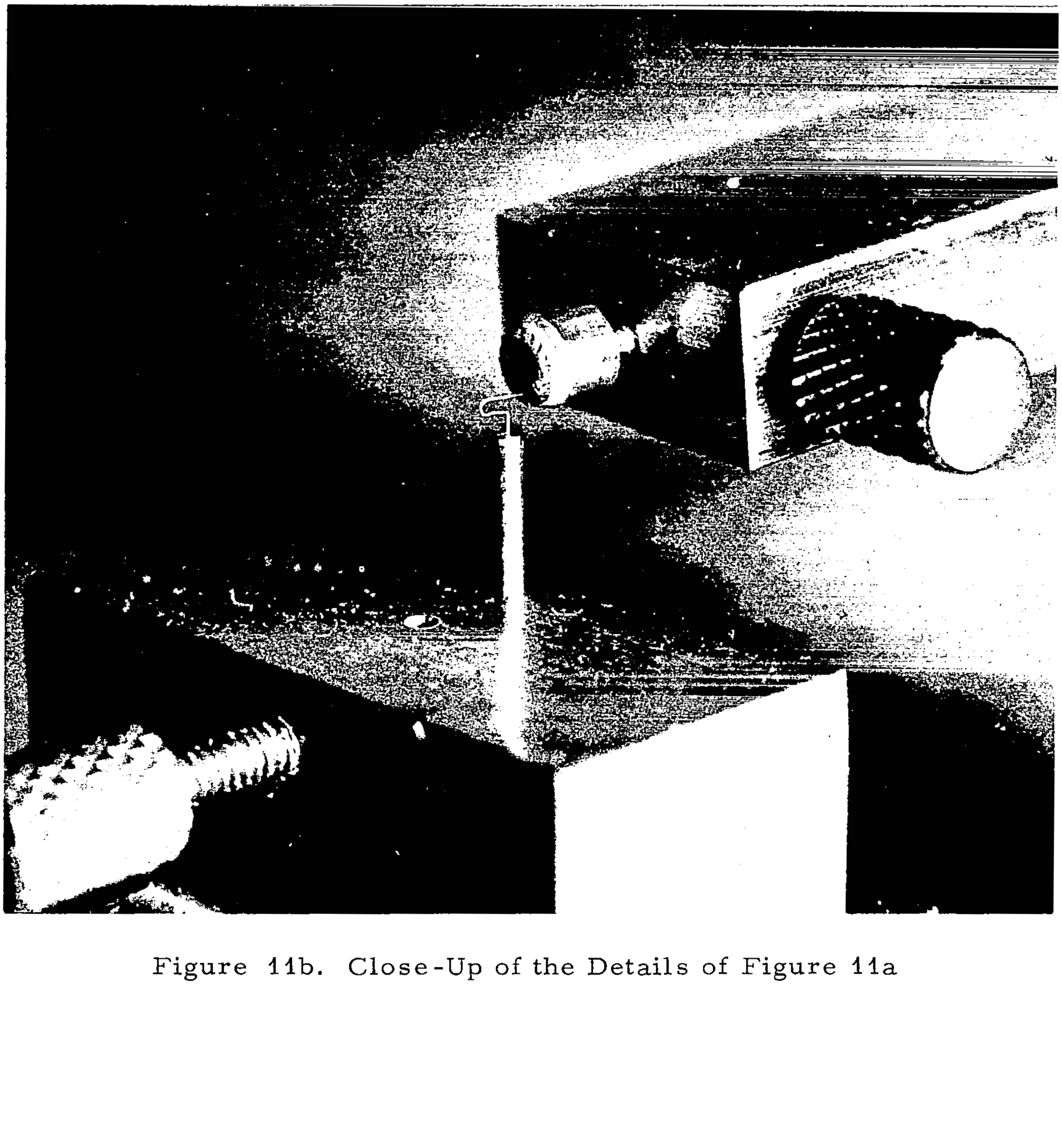


Figure 11b. Close-Up of the Details of Figure 11a

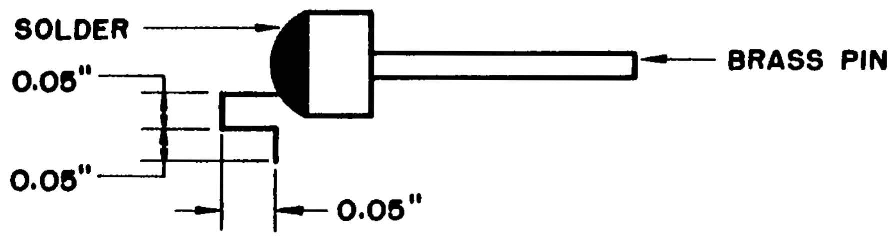


Figure 12. Wire Point "Catwhisker" to be Used in Junction Forming Jig

After contact is made, the junction is formed by passing an electrical current pulse through the metal wire to semiconductor contact point. The methods and instruments for the observation of the contact and the junction forming will be discussed in detail in Part V of this report.

Junction Potting - Epoxy

The wires or foil that are used in making these point contact tunnel diodes are usually so soft that they are very difficult to handle. This is true especially when they are compared with the phosphor-bronze or tungsten wires generally used for the more conventional microwave point contact diodes. To overcome the inherent fragility in the assembly of these devices, an epoxy compound is used to completely fill the space around the wire point and above the semiconductor, and in particular in the immediate vicinity of the junction formed at the point of contact between the wire and the semiconductor. This non-conducting epoxy not only ruggedizes the physical structure but also passivates the semiconductor surface, in particular around the junction, in that the surface and junction are sealed off from the atmosphere and hence from any contaminants that might possibly degrade the electrical properties of the junction.

Examination of Figure 5 may lead one to believe that the application of the epoxy to the junction could prove to be a difficult operation. But it has been found that a steady hand and the use of a fine pointer, such as the use of a small anchor drill held in a pin vise, can apply the epoxy in small enough quantities so that the wire point will not be disturbed. The operation actually proceeds by placing a small dropy of epoxy on the upper surface of the ceramic ring. The curvature of the ceramic ring, and the slight skin tension of the epoxy combine so as to tend the flow of the epoxy off the ring and into the hole rather than down the outside. The whole operation, of course, is done under the microscope as shown in the over-all system set-up in Figure 13.

The Araldite product of Ciba Products Company has given the best results as a potting compound to stabilize or "set" the point contact wire of the diodes being fabricated. Many combinations of epoxy resins and hardeners have been tried. The following combinations (Reference 15) gave better than the average results.

1.	Epon 815 Versamid 140	70 parts 30 parts	50 parts 50 parts
2.	Epon 828 Versamid 140	70 parts 30 parts	
3.	Epon 871 Epon 828 AEP	70 parts 30 parts 14 parts	
4.	Hysol C8-4148 Hardener H2-3475	40 parts 40 parts	
5.	Araldite 502 Hardener 951	40 parts 10 parts	

The Versamid 140 is a polyamide resin produced by General Mills, Inc. The Epon resin is a Shell product. The Epon 815 is equivalent to Epon 828 plus phenyl glycidyl ether in the concentration of one part per 40 parts resin. The AEP is a polyamine curing agent yielding a low mix viscosity. AEP is N-amino-ethylpiperizine and is also available from Shell. All the combinations should yield a low viscosity mix with good resilience. The No. 2 combination is less resilient than the other two, a little harder and a better adhesive. The No. 5 combination has good resilience and is also a good adhesive; further, it has the very low viscosity needed. Hence, the final selection was the Araldite 502 modified epoxy with the 951 Hardener at a hardener ratio of 25 phr. The resin to hardener is mixed 4:1 and cured according to the schedule: 1/2 hour room temperature gel; 1/2 hour at 70°C. The hardener ratio 25 phr used, rather than the manufacturer

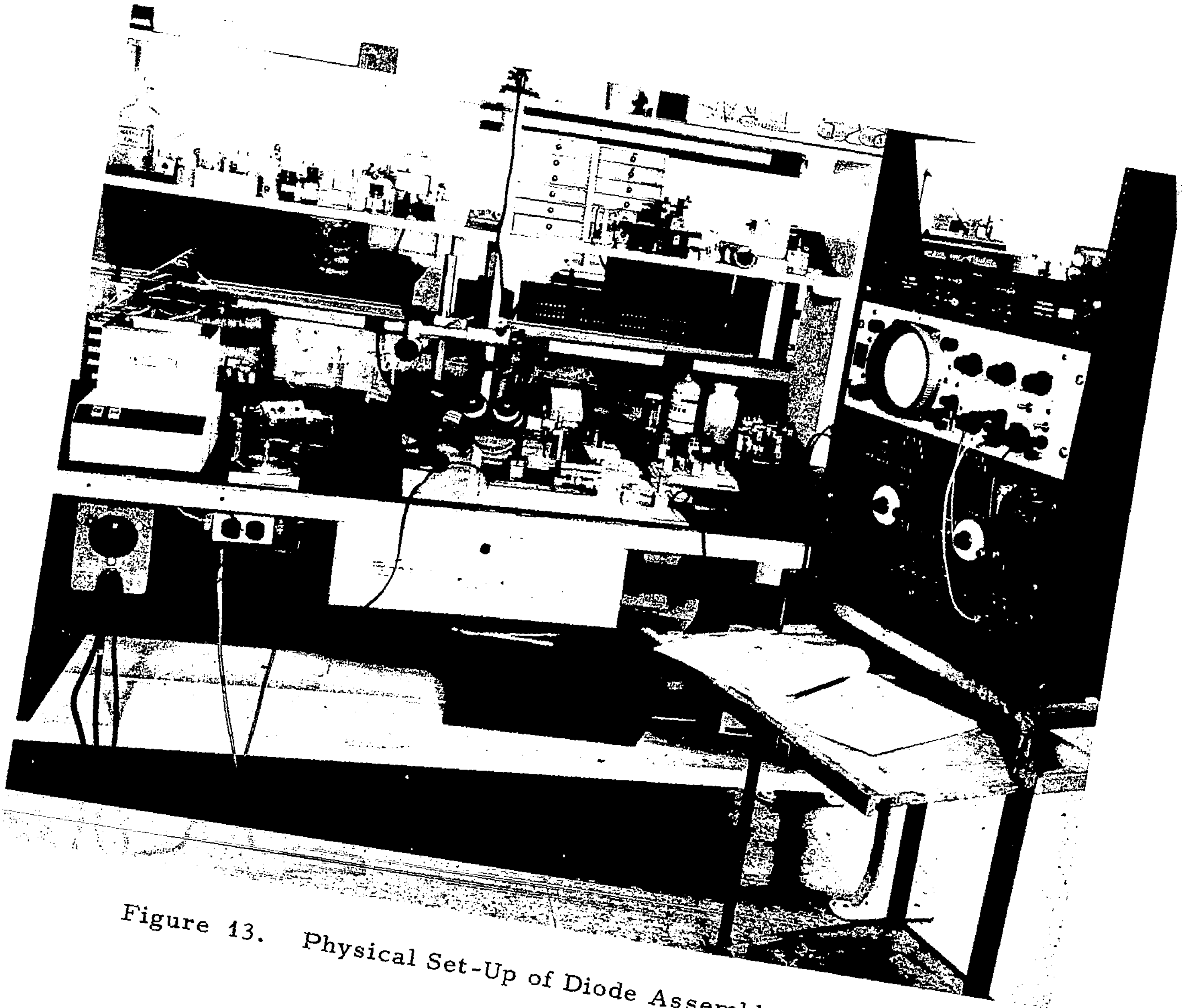


Figure 13. Physical Set-Up of Diode Assembly Apparatus

recommended 10 phr, was found necessary when trying to cure the compound in the small quantities actually being used. The volume of the cavity being filled is approximately 2×10^{-6} cubic inches.

The diode is completed by the application of a highly conductive silver epoxy in a sufficiently thin layer to bond, without introducing excess material, the diode contact wire to the ceramic and at the same time bond on to the diode upper surface, the 0.001" copper cap. The silver epoxy actually performs the operation of capping the diode, but in the small volume being here considered, the silver epoxy appears almost granular. The copper cap acts as a broad area contact between the epoxy and the circuit, and also acts as a "skin" to minimize the tendency of the particles of the epoxy to flake off. The adherence between the ceramic surface, the epoxy and the copper cap all appears to be satisfactory. The silver epoxy cement used is a highly conductive epoxy putty produced by Hysol, type K8-4238, used with the Hardener, Hysol type H2-3475. Figure 14 is a more detailed sketch showing the use of the epoxy.

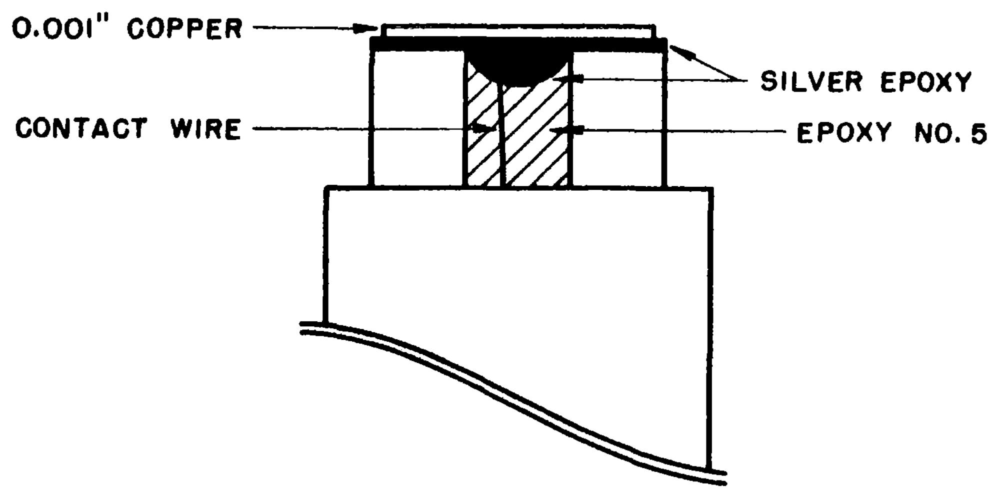


Figure 14. Details of Epoxy Use in Tunnel Diode Package

V. JUNCTION FORMING

A. GENERAL

The junctions employed in the tunnel devices are extremely small area alloyed junctions. The heat for alloying is generated by passing an electrical current through the wire to semiconductor contact; the heat is generated by the I^2R losses in the vicinity of the contact. The heating cycle needed for proper alloying of the different metal wire and semiconductor combinations varies drastically.

Forming techniques used for diodes have for the most part, been derived by trial and error. There have been two fairly rigorous attempts at analyzing the problem. One by Sim[16] and one by Lory[17] which was done on this project. Sim solved the Maxwell's equations and the heat flow (with source) equation, making a number of assumptions primarily valid for a diode of the type commonly used for detectors, harmonic generators, and the like. He assumed that the semiconductor material was nearly intrinsic, that the point material was more refractory than the semiconductor and that thermal impurity states (presumably due to indiffused copper atoms or lattice dislocations) are the primary cause of the formation of a p-n junction in the neighborhood of the contact.

None of these assumptions are entirely valid in the present case. With the heavily doped materials used in tunnel diodes, the extrinsic conductivity cannot be ignored, at least at the lower temperatures, and the point materials, more often than not, melt at temperatures low compared to semiconductor melting points. The p-n junction is formed by indiffusion of dopants contained in the wire; these dopants ordinarily being Column III and V elements in the case of the elemental semiconductors and Column II-VI elements in the case of the III-V semiconductor compounds. Sim came to several conclusions as a result of his analysis, one of which was the desirability of a high impedance pulse supply as a source of the electrical overload in forming the junction. Burrus and Trambarulo[18] as well as the workers of this project have, on the other hand, found that even for relatively low-current units, a pulse supply having a low, but accurately controlled impedance is to be preferred.

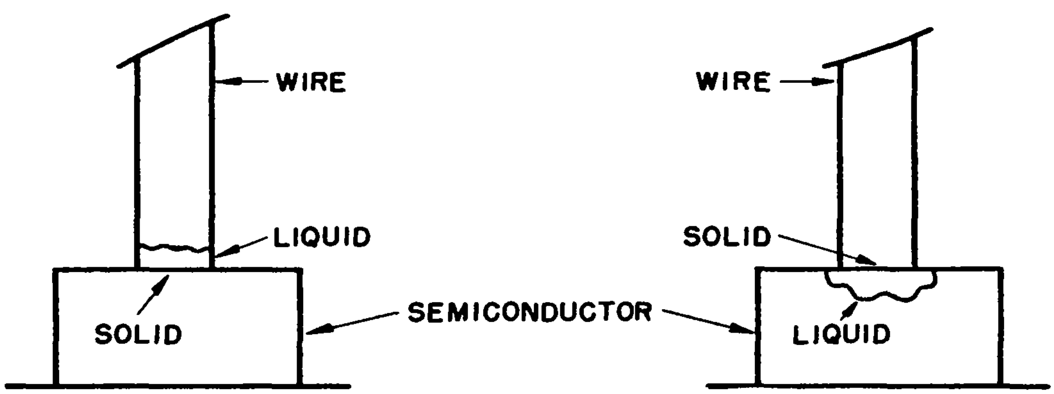
The range of voltage normally used in forming the diodes is such that junction effects may reasonably be expected to play a part in limiting the forming current. There exists a critical forward pulse voltage, below which the parameters of the diode undergo no change; this obviously must be the case if the diode is to function as a diode. Lory[17] has calculated this critical voltage and experimentally verified the value of 1.2 volts for a germanium diode with aluminum wire. Again using Lory's work it can be shown that the critical voltage is only about 0.87 volts for a germanium diode with an indium-gallium wire. The calculations of the critical voltage have not been made for the III-V semiconductor compounds. Lory also calculated the thermal time constant of the Ge-Al combination to be about 8 μsec. This means, essentially, that if a pulse is applied to a contact and the pulse is of sufficient height and with proper source impedance to form a good diode, but with a pulse duration which is close to the thermal time constant, then it is expected that whatever I-V characteristic is obtained for the resulting diode, a second pulse of the same length will alter it. If, however, a pulse is applied which is long compared to the thermal time constant, the resulting I-V characteristic will remain relatively unchanged with succeeding pulses, presuming that no factor such as excess contact pressure exists to disturb the junction as initially formed.

It has been experimentally determined that the thermal time constant for the Ge-In/Ga combination must be equal to or less than 3 μsec, by the above definition. This also appears to apply to both the n-type and p-type GaAs combinations.

The information on the time constants is relatively important but the value of the critical voltage is only important when the forming is to take place by forward pulsing. Of the four materials used (n-Ge, n-GaAs, p-GaAs, n-GaSb) only two are successfully formed by forward pulsing. It has been determine experimentally that reverse pulsing is necessary in some instances if high valley current with the resulting low peak current to valley current ratio is to be avoided. The following has been found to hold:

1. n-type Ge with Al point requires reverse forming
2. n-type Ge with In/Ga point requires reverse forming
3. p-type GaAs with Sn point requires forward forming
4. n-type GaSb with Cu point requires forward forming
5. n-type GaAs with Zn point requires reverse forming

In each case it can be shown that the electron current of the forming pulse flows across the contact surface, on the surface considered the interface between the solid and melted material, from the liquid to the solid side. See the following sketch (Figure 15).



(a) This Sketch Applies to Nos. 1, 2, 3, and 5 Semiconductor Combinations

(b) This Sketch Applies to No. 4 Semiconductor Combination

Figure 15.

On the basis of I^2R heating alone, if a very large voltage and high pulse impedance is used such that the source is a current source, then the direction of current flow should make no difference in the resulting alloying. But there does exist a very large electric field at the position of the liquid-solid interface. Now if there exists positive ions of the metal or semiconductor (whichever is molten) at the interface, under the stress of the high electric field these ions may migrate through the interface and set up deep lying states within the p-n junction region which is formed when the material cools and epitaxial regrowth occurs. These deep states could then give rise to an increased tunnel current at higher forward bias voltages than would be otherwise experienced[19-20].

Further analytical investigation has not been performed on the subject of forming of the junctions by electrical overload. Rather, using the above information as a basis, the following procedures have evolved.

B. n-TYPE GERMANIUM

Suitable points for use with germanium are made of 1% Ga in Indium as a carrier. The wire diameter is equal to or less than 1 mil. Indium, tin, and other soft metals such as this can be made very simply by the Taylor process[21]. To make the wires by the Taylor process, the metal is melted (preferably in an inert atmosphere) and sucked up into a thin wall capillary tube of soft (lime) glass. The tube containing the metal as a core is heated over a short length by surrounding the glass by a few turns of tungsten wire through which enough current is passed to effect the necessary heating. As the glass softens, one end can be slowly drawn out. The result will be a fine filament of glass with metal core. The diameter of the metal wires produced in the composite drawn fibers is controlled by the temperature produced by the heater coils and the rate at which the fiber is drawn. The glass is removed from the composite fibers with hydroflouric acid which dissolves the soft glass readily but scarcely corrodes or etches the metal. The HF acid is diluted with a little water to suppress fuming.

With the proper wire mounted and bent as shown in Figure 12, a very light contact is made between the freshly clipped (pointed) wire and a recently etched semiconductor surface. The establishment of a very light contact is indicated by the small conduction shown when the I-V curve is viewed on an oscilloscope. The forming and display circuits are shown in Figure 16. The initial contact is made through a 10 k series resistor. After the contact is made, the 60 cps sweep voltage is increased to slightly a. c. form the curve; the 10 k resistor is swtiched out and the resulting forward characteristic should be similar to that of Figure 17a. The 60 cps sweep is then reduced to zero and the selector switch is placed in the -Pulse position. The junction is reverse pulsed, one pulse at a time, starting with a pulse amplitude of 3 volts. The pulse width is held constant at 3 μsec and the pulse z is started at 51 ohms. Figure 17b shows the result of one pulse at 3.0 v. Increasing the pulse in increments $\Delta v = 1$ volt, the fourth pulse, at 6.0 volts, yields the curve of Figure 17c. Next a succession of pulses yielded the results as shown in Figures 17d-17f. The pulsing sequence was to start pulse z at 51 ohms and

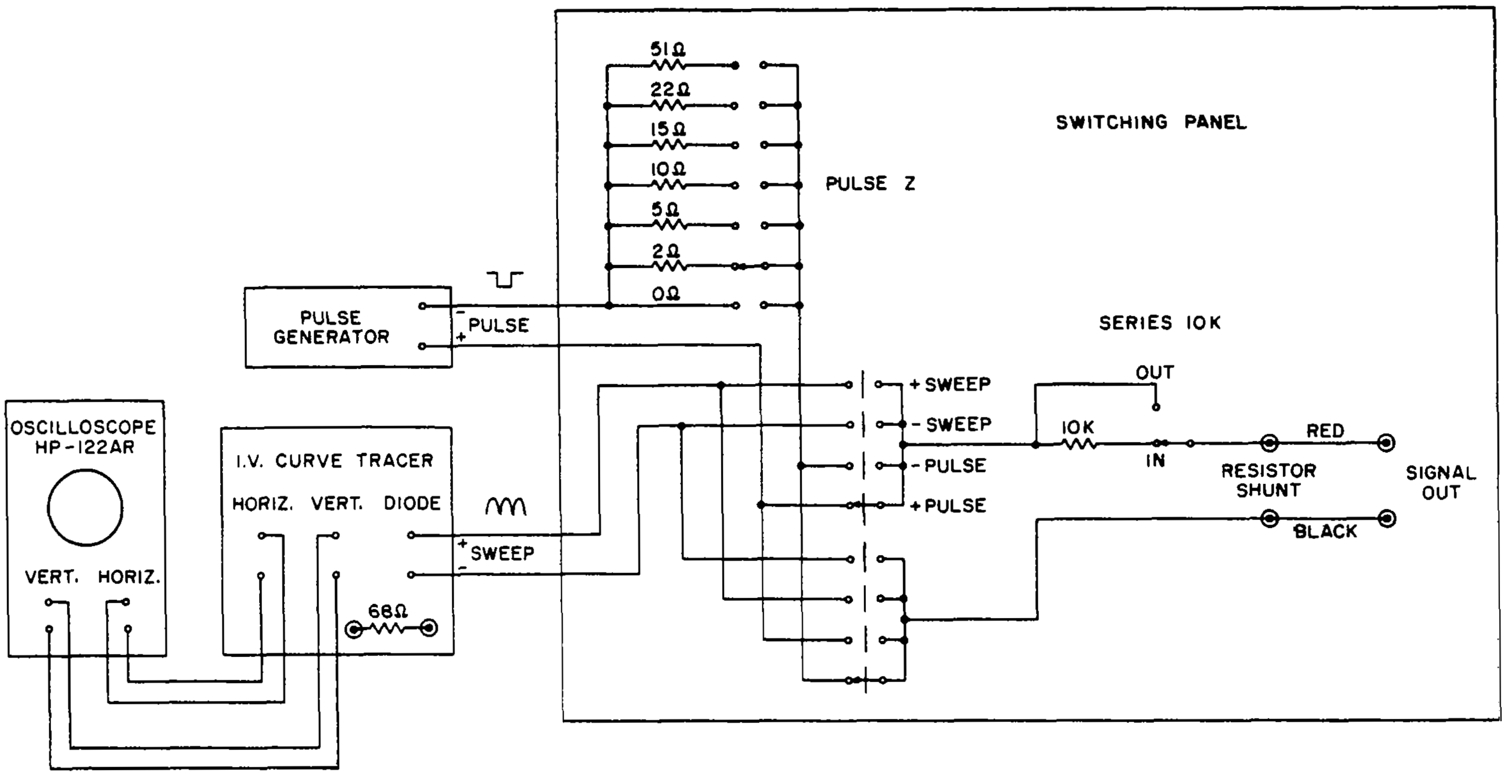
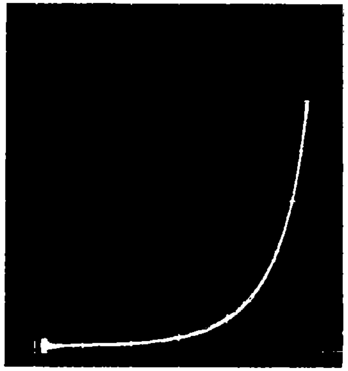
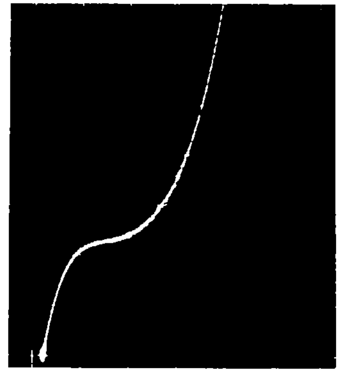


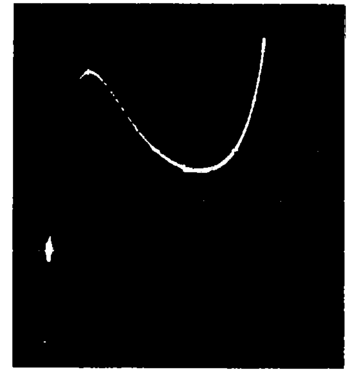
Figure 16. Forming and Display Circuits



(a) Germanium - no pulse
Vert.Sens. 10 μa/cm; Horiz. Sens. 0.1 v/cm



(b) Germanium - 1 pulse to 3 v, z = 51 Ω V.S. 10 μa/cm; H.S. 0.1 v/cm



(c) Germanium - 4 pulses to 6 v, z = 51 Ω V.S. 10 μa/cm; H.S. 0.1 v/cm

(d) Germanium - 12 pulses to 9 v, z = 22 Ω V.S. 100 μa/cm; H.S. 0.1 v/cm

(e) Germanium - 13 pulses to 10 v, z = 22 Ω V.S. 100 μa/cm; H.S. 0.1 v/cm

(f) Germanium - 21 pulses to 8 v, z = 10 Ω V.S. 100 μa/cm; H.S. 0.1 v/cm

Figure 17.

pulse amplitude at 3.0 v. Increase pulse amplitude in $\Delta v = 1.0$ volt until pulse amplitude reaches 10.0 volts. Then swtich pulse z to 22 ohms and start pulse amplitude at 6.0 volts again increasing this, in $\Delta v = 1.0$ v, to 10.0 volts. If higher current units are required, the process is repeated, stepping pulse z down and repeating pulse amplitude from 6.0 to 10.0 volts.

C. n-TYPE GALLIUM-ARSENIDE

The procedure for forming the n-type GaAs diodes is quite similar to that used for the n-Ge. The point material is zinc, either wire or foil. Zinc wire of about 3 mil diameter is commercially available. These wires can then be nicely etched to less than 1 mil diameter by use of the following solution.

<u>Zinc Etch:</u>

1. Chromic acid 149 gm/500 mℓ H_2O
2. Sodium sulfate 14.9 gm/500 mℓ H_2O

Use at room temperature; if yellow film occurs, remove by dip in 1% solution H_2SO_4.

With the wire mounted and bent as shown in Figure 12, a light contact is made with the freshly etched semiconductor surface. Contact is made through the 10 k ohm series resistor. Increase the 60 cps sweep to slightly ac form the diode. Remove the 60 cps and switch out the 10 k resistor. With pulse $z = 22\,\Omega$ and pulse amplitude of 3 v at a pulse width of 5 μsec, only one pulse (reverse) was needed to yield a characteristic similar to that shown in Figure 18a. The peak current can be increased by successive pulsing just as with the n-Ge. Figure 18b is representative of the characteristic I-V that can be obtained with application of four (3-4-5-6 v) pulses in the reverse direction. Figure 18c shows the same unit as in Figure 18b but with the application of one more pulse. The peak current of the unit in Figure 18c is about 5 ma.

To obtain $I_p > 5$ ma the procedure changes slightly, in that when the pulse voltage reaches 6.0 v the voltage should be increased no further. Rather, the pulse z should be decreased, starting from 22 Ω in steps down to 2 Ω. With this procedure, peak currents in excess of 100 ma are readily obtainable while the peak current to valley current ratio ranges from 3:1 to 8:1.

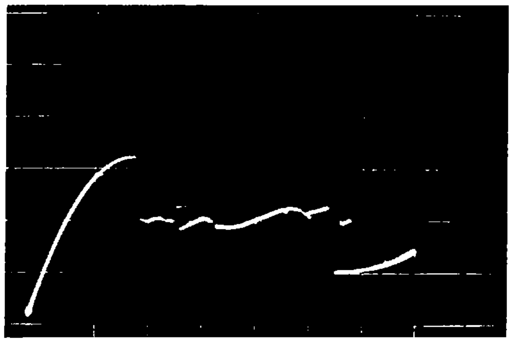
D. p-TYPE GALLIUM-ARSENIDE

The point material for use with p-GaAs is normally pure tin wire, made by the Taylor process, or foil. Tellurium and selenium doped tin wires have been tried, but with no noticeable improvement in the resulting characteristics.

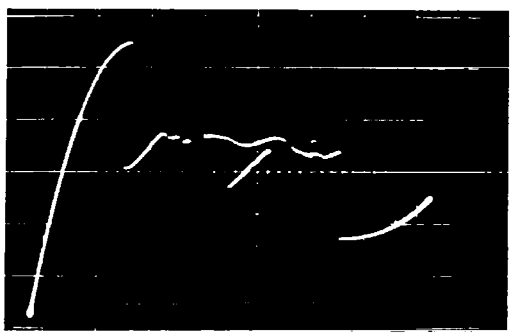
For very good back diodes of p-GaAs, the semiconductor used is less heavily doped than that used for the tunnel diodes. A dopant density of about 2.5 E 19 yields quite acceptable backdiodes.

The procedure for GaAs backdiodes is as follows. Make original "grazing" contact to freshly etched GaAs material. AC form in the reverse direction through the 10 k resistor until slight, but stable diode action is visible (on the 10 μa/cm range of I-V curve tracer). Switch out the 10 k resistor and ac form in <u>reverse</u> direction; increase 60 cps sweep slowly until breakdown occurs, at which time return sweep immediately to zero. On I-V curve tracer, junction now appears almost short-circuited. Now switch to <u>forward</u> sweep and

(a) n-GaAs, 1 pulse at 3.0 v, z = 22 Ω V.S. 100 μa/cm; H.S. 0.1 v/cm



(b) n-GaAs, 4 pulses to 6 v, z = 22 Ω V.S. 1.0 ma/cm; H.S. 0.1 v/cm



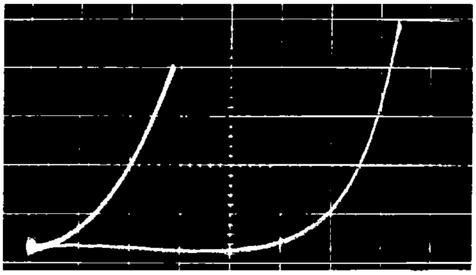
(c) n-GaAs, same unit as 18b plus 1 pulse to 7 v, V.S. 1.0 ma/cm; H.S. 0.1 v/cm

Figure 18.

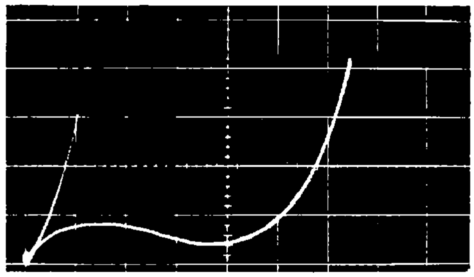
increase 60 cps sweep slowly until the curve sharply falls down and assumes a reasonable characteristic. Figure 19a shows the forward (and superimposed reverse) characteristic of one diode which had only been ac formed. The vertical sensitivity of the presentation in Figure 19a is 10 μa/cm, the horizontal is 0.1 v/cm. In most cases, however, the result of ac forming will not be so good and the junction must be pulsed. It has been determined that the most consistent forming of these backdiodes is obtained by using a fixed forward pulse amplitude of 3 v at a pulse width of 5 μsec and begin pulsing with a pulse z = 51 ohms. If additional forming is needed, the pulse amplitude and width are held constant but the pulse z can be either held constant or reduced sequentially from 51 Ω to 22 Ω, ... 5 Ω, as needed.

Figure 19b shows an I-V curve of a junction which has been ac formed and pulsed one time with a forward, 3 v amplitude pulse at a pulse z = 15 Ω. Figure 19c shows the same unit pulsed once more but with the pulse z = 10 Ω. Figures 19d, 19e, and 19f show the characteristics of a unit which in Figure 19d had been ac formed and pulsed once at 3 v with pulse z = 15 Ω, in Figure 19e the junction had received two pulses (3 v, 5 μsec, 15 Ω) and in Figure 19f the junction had received three pulses. By observing the I-V curve on the scope, while pulsing the unit, very consistent results, one diode to another, may be obtained. Notice that the result of pulsing p-GaAs is almost the reverse of pulsing n-GaAs or n-Ge, as with p-GaAs the tunnel current decreased with additional forming and with n-GaAs the tunnel current was made to increase by additional forming.

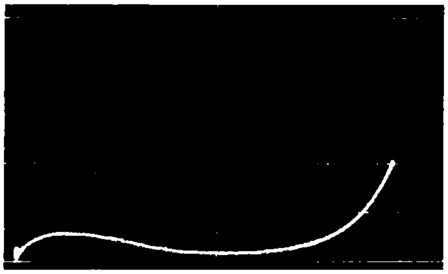
Regular tunnel diodes can be made from the most heavily doped (as already described) p-GaAs material. Tin points are used and the initial ac forming is the same as for the GaAs-Bd. Figures 20a, 20b, and 20d are representative of the curves that can be obtained by ac forming alone. Figure 20c was obtained by pulse forming the junction of Figure 20b. The pulse was a 3 v forward pulse of 5 μsec duration. The pulse z = 5 ohms. In Figure 20c, the peak current is



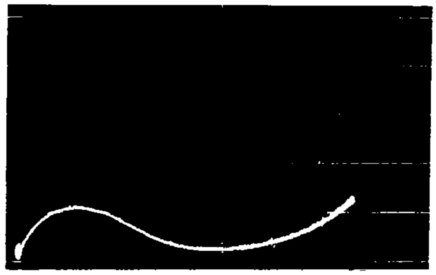
(a) p-GaAs-Bd - ac Formed Only
V.S. 10 μa/cm; H.S. 0.1 v/cm



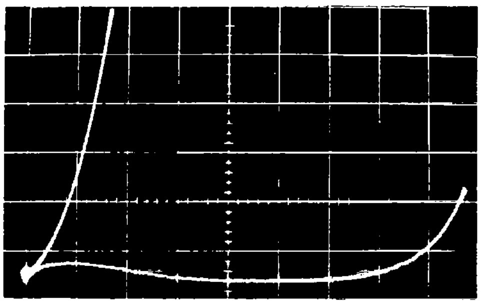
(b) p-GaAs-Bd - Same Scales
1 Pulse Forward at 3 v,
z = 15 Ω



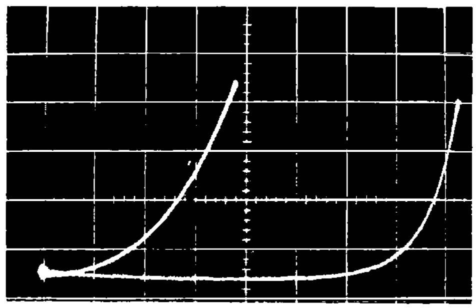
(c) p-GaAs-Bd - Same Scales
1 Pulse at 3 v, z = 15 Ω;
1 Pulse at 3 v, z = 10 Ω



(d) p-GaAs-Bd - Same Scales
1 Pulse at 3 v, z = 15 Ω

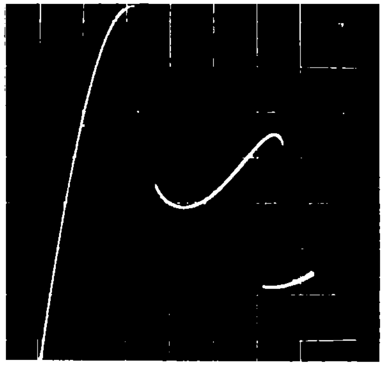


(e) p-GaAs-Bd
2 Pulses at 3 v, z = 15 Ω

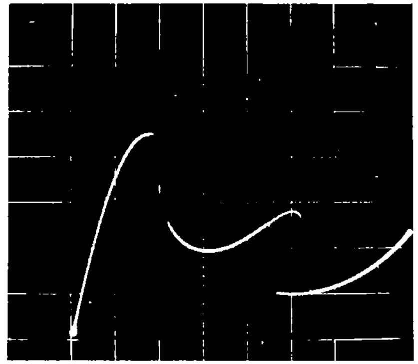


(f) p-GaAs-Bd
3 Pulses at 3 v, z = 15 Ω

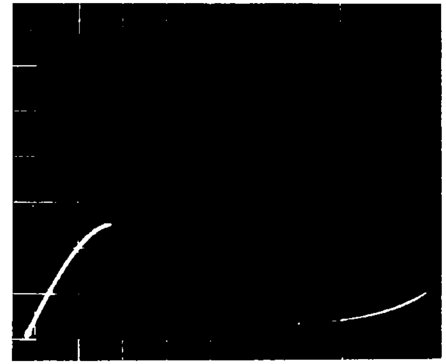
Figure 19.



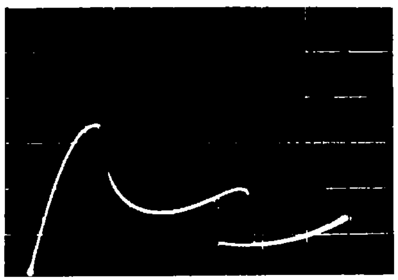
(a) p-GaAs - ac Forming Only
V.S. 100 μa/cm;
H.S. 0.1 v/cm



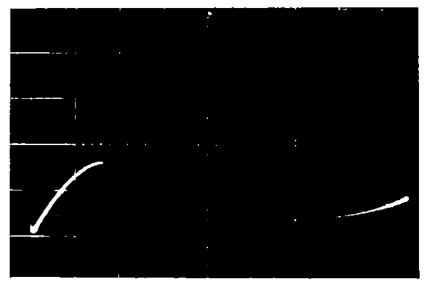
(b) p-GaAs - ac Forming Only
Sens. Same



(c) p-GaAs - V.S. 10 ma/cm;
H.S. 0.1 v/cm, 1 pulse forward,
3 v, z = 5 Ω, PW = 5 μsec



(d) p-GaAs - V.S. 100 μa/cm;
H.S. 0.1 v/cm, ac Forming
Only



(e) p-GaAs - V.S. 10 ma/cm;
H.S. 0.1 v/cm, 1 pulse forward,
3 v, z = 5 Ω, PW = 5 μsec

Figure 20.

25 ma with a peak to valley ratio of 10:1. Figure 20e was obtained by identically pulsing the junction of Figure 20d.

E. n-TYPE GALLIUM-ANTIMONIDE

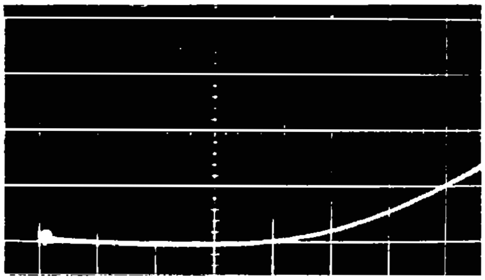
The point material which has been used most by this project for making good looking backdiodes and tunnel diodes with GaSb is pure copper wire. Common solenoid wire, No. 42, is stripped of its enamel and etched down to a diameter just under 1 mil.

Copper Etch:

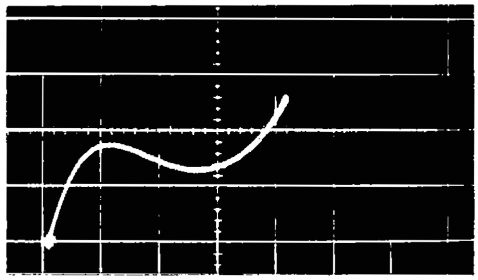
1. 8 parts H_2SO_4
2. 4 parts HNO_3
3. 1 part H_2O

The copper wire is mounted and shaped as shown in Figure 12. Contact is made to the freshly etched semiconductor surface. The contact is monitored by use of the I-V curve tracer with the series 10 k resistor in the circuit. The 60 cps sweep is increased in the forward direction until a stable diode curve is obtained. Remove the series 10 k resistor. The junction is pulsed in the forward direction with a pulse amplitude starting at 1.5 v, the pulse width is about 1700 μsec and the pulse z = 51 ohms. With each additional pulse used, the amplitude is increased by 0.5 volt.

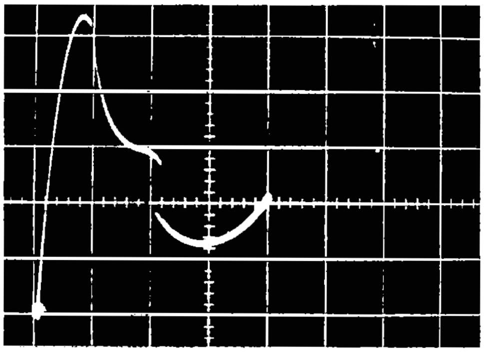
Figure 21a shows the I-V curve of the n-GaSb unit with only slight, forward, ac forming. Figures 21a through 21f are photographs of a typical forming sequence. This sequence shows a trend similar to that encountered with p-GaAs, in that after the initial few pulses with which the junction is formed as shown in the figures up to 21c, the peak current reaches a maximum (anywhere from 20-60 μa) and then begins to decrease but still maintaining a good peak current to valley current ratio. As the pulse amplitude continues to increase with each additional pulse, there is a point beyond which the current begins again to increase. After this point the junction behaves essentially



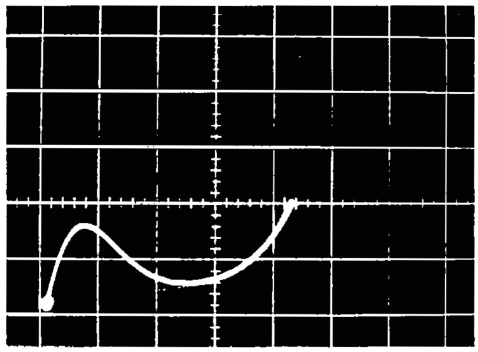
(a) Slight ac Forming Only



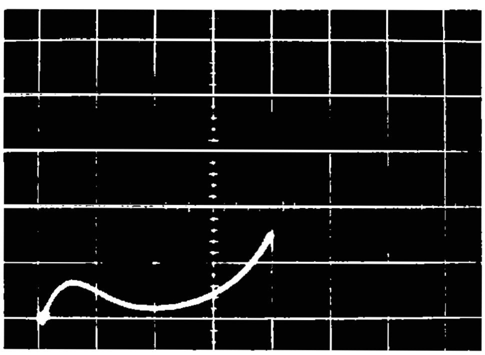
(b) 1 Pulse Forward at 1.5 v, PW = 1750 μsec, z = 51 Ω



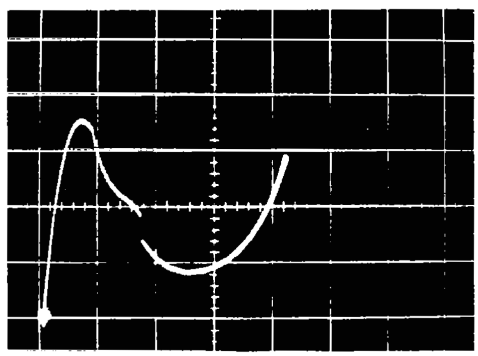
(c) 4 Pulses to 3 v, z = 51 Ω



(d) 5 Pulses to 3.5 v

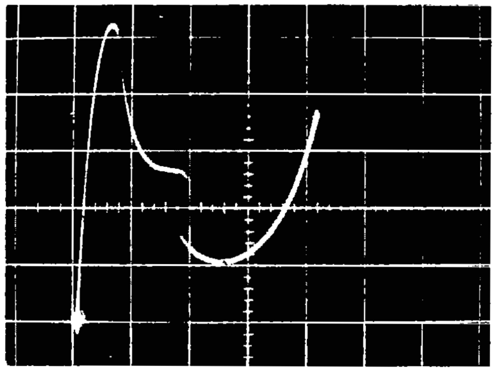


(e) 6 Pulses to 4.0 v

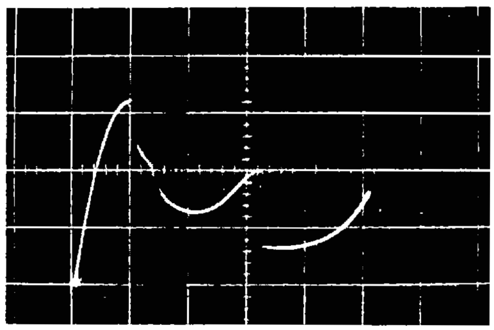


(f) 7 Pulses to 4.5 v

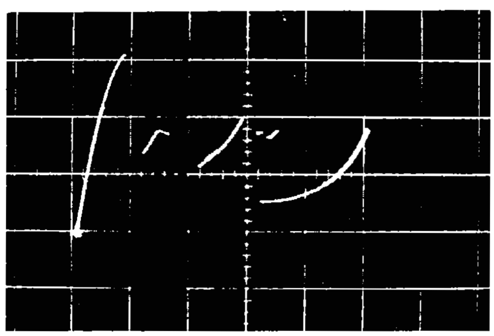
Figure 21. n-GaSb, Forming Sequence



(g) 8 Pulses to 5.0 v, V.S.
10 μa/cm; H.S. 0.1 v/cm



(h) 12 Pulses to 7.0 v, V.S.
100 μa/cm; H.S. 0.1 v/cm



(i) 16 Pulses to 9.0 v, V.S.
1.0 ma/cm; H.S. 0.1 v/cm

Figure 21. n-GaSb, Forming Sequence (Contd)

as the n-Ge or n-GaAs in that the peak current smoothly increases with each additional pulse. Figures 21g through 21i show this increase. Figure 21g shows a peak to valley ratio of about 5:1 while as the peak current increased so did the peak to valley ratio increase to more than 6:1.

F. FORMING AND DISPLAY CIRCUITS

The test assembly which contains all the equipment necessary to both monitor the I-V curve of the diode under fabrication and supply the necessary electrical forming pulses was shown in Figure 13 which is a photograph of the physical set-up of the diode assembly apparatus. The rack mounted equipment which contains the forming and display circuits is shown, again, in Figure 22. The diagram for this equipment was already given, in block form, in Figure 16.

The schematic diagram of the curve tracer is given as Figure 23. It is a relatively standard circuit. When operating ac output, the sweep is half-wave rectified, and is used to display the I-V curve on the oscilloscope. If a permanent recording on an X-Y recorder is desired, the dc position is used and the supply then becomes a full-wave bridge with heavy filtering on the output which is sufficient to give good results with the X-Y recorder. Facility is there also to bring in an external sweep voltage or bias voltage, if it be so desired. The vertical gain control is used to adjust the conversion from the current sensing resistor to the scope vertical input such that the scope vertical sensitivity calibration can be read directly in milliamperes. The calibration is effected by switching the TEST-CAL. switch to Calibrate. This places a 500 Ω resistor in the place of a diode. The scope vertical sensitivity switch is placed in the 1.0/cm scale and the horizontal for 0.10 volts/cm. Then, using the control ac volts, the sweep is adjusted for 10 cm deflection. The VERT GAIN control is adjusted to give a sweep display slope of 0.2 (2 cm vertical for 10 cm horizontal). All the positions of the scope

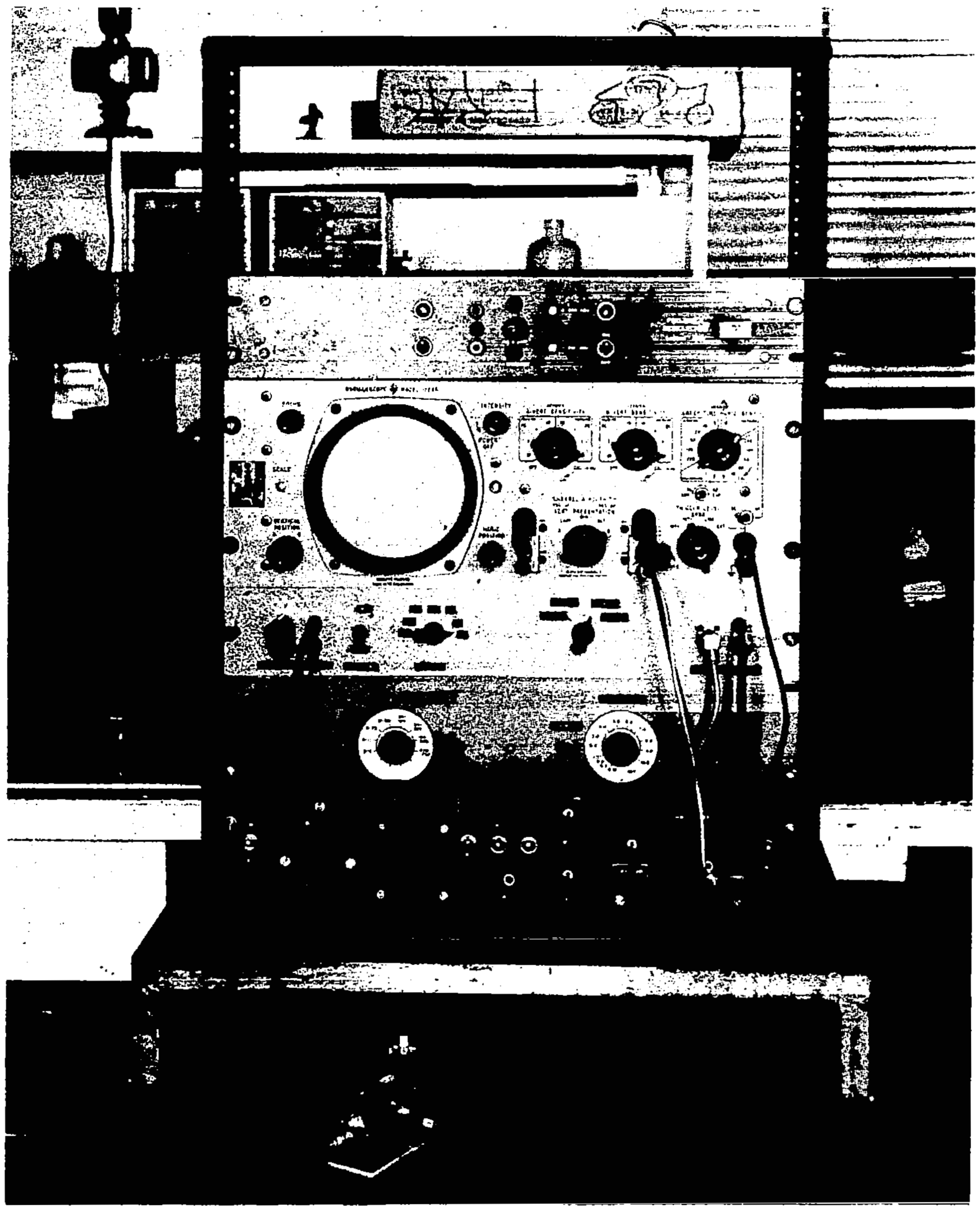


Figure 22. Photograph of Forming and Display Equipment

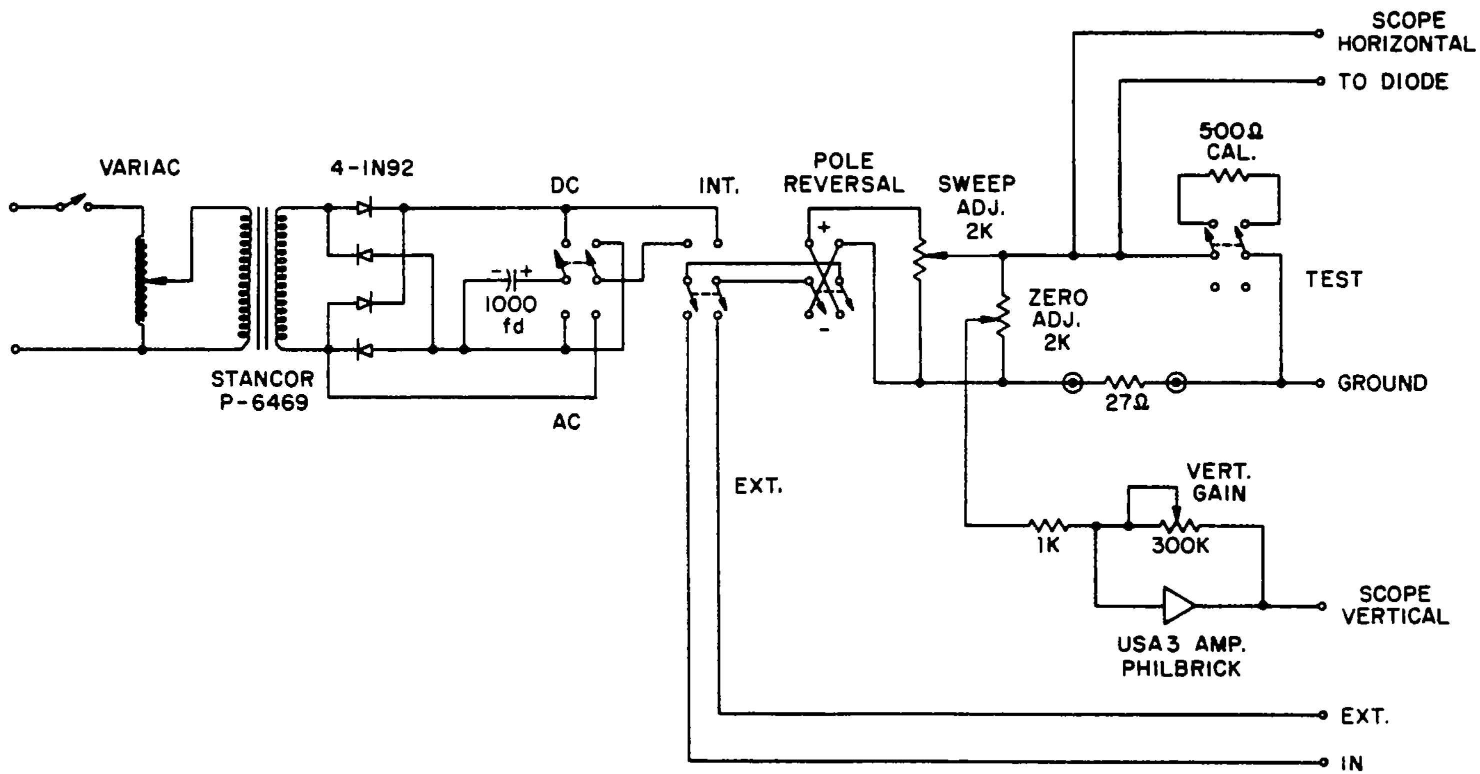


Figure 23. Schematic Diagram - I-V Curve Tracer

sensitivity control are now calibrated. The ZERO adjust potentiometer is used only when a stablizing resistor is used in parallel with the diode under test, and is adjusted such that with no diode connected to the test terminals, the I-V display will be perfectly horizontal (no slope).

The schematic diagram of the pulse generator is shown in Figure 24. It is basically a one-shot transistor multivibrator, with a one-shot multivibrator as a trigger. The on-time of the triggering multivibrator is used to insure that chatter from the PULSE TRIG microswitch could not cause the pulse generator to multiple fire. The MANUAL-RECURRENT switch is used only for testing the pulse generator itself. With the switch in the RECURRENT position it is much easier to check the ranges of the pulse width and amplitude controls. The output pulse capability of this circuit, is a pulse variable in amplitude from zero to 10 volts peak, and is capable of delivering up to about 20 amperes peak pulse current.

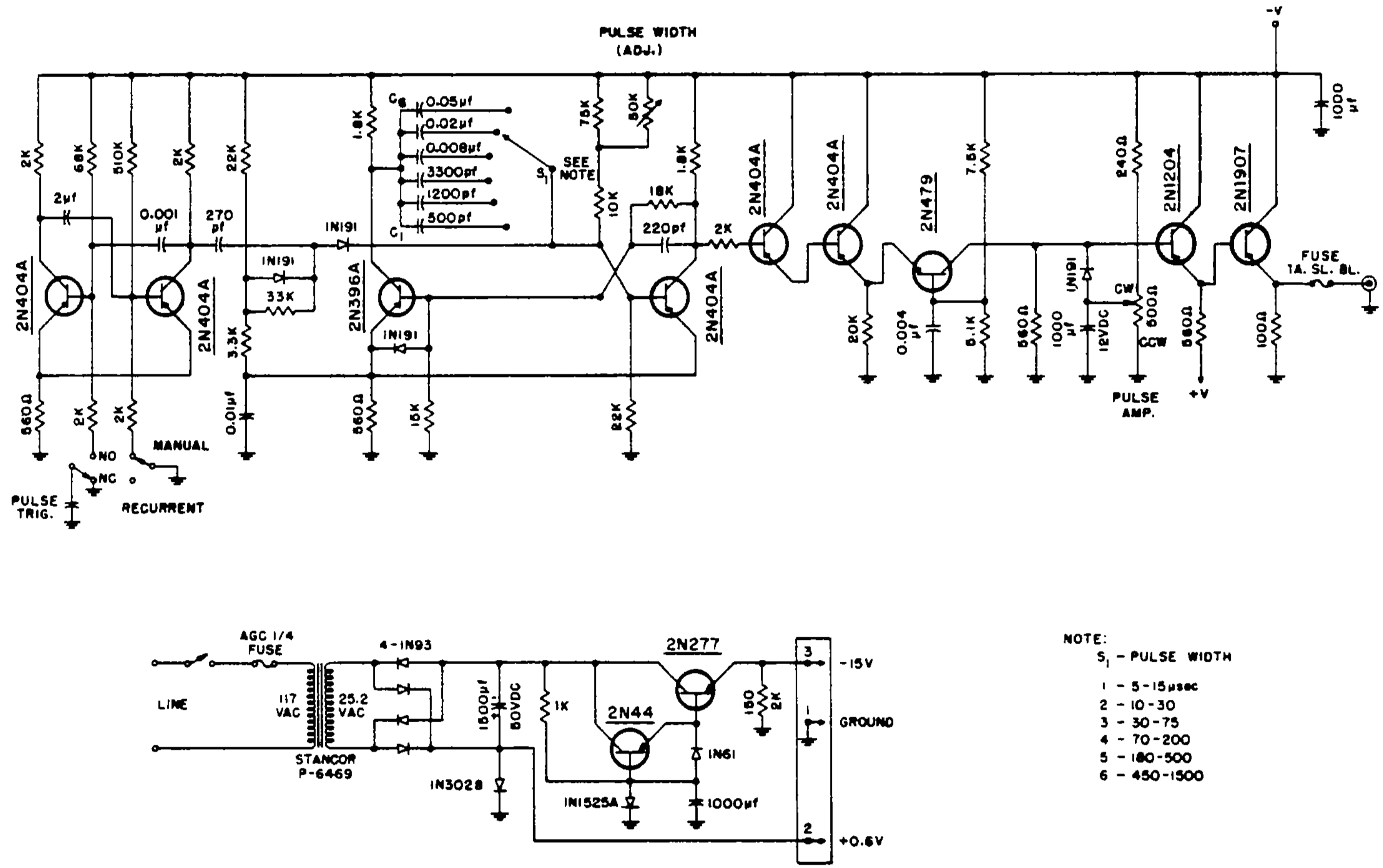


Figure 24. Schematic Diagram - Pulse Generator and Power Supply

VI. EXPERIMENTAL WORK

Of the experimental investigations of diodes as oscillators and detectors it can be reported that in a G band holder, a p-GaAs diode has attained a frequency of oscillation of 82.5 gc. At 20 gc both p-GaAs and n-GaAs with peak currents of 5-10 ma have yielded power of about -10 dbm. To quote one typical case: n-GaAs, with I_p = 5 ma and P/V = 5:1 had a measured power out, P_o = -10 dbm and frequency tunable across the range 17-21 gc.

The experimental work of determining the sensitivity of the video detector backdiode is being performed in detail. The fabrication techniques developed by this project have proceeded to the point where it is felt that sufficiently reproducible results can be obtained to warrant a full scale program to determine electrical parameters. Both 25 gc and 70 gc test systems have been assembled and accurately calibrated. The video bandwidth of the test system is about 30 kc. The integrated noise figure of the transistorized video amplifier is less than 2 db; the gain is 80 db.

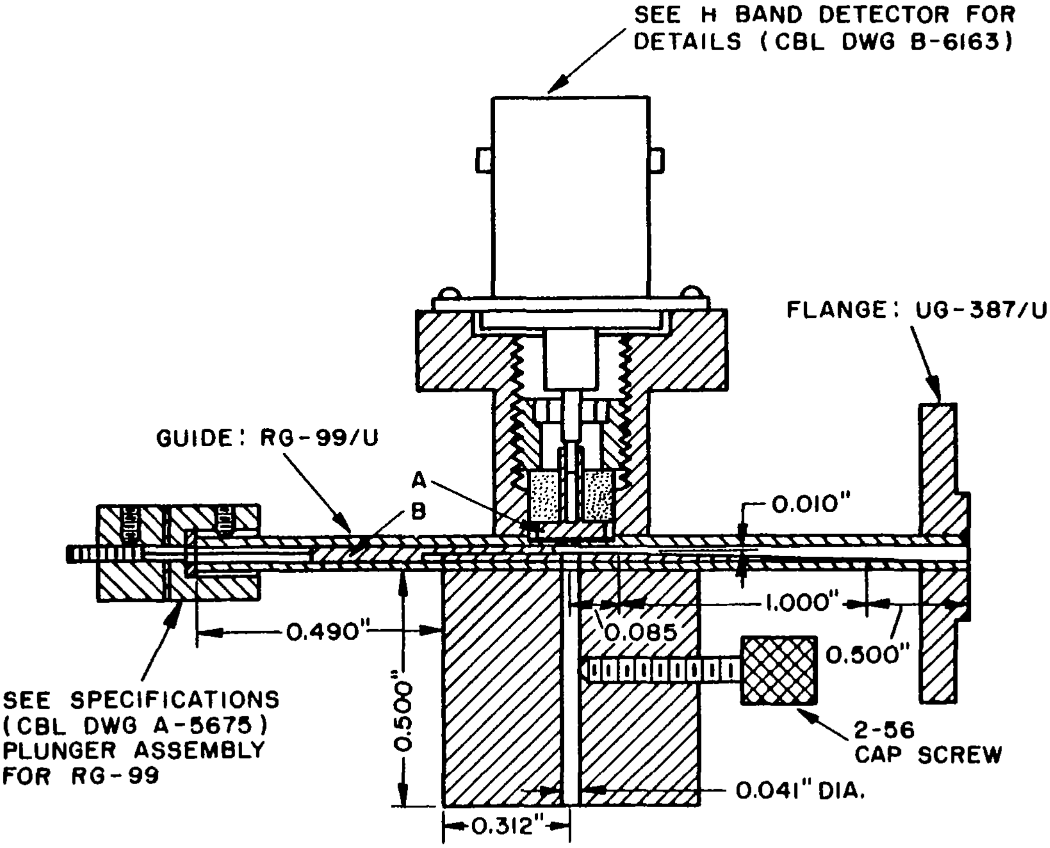
Several commercial diodes have been tested at 25 gc to obtain a basis for comparison with the backdiodes produced at the Carlyle Barton Laboratory. Both dynamic range curves and an MDS (Minimum Detectable Signal) are obtained for these diodes. At 25 gc, the best MDS for the commercial units was -64 dbm and was obtained with one MA-IN26. The other IN26's were considerably less sensitive. For the standard laboratory "run-in" type of diode using a silicon wafer and a frequently resharpened tungsten catwhisker, the absolute best that could be obtained was an MDS of -68 dbm with quite an unstable junction (measured only once before failure). As the junction was stabilized, this sensitivity was markedly reduced. MDS levels have been obtained for many back diodes with peak currents that range from 30 μa to 400 μa for the germanium units and 1 μa to 4 μa for the p-GaAs units. The sensitivity of the Ge units could normally be improved 1-2 db with application of approximately 10 μa of dc bias.

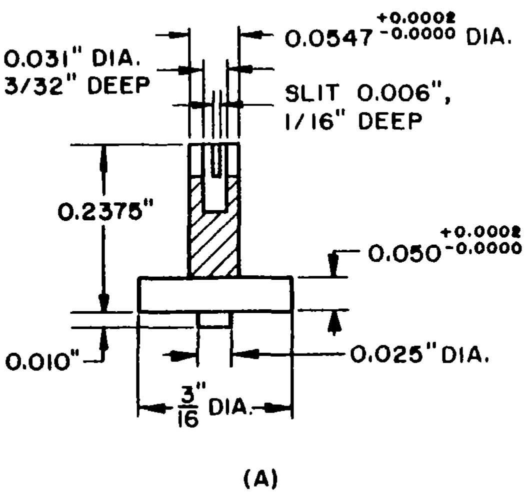
At 70 gc a De Mornay-Bornardi holder with a IN78 diode gave a sensitivity of -48 dbm. The standard silicon-tungsten "run-in" type had -63 dbm as an absolute best sensitivity with no improvement by application of dc bias. The back diodes tested in the 25 gc circuits were also tested in the 70 gc set-up. The 30-40 μa devices gave -64 dbm sensitivity at zero bias and about -65 to -66 dbm with bias. Devices (still germanium) with 60 μa peak current exhibit a sensitivity of about -62 dbm. The 30-40 μa devices are well matched to the 10 mil (reduced height) waveguide. This is evidenced by the fact that there is less than 1 db change in the MDS when an E-H Tuner is used as an impedance matching device to match the signal into the diode holder.

To date only a relatively few diodes have been completely fabricated and tested in the detector circuits. Several of the steps in the fabrication process are still under development and therefore very little of the process is considered as finalized. But it is important to note that of those diodes tested in both the 25 gc and the 70 gc test sets, the same sensitivity is obtained at both frequencies, which implies that the diode junction and its package have not, at 70 gc, begun to approach the frequency cut-off point.

The tunnel diode holders, and the backdiode holders to which reference has been made above, are shown in detail in Figures 25, 26a, 26b, and 27a, 27b. Figure 25 shows the details of the H band (60-90 gc) detector mount. The input is from standard RG-99/U waveguide which is then reduced to 0.010 height waveguide for impedance matching into the diode. This basic structure has been used for the fabrication of diode holders for frequencies which range from 25 gc to 140 gc.

Figures 26a and 26b show the details of the K-band (18-26 gc) detector mount. As stated above, the basic structure is the same as that for H-band, only the waveguide sizes have been modified to adapt to the proper frequency band.





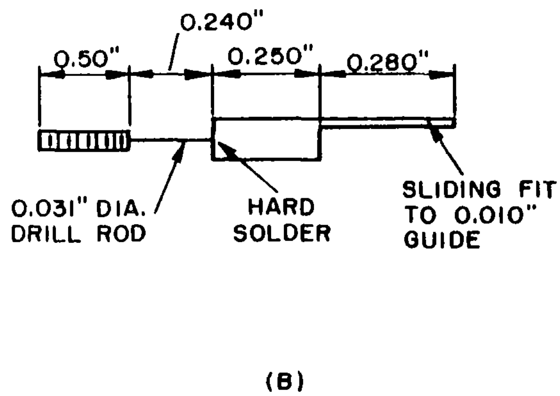


Figure 25. H-Band Diode Mount

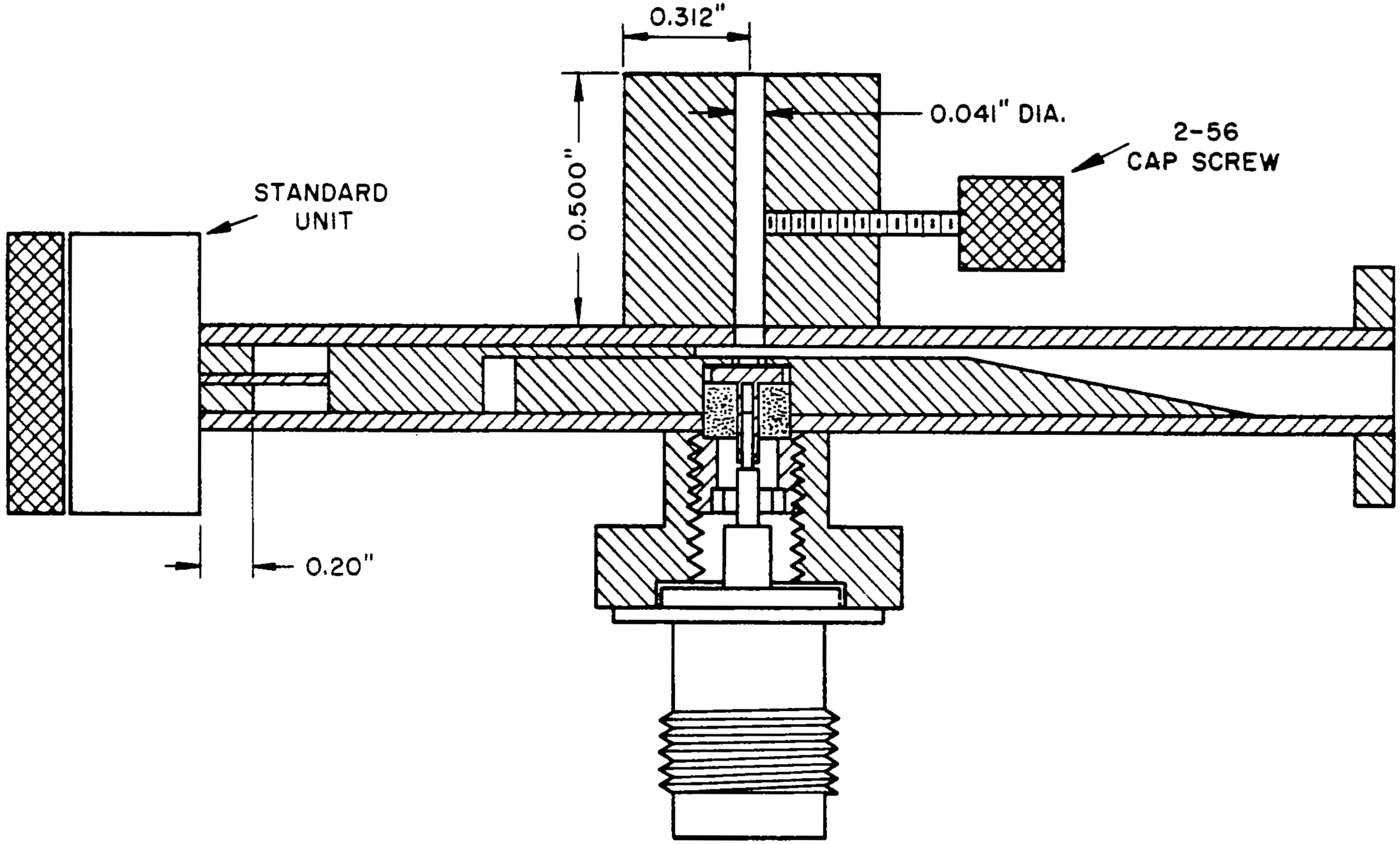


Figure 26a. K-Band T-D Mount Mod. 3

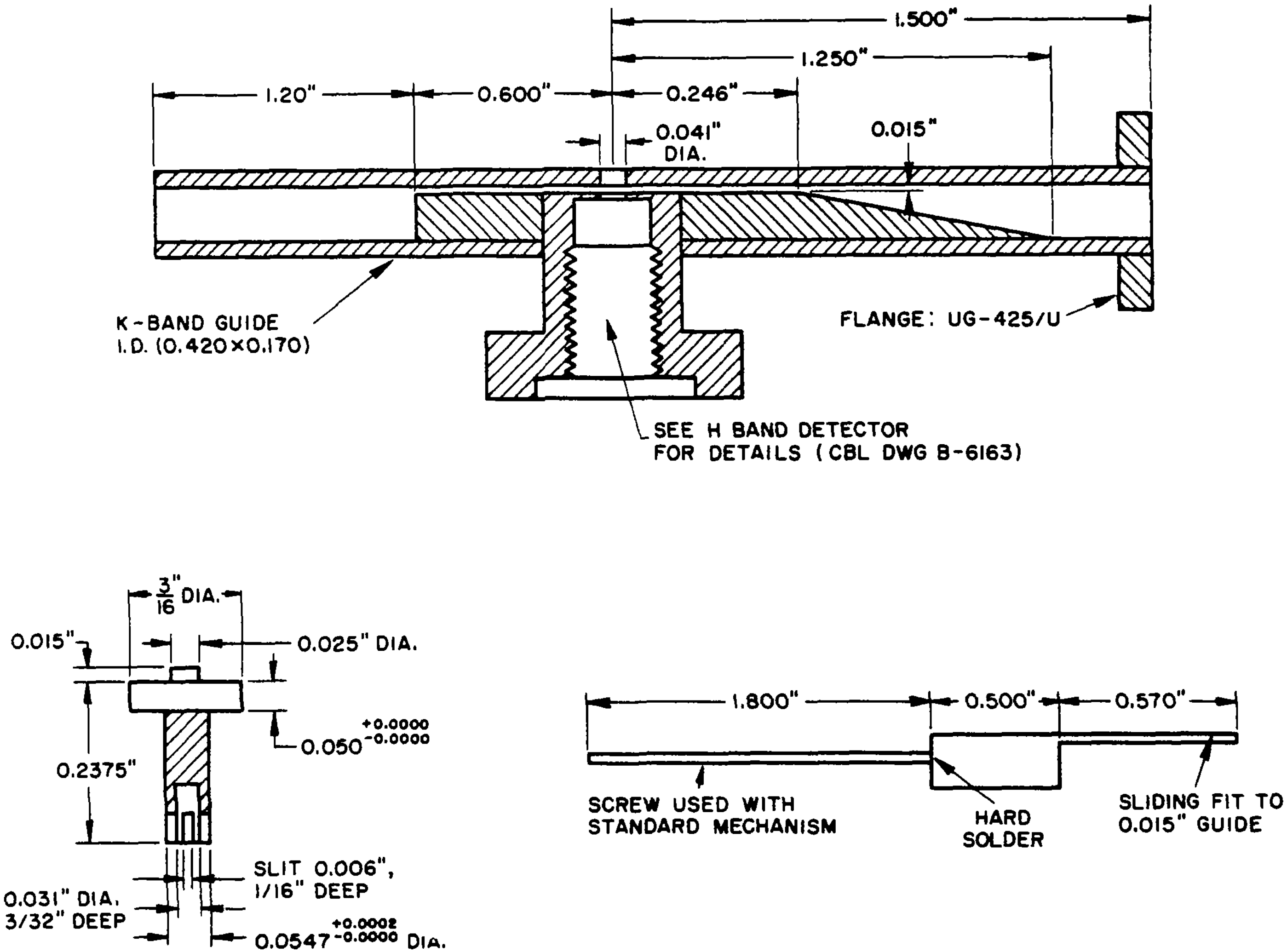


Figure 26b. K-Band Diode Mount

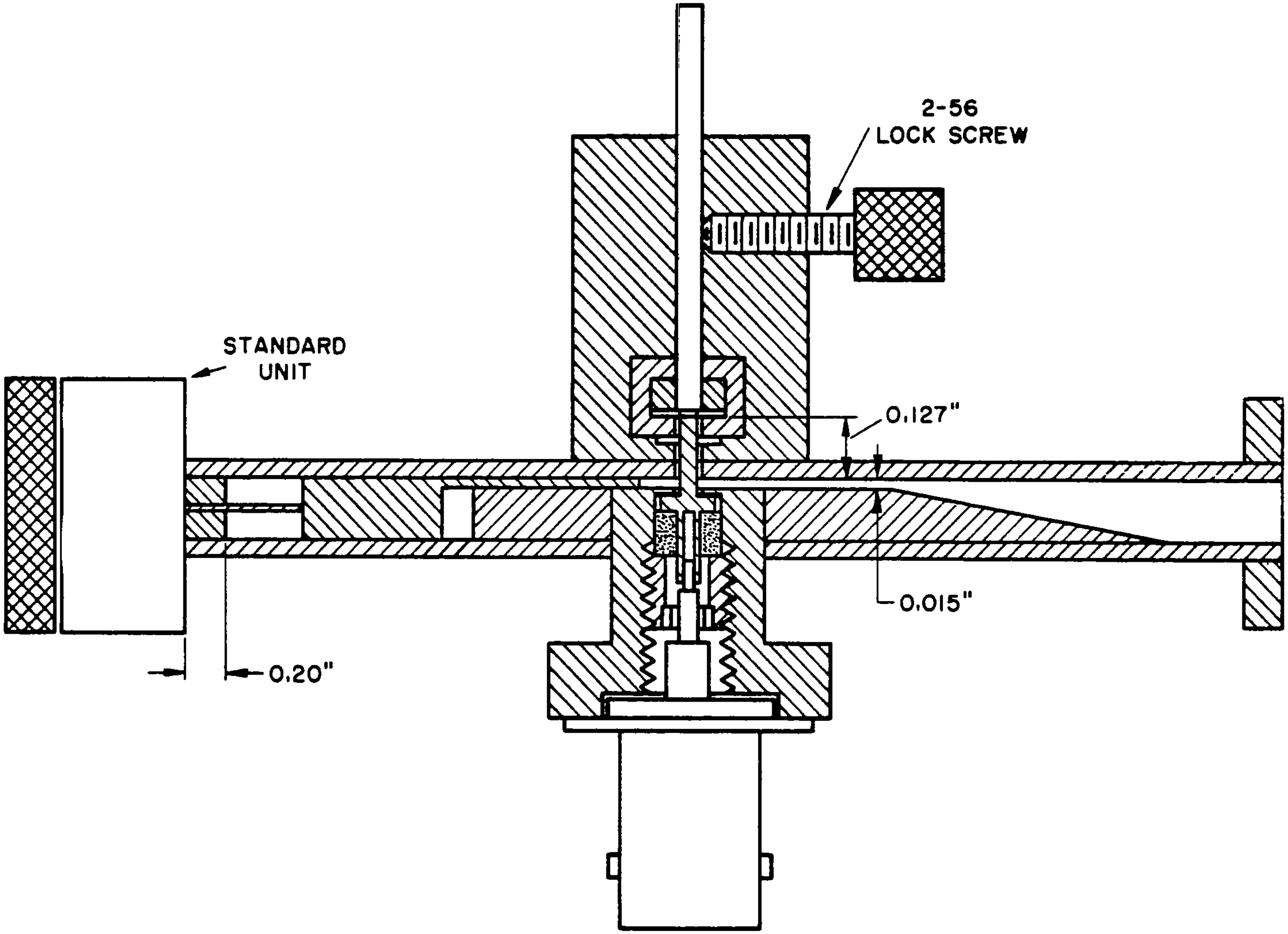


Figure 27a. K-IH Band Harmonic Generator

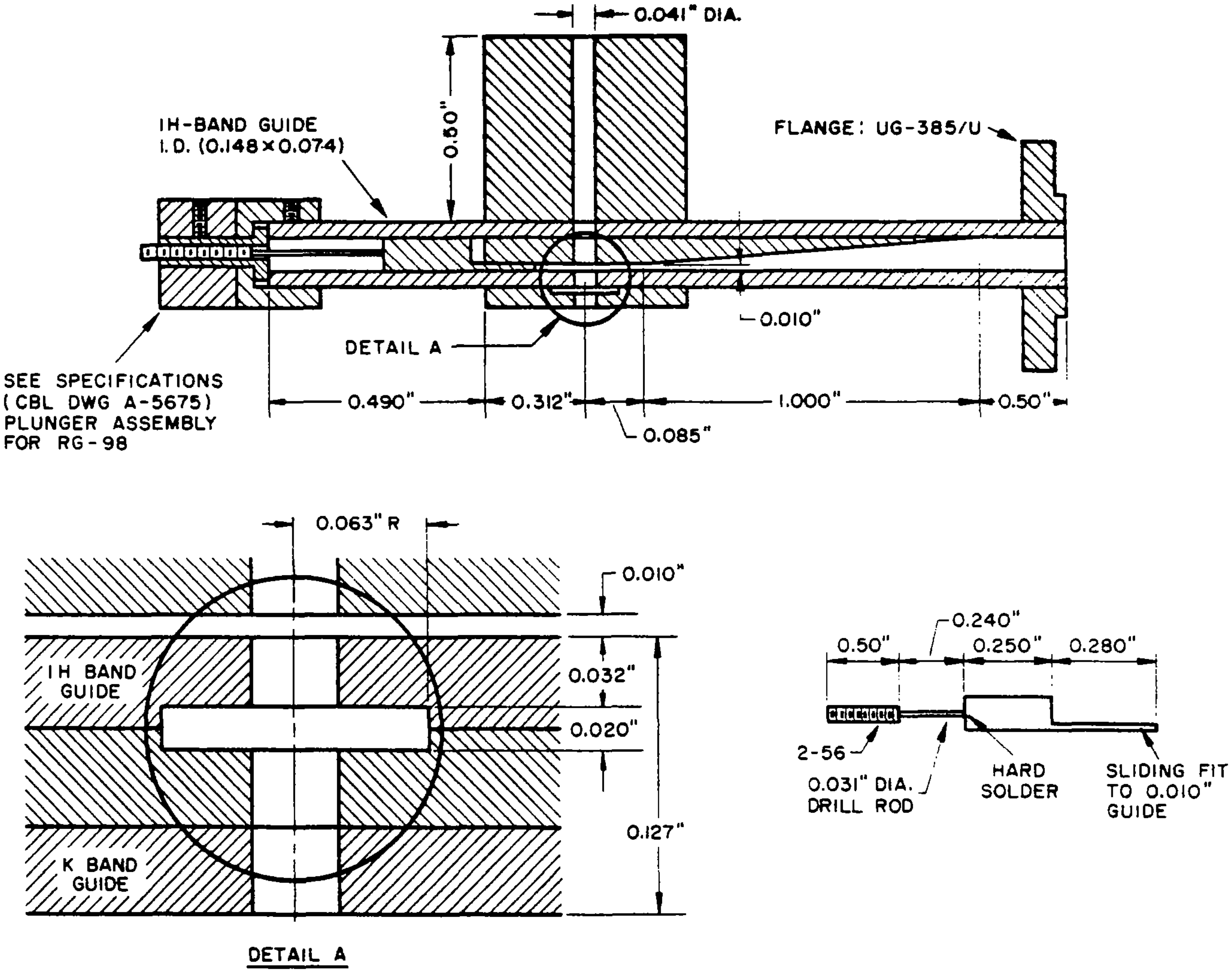


Figure 27b. K-IH Band Harmonic Generator

Figures 27a and 27b present the details for a K-1H bands (IH is 50-75 gc) Harmonic Generator. A radial cavity is used to isolate the K-band guide from the IH-band guide so that an IH-band signal will not be transmitted to the K-band guide and thus affected by K-band tuning adjustments. The K-band signal cannot propagate through the IH-band guide and thus the canonical design is approached, for which the tuning at the two different frequencies is noninteracting. This device has not yet been tested as a harmonic generator, but the canonical design feature has been tested by subjecting the K-band and the IH-band portions individually to tests by using them as simple video detectors and checking to determine the extent of the interaction of the frequency tuning adjustments. The design appears to be sound as the tuning interaction is nonexistent and the detector efficiency (at both bands) appears to be the same as was measured in the regular detector mounts of Figures 25 and 26.

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