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Final Report

Covering the Period February 9, 1969 to April 9, 1970

LOW-TEMPERATURE THERMIONIC EMITTER

Prepared for:

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
ELECTRONICS RESEARCH CENTER
575 TECHNOLOGY SQUARE
CAMBRIDGE, MASSACHUSETTS 02139

CONTRACT NAS 12-607

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Final Report *May 15, 1970*

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By: A. ROSENGREEN and D. NITZAN

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PREFACE

This project was established by the National Aeronautics and Space Administration, Electronics Research Center, Cambridge, Massachusetts, on February 8, 1968. The Technical Monitor for the project is Andre Garfein, and the program is under the technical direction of Arne Rosengreen. Laboratory work is being performed by David M. Murdock and Joseph H. Hunt, Jr.

ABSTRACT

The processes developed for fabrication of integrated vacuum circuits (IVCs) are described. A 1-bit shift register is discussed. It is analyzed graphically by dividing the circuit up in smaller test circuits consisting of an inverter, two inverters in cascade, and a NOR gate. Fabrication and testing of these circuits are discussed. Particular attention is paid to the emission of diodes and triodes in the reverse direction, which prevented the circuits from operating. It is suggested that this problem is associated with flow of molten barium along the metal films during the activation process. Data from life test of dual triodes are presented. All coplanar triodes, with a few exceptions, have proved to have relatively short lives, although conventional triodes with cathodes made from the same mixture of triple carbonates and photoresist used for the IVCs have shown no deterioration after more than 12,000 hours. It is suggested that the problem is connected with a slow migration of barium from the cathode along the connecting films.

CONTENTS

LIST OF ILLUSTRATIONS

Figure		Page

LIST OF TABLES

I INTRODUCTION

During the last two decades the development of new electronic devices has been almost exclusively in the area of solid state devices. Since the existing vacuum devices have not seemed as amenable to miniaturization and mass production as the solid state devices, little attention has been paid to the development of new vacuum devices.

A major disadvantage of vacuum devices has always been their need for thermionic emission. To obtain reasonable current densities, the required operating temperature of the cathode may be as high as 750-800°C with consequent reduction in the life of the cathode, which is a direct inverse function of the cathode temperature. However, the recent development at Stanford Research Institute of a triple-carbonate photoresist method for producing cathodes has made it possible to reduce the cathode temperature to about 650°C without reduction of the current density. In addition, the use of coplanar structures opens up the possibility of microminiaturization using conventional photolithographic techniques. The purpose of the present contract was to explore these possibilities for development of coplanar microminiature low-temperature thermionic devices and circuits that should be able to operate at much higher ambient temperatures than their counterpart solid state devices.

II OBJECTIVES

The objectives of this program are to develop techniques for fabrication of complex circuitry of coplanar low-temperature thermionic devices on a single substrate with subsequent development and fabrication of a high-speed 1-bit and 10-bit shift register.

III TECHNICAL DISCUSSION

A low temperature thermionic emitter was developed at Stanford Research Institute during an earlier portion of this program. In the course of this work it was found that Richardson's constant, governing the electron emission, was increased by mixing the triple carbonate powder, normally used for thermionic emitters, with a photoresist. Operating temperature could thus be reduced without any sacrifice of current density. Also, the mixture allowed the cathode material to be placed at selective areas by the use of photolithographic techniques. Using this technique and the improved electron emission characteristics of the cathode, a new concept in integrated circuits was conceived: the integrated vacuum circuit (IVC).

The IVC consists of an interconnecting pattern of coplanar cathodes, grids, and anodes evaporated or sputtered on an insulating substrate to form a network of diodes and triodes. The substrate is heated uniformly to about 650°C and electrons are emitted from the cathode into the vacuum surrounding the substrate and are collected on the anodes. The IVC has no resistors in the conventional sense. Their function is taken over by the dynamic resistance of the diodes. Since a photolithographic process is used exclusively in the fabrication of the IVCs, they lend themselves to microminiaturization. The following sections describe the techniques developed in this program for production of microminiature circuits, specifically a 1-bit and a 10-bit shift register.

A. Fabrication Techniques

The substrate used for the IVCs must be mechanically stable up to about 1000°C in order to withstand the activation temperature of the cathodes. It must also be able to sustain operating temperatures in the range 600 to 700°C for a long period of time and have a high resistivity in this temperature interval. In the original work on low temperature thermionic emitters, the substrate material employed was polycrystalline alumina, but this was later changed to single crystal sapphire to avoid the grain boundary effects in the alumina that might create problems in microminiaturization. The sapphire substrates are 0.03-inch thick circular platelets with a diameter of 3/4 inch that have been polished and cleaned by etching.

The first process consists of sputtering a thin film of tungsten on one side of the wafer. By use of photoresist and photolithographic techniques, a pattern is then etched in the tungsten film leaving tungsten only at the cathode areas. Figure 1 shows a typical layout of an IVC; the circuit is an inverter that is part of the 1-bit shift register. This circuit will be discussed in detail later. Here it is mentioned merely as an aide for understanding the fabrication of the IVCs. The cathode consists of the black and the crosshatched areas. Actually, the tungsten extends slightly beyond these areas to allow for an overlap with a second metal film to be applied next. The metal film can be of either nickel-titanium or molybdenum-zirconium sputtered over the entire surface and then delineated in the same way as the tungsten film. The entire circuit pattern with the exception of the cathodes is made using one of these metal combinations. Originally, nickel-titanium was used but it was difficult to remove the unwanted part of the metal film completely and excessive current leakage of the devices resulted. Improvement was made by evaporating (rather than sputtering) the nickel-titanium, but the adhesion of the film was poor. This problem was partly overcome by delineating the whole circuit in tungsten and then covering everything except the cathodes with nickel-titanium. However, the etching of the nickel-titanium proved to be a problem. The nickel-titanium layer is obtained by evaporating first a layer of titanium and then a layer of nickel. The etching rate of nickel is much faster than the etching rate of titanium, and results in a strong undercutting of the nickel and hence a very small yield. For best etching, the titanium should be on top of the nickel, but then contact problems would arise because of oxidation of the titanium. Contacts are made to the films by small spring clips contacting small contact pads at the periphery of the wafer, as will be described later. Another solution to the etching problem would be to evaporate and etch the titanium first, but because of oxidation of the titanium, the nickel layer does not stick to the titanium.

The main reasons for selecting nickel-titanium initially were the compatibility of these materials with the processes encountered in the tube and the known ability of titanium to "kill" the thermionic emission as well as to provide a good bond to the sapphire by forming an oxide at the interface, presumably by reacting with the sapphire. However, the reaction apparently takes place only when the titanium is sputtered and in this case the reaction leads to significant leakage. Other metal combinations, such as molybdenum-zirconium that have properties similar to the nickel-titanium, were therefore investigated. The molybdenum-zirconium, besides sticking well to the sapphire, proved easy to remove by aqua regia without any subsequent leakage problems. This combination of metals was therefore used for most of the circuits to be discussed later.

After sputtering and delineation of molybdenum-zirconium, the circuit is ready for its last process in open air, the application of the mixture of triple carbonates and photoresist. The mixture is applied to the crosshatched areas of the cathodes in up to three layers, again using the photolithographic technique. The wafer, shown in Fig. 2, which has undergone this process, is now ready for mounting in a tube, as is shown in Fig. 3. A heater strip of Grafoil is sandwiched between the sapphire wafer and an alumina wafer held together by two large clips. The structure is positioned in the tube by two heavy wires that also function as conductors for the heater strip. Contacts are made to the small contact pads at the periphery of the sapphire wafer with small tungsten clips connected by fine wires to the pins of the header of the tube. The tube, after bakeout, is pumped down below 10^{-8} torr for activation of the cathode.

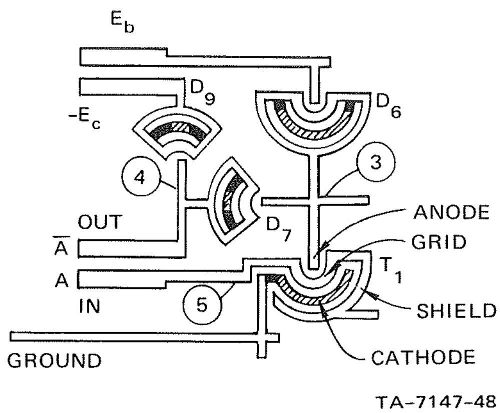

FIGURE 1 LAYOUT OF AN INVERTER

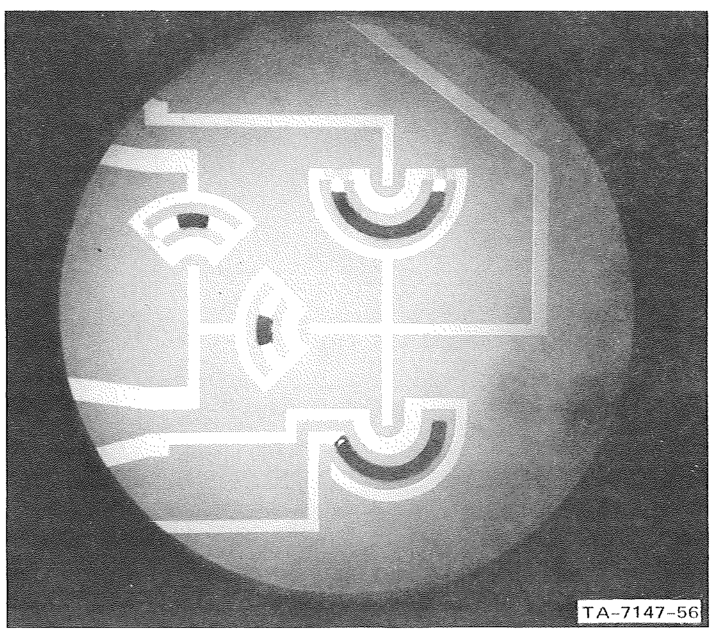

FIGURE 2 MICROGRAPH OF AN INVERTER ON A WAFER READY FOR MOUNTING

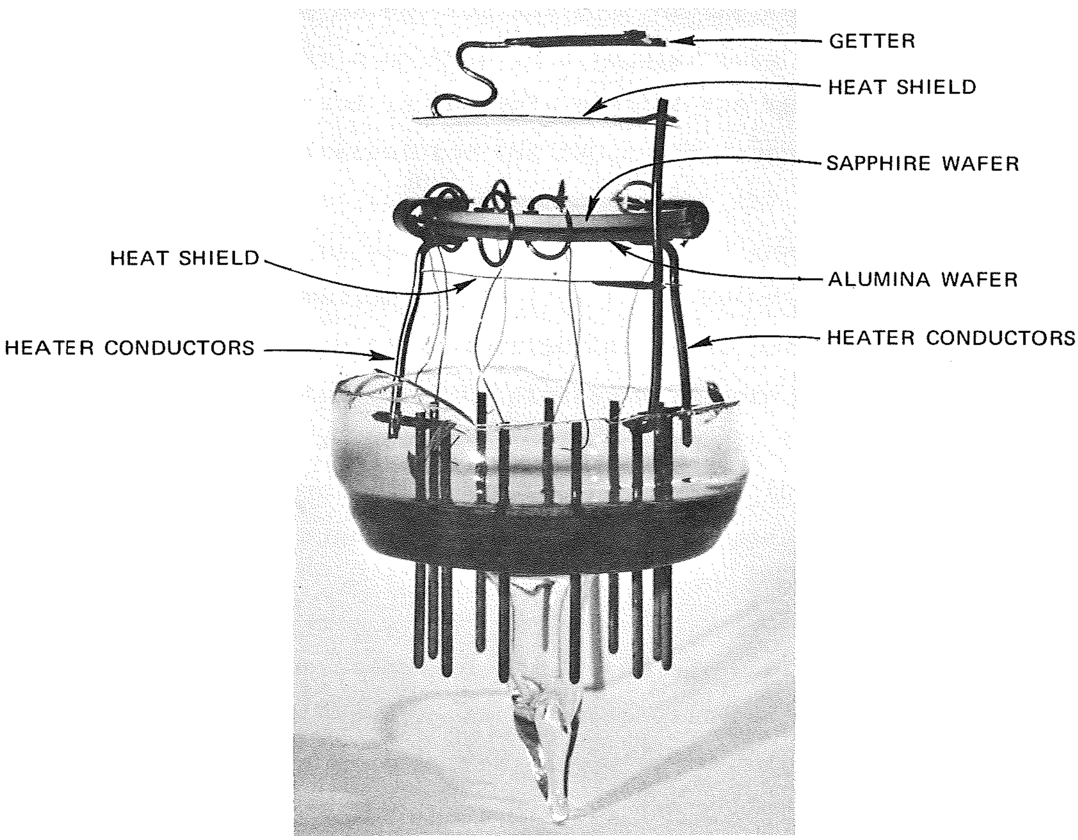

FIGURE 3 MOUNTING OF WAFER IN A TUBE

The activation is performed by heating the sapphire wafer to about 900°C for a short time by use of the heater strip. The carbonates decompose to oxides at these temperatures and the barium oxide is reduced to barium, presumably by the carbon left from the photoresists. Since the melting temperature of barium is 725°C, it is molten during the activation which allows it to coat the other oxides with a few monolayers of barium. Their work function is thus significantly reduced and good emitters are formed. The process itself is the same as is used in a regular tube except for the role of the carbon from the photoresist. However, while a cathode in a regular tube is isolated from all other parts by vacuum and is the only part of the tube that is hot, the situation is quite different for the IVC. The sapphire wafer with the entire circuit is at activation temperature and the cathode material is connected to the rest of the circuit by sapphire and strips of metal film. The proper operation of the IVCs is therefore strongly dependent on the physical and chemical properties of these parts with respect to the barium. This feature will be discussed in more detail in connection with the experimental results. It suffices to say that the activation is the most crucial process of the fabrication of the IVCs. After activation the getter is flashed and the tube is tipped off.

B. Crossovers

The fabrication techniques described above are used for simple circuits with a small layout. As the complexity of the circuits increases and the layout gets larger, there is need for crossing the conductors requiring an insulator between the conductors. The circuit of the 1-bit shift register alone requires 15 such crossovers. The insulator to be used in the crossover must meet the following requirements:

1. It must have a coefficient of thermal expansion close to that of sapphire.
2. Its resistivity must be high at the operating temperature (600-650°C).

3. The melting temperature of the material must be greater than the temperature used for activating the cathode (900-1000°C).

4. It must bond to tungsten without dissolving or lifting it from the substrate.

5. The surface must be smooth to allow reliable cross connections.

An eutectic mixture of CaO, MgO, and Al_2O_3 appears to have these properties. The materials are mixed in powder form, using $CaCO_3$ to provide the CaO. The mixture is premelted at 1500-1550°C in a platinum crucible and quenched in water. The resulting glass frit is reduced to a fine powder, using an alumina ball mill for 24 hours. The powder is mixed with photoresist and applied to the wafer, after which the normal photolithographic process is used, leaving photoresist with the glass mixture only at the crossover points. The wafer is then heated to the melting temperature of the glass (about 1345°C) in either vacuum or argon gas.

The crossover material has been tested on both tungsten and molybdenum-zirconium films. The initial experiments were made by use of small pieces of a hardened mixture of photoresist and oxides placed by hand across the film strips. The crossovers on the molybdenum-zirconium films looked excellent, but on the tungsten films there was some evidence of narrowing of the tungsten strip, probably due to the contraction of the crossover material during cooling. Also, the tungsten film lifted from the substrate in several areas.

The technique used in fabricating IVC circuits consisted of spinning three layers of the mixture of photoresist and oxides across the wafer before the lithographic processing in order to obtain sufficient material at the crossover points. The process was tested on a tungsten film and it was found that the crossover material did not wet the tungsten film but formed small pools at the edges of the film strip. This problem was solved by oxidizing the tungsten prior to applying the photoresist. This changed the metal surface so the crossover material wetted the entire crossover area. It was also noticed that the crossover material now had only a slight effect on the tungsten film, probably because of the smaller amount of the crossover material that was used compared to the experiment above and perhaps also because of the changed surface of the tungsten film from the oxidation. Similar tests have not yet been performed on the molybdenum-zirconium films, but the process looks hopeful because of the excellent results obtained in the preliminary tests with the hand-placed crossover material.

C. Dual Triodes

When this program started, there were no data on which to base the design of the 1- and 10-bit shift registers. Also there was no information as to how small the elements could be made without impairment of their operation. To obtain this information and to test and improve the processing, a simple dual triode was designed. Figure 4 shows a microphotograph of a dual triode. The two sections, A and B, are separated by a shield. The width of the films and their separations are about equal and were initially chosen to be 5 mil. Later, dual triodes of a 1-mil geometry were produced, as shown in Fig. 5. The triodes were tested and their characteristics recorded. Since the voltage of gate and shield can be varied independently, three types of measurements are possible, as listed in Table I. Examples of these characteristics are shown in Figs. 6 through 9.

The characteristics shown in Fig. 6 were obtained with triode 224A (1-mil), which was processed at a higher temperature than earlier triodes. The amplification factor μ for this triode is in excess of 20 while that of the earlier triodes, such as 216B in Fig. 7, is in the vicinity of 10. It is possible that the higher temperature process results in a more uniformly low cathode work function. For low values of V_{g+s}, the current going to the shield and gate is negligible; it is plotted in Fig. 6 for $V_{g+s} = 4V$ and attains a maximum value of 1 μA.

The curves in Figs. 7 through 9 were taken on triode 216B to demonstrate the effect of different connections on the triode parameters as indicated in Table I.

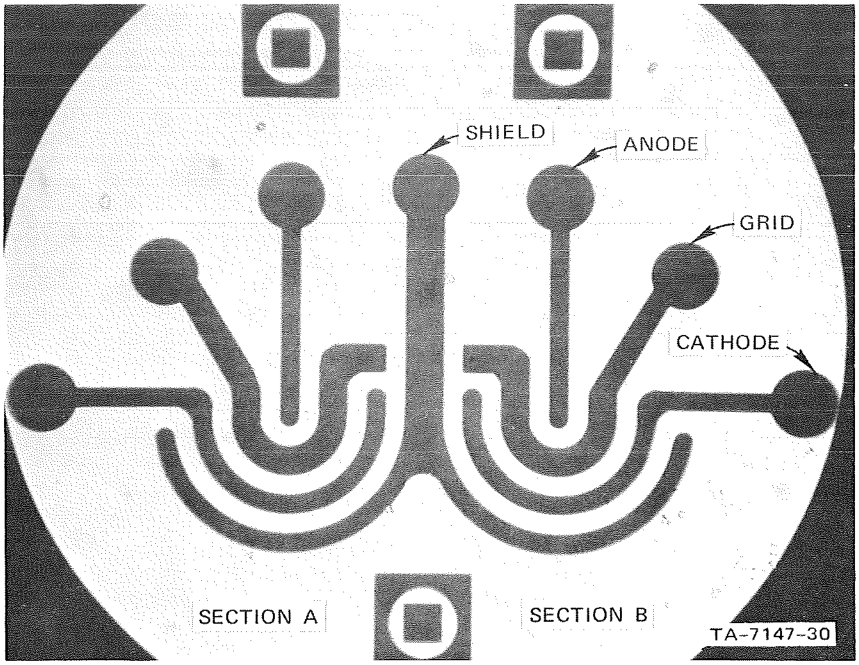

FIGURE 4 MICROGRAPH OF COPLANAR DUAL TRIODE

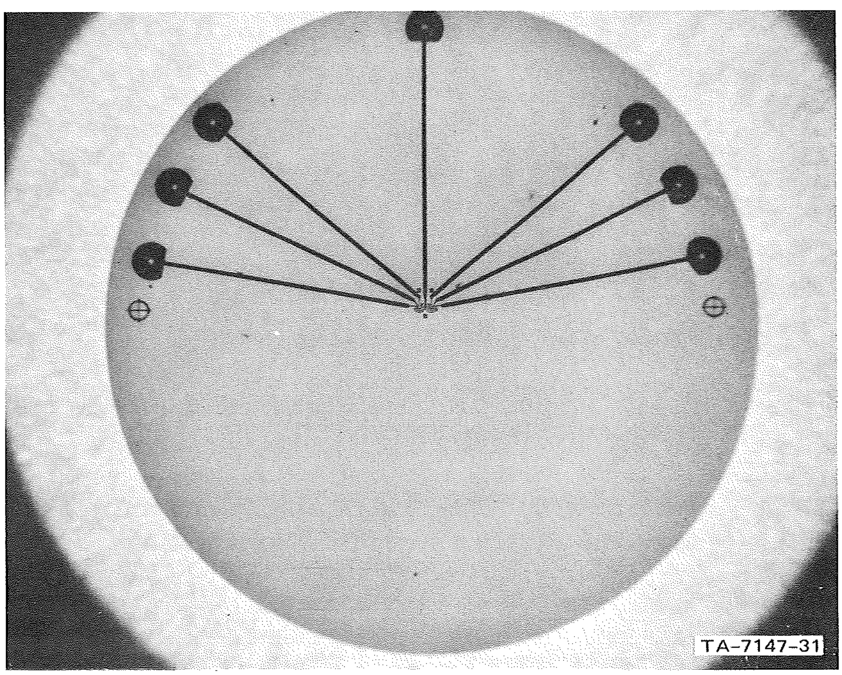

FIGURE 5 PHOTOGRAPH OF A COPLANAR DUAL TRIODE ON A 3/4-inch SUBSTRATE (1-mil geometry)

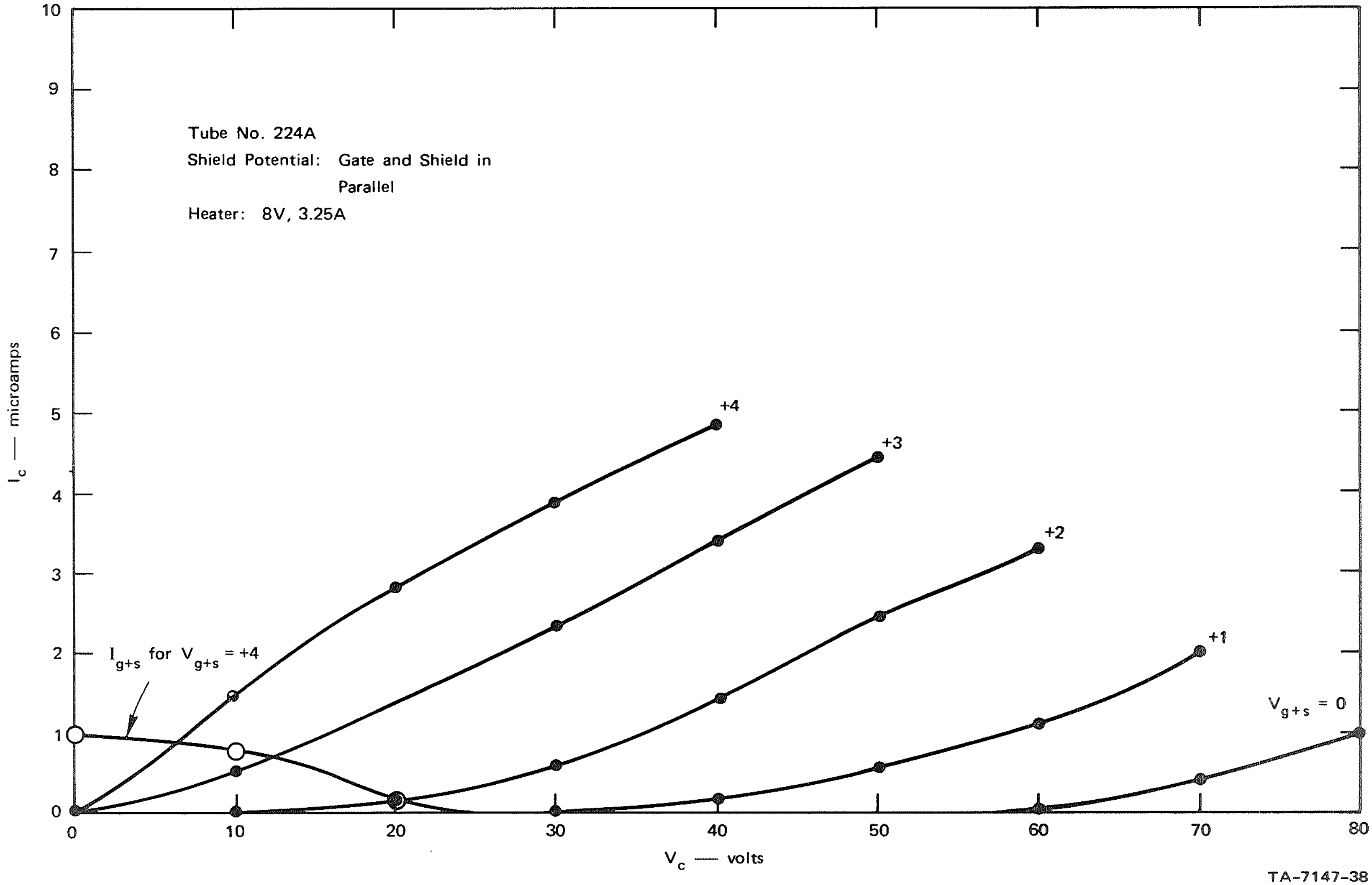

FIGURE 6 CHARACTERISTICS FOR IVC TRIODE 224A

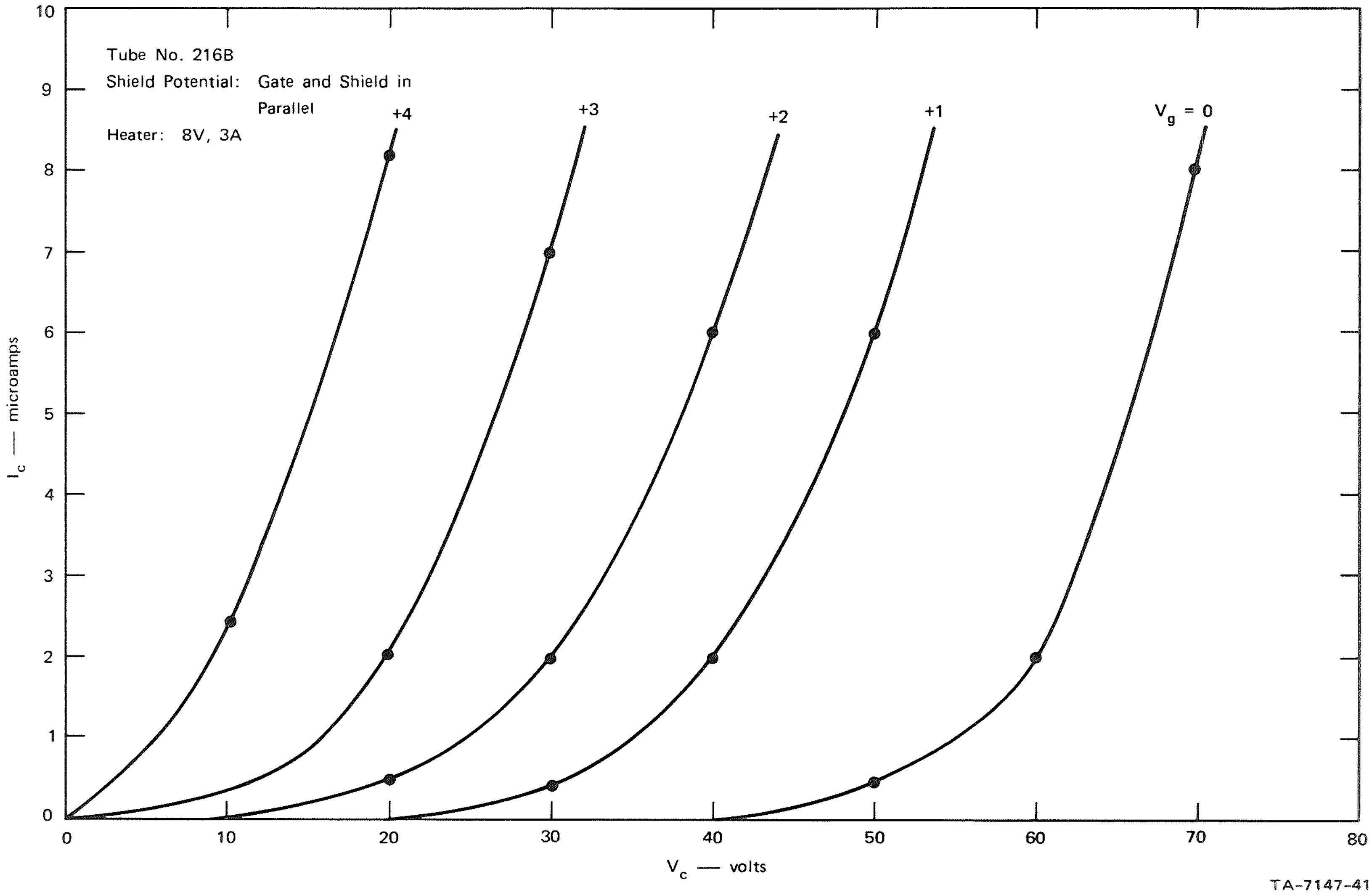

FIGURE 7 CHARACTERISTICS FOR IVC TRIODE 216B

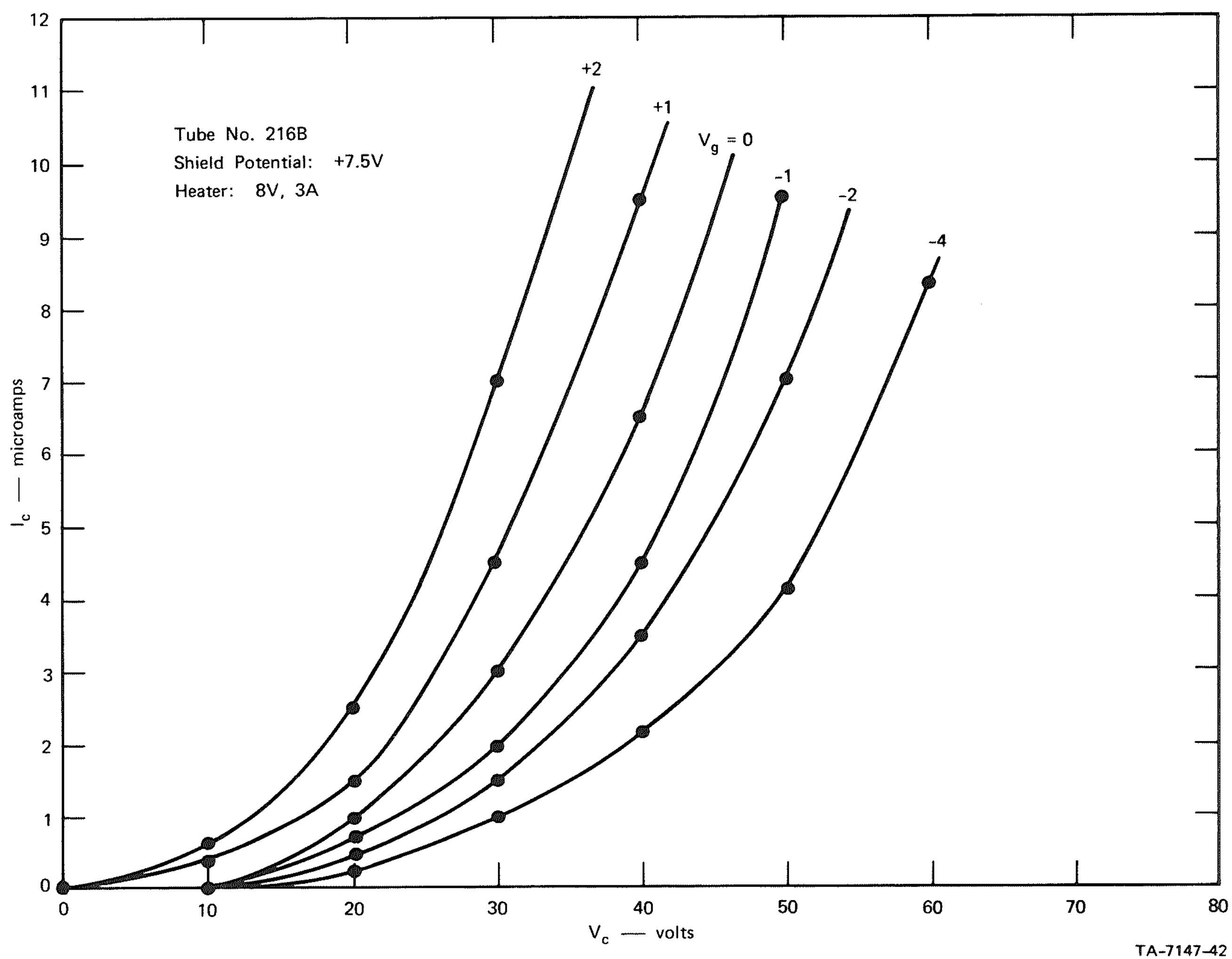

FIGURE 8 CHARACTERISTICS FOR IVC TRIODE 216B WITH V_s = 7.5 V

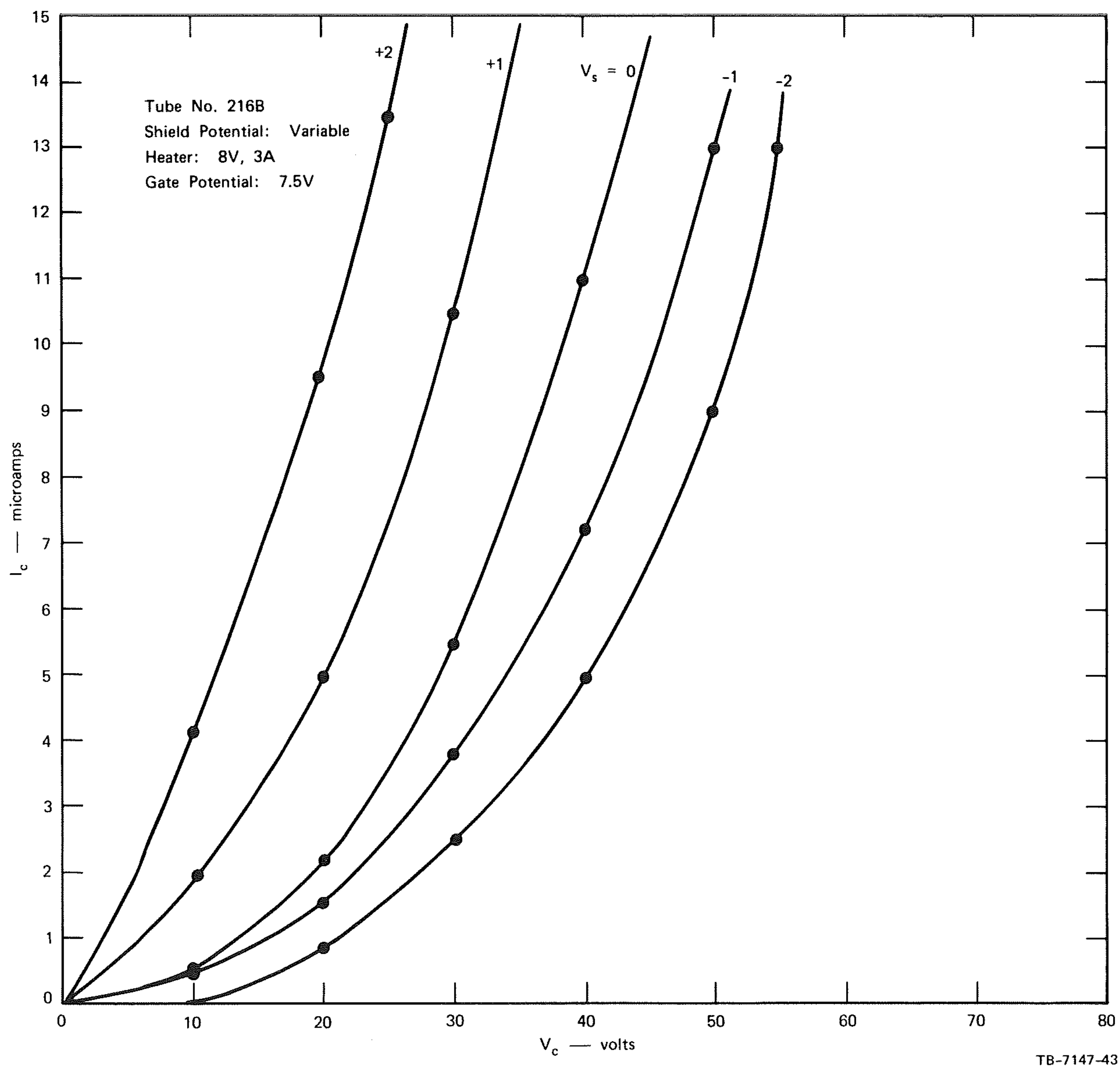

FIGURE 9 CHARACTERISTICS FOR IVC TRIODE 216B WITH V_g = 7.5 V

Table I

COMPARISON OF MEASUREMENTS ON COPLANAR TRIODES

Type of Measurement	Results	Examples
Gate and shield connected in parallel	Triode-like characteristics Highest μ^* values Largest offset voltage	Figs. 6 and 7
Shield potential set and gate potential varied	Triode-like characteristics Lowest μ values Smaller offset voltage	Fig. 8
Gate potential set and shield potential varied	Tetrode-like characteristics Intermediate μ values Smallest offset voltage	Fig. 9

* Amplification factor.

Figures 10 and 11 show measurements made on triodes 221A and 221B and illustrate the similarity of the two triodes on the same substrate. Figure 12 shows the higher currents obtained by operating two triodes, 224A and 224B, in parallel.

Figure 13 presents typical data obtained with a 5-mil geometry triode, designed, fabricated, and tested on another program under commercial sponsorship. It is included here to illustrate the similarity in the characteristics of the 1-mil and the 5-mil triodes.

Several of the dual triodes were put on life test and compared with the life test of two conventional triodes, 147 and 159, with cathodes made of the triple carbonate-photoresist mixture. The conventional triodes have been operating for about 12,600 hours without any serious deterioration. Unfortunately the picture for the dual triodes is somewhat different, as can be seen from Table II where the results from the life test of the triodes are listed. The tubes were tested with the cathode at about 700°C at a collector and gate voltage, V_c and V_g. The first tube listed, 189, operated for a long time at a current range of about 1.5 to 2.0 μamps but during the last few weeks the current has been

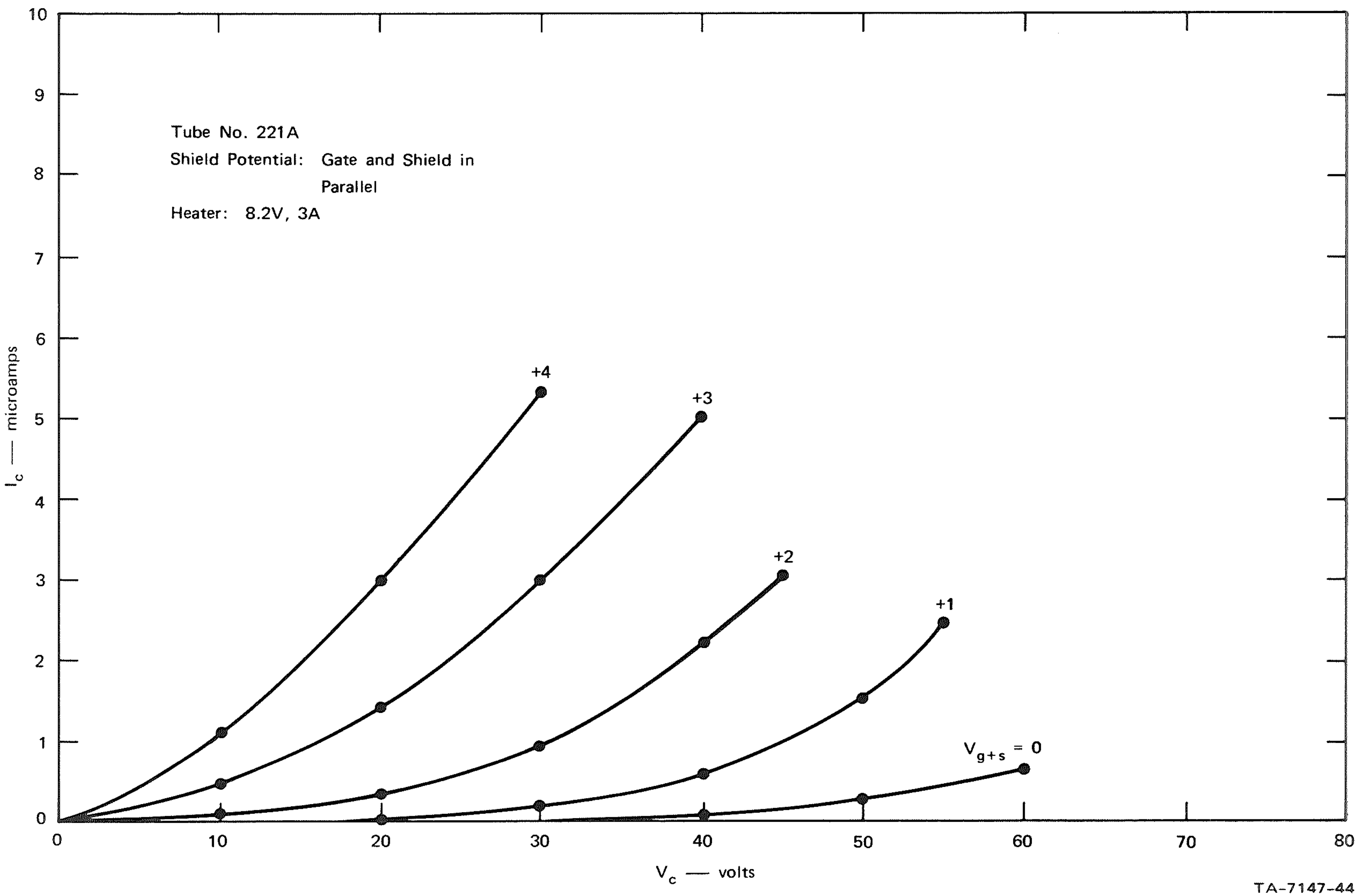

FIGURE 10 CHARACTERISTICS FOR IVC TRIODE 221A

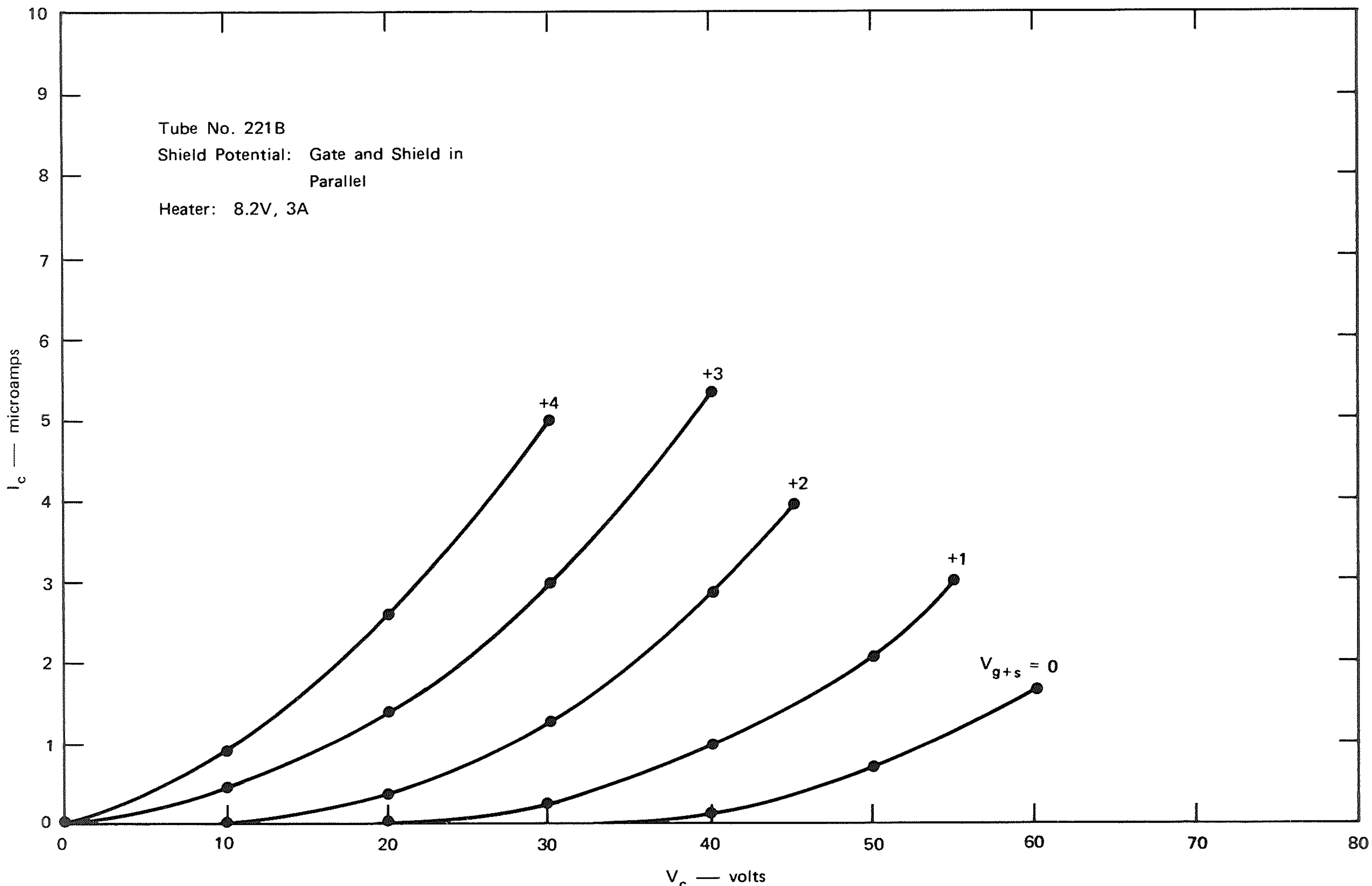

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FIGURE 11 CHARACTERISTICS FOR IVC TRIODE 221B

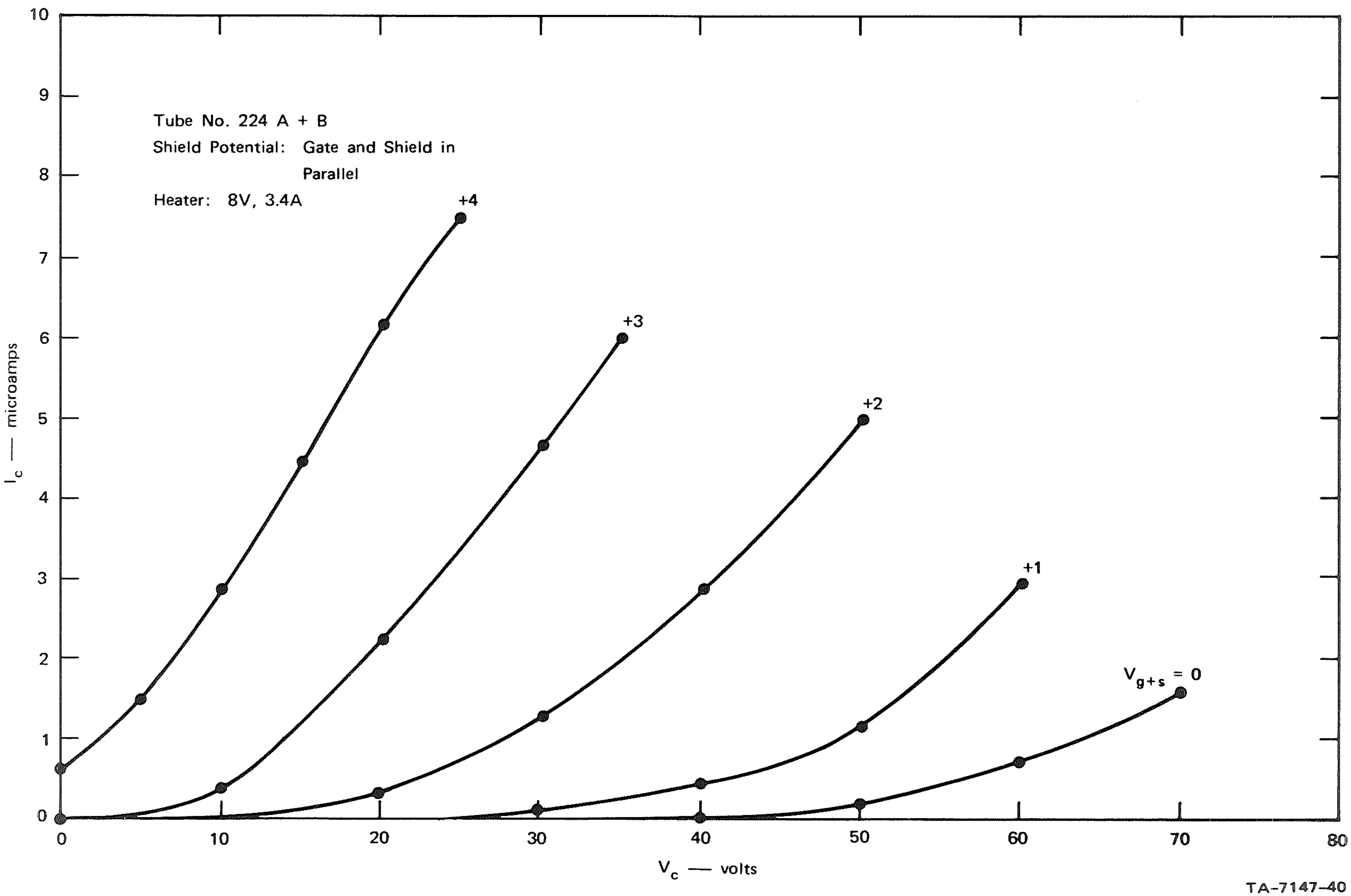


FIGURE 12 CHARACTERISTICS FOR IVC TRIODE 224A AND 224B IN PARALLEL

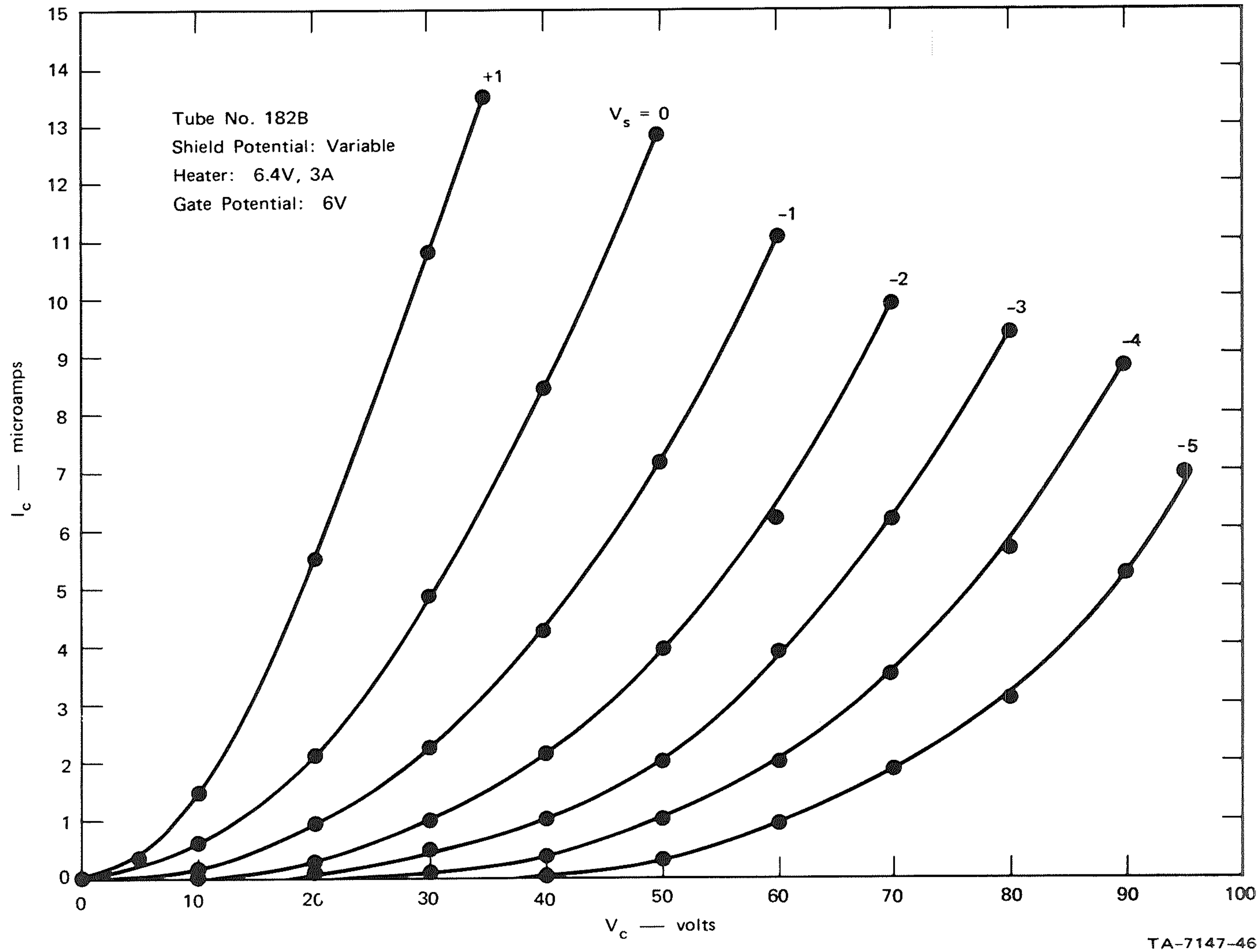


FIGURE 13 CHARACTERISTICS FOR IVC TRIODE 182B (5-mil geometry)

Table II

RESULTS FROM LIFE TEST

Run	Tube	V_c (Volts)	V_g (Volts)	I_c Initial (μamps)	I_c Subsequent (μamps)	Hours Tested	Metal Used	Tube Configuration	Comments
	189	47	6.2	2.4	0.9	8500	Ni-Ti	5-mil dual triodes	One triode only
8-D	216	47	3	6.2	0.1	5000	Mo-Zr	1-mil dual triodes	One triode only
	218	47	3	4.4	0.05	1300			Both triodes in parallel
9-D	224	47	6.2	1.6	0.09	3700	Mo-Zr	1-mil dual triodes	Both triodes in parallel
10-D	227	47	3	1.8	0.18	2700	Mo-Zr	1-mil dual triodes	Both triodes in parallel
	230	47	4.5	0.7	0.4	2700			One triode only
1	237	90	0	400	310	1800	Mo-Zr	5-mil single triodes	On alumina substrate
	238	90	0	660	200	1800			
3	242	47	3	1.2	0.5	500	Ni-Ti	1-mil dual triodes	Both triodes in parallel
	243	47	3	1.2	0.1	500			One triode only

decreasing. Tubes 216, 218, 224, 227, and 230 showed an immediate decline in emission and for most of them the emission is now seriously reduced, but not all to the same degree.

Tubes 216 to 230 were the first tubes produced with the molybdenum-zirconium film. Also they were the first tubes to employ a 1-mil configuration. The poor performance of these tubes, therefore, raised the question whether either or both of these factors was involved in the rapid deterioration. For this reason two tubes, 237 and 238, were built, again using molybdenum-zirconium, but with a different configuration. The tubes were single triodes with a spacing of 5 mils between cathode and gate and 20 mils between gate and anode. Also, the tubes were made on an alumina substrate. Tubes of this type have been built previously using nickel-titanium and two of the tubes, 6 and 7, have been on life test for more than 10,000 hours allowing a good comparison with tubes using molybdenum-zirconium. Tubes 6 and 7 have changed very little during this life test. The emission of tube 6 is now down by about 25%, but tube 7 shows no change. In contrast, the tubes with molybdenum-zirconium, 237 and 238, show some changes already after 1800 hours.

Two 1-mil dual triodes with nickel-titanium, tubes 242 and 243, have been on life test for 500 hours and do not appear to be greatly different from the tubes of molybdenum-zirconium. The results of these tests will be discussed in a later section.

D. 1-Bit Shift Register

The 1-bit shift register was designed to have a 1-mil geometry since it is part of the 10-bit shift register that requires the 1-mil geometry to fit on the sapphire wafer. The design was made by Integrated Circuit Systems Technology and was based on a triode characteristic similar to the one shown in Fig. 7. The circuit diagram and the physical layout of the 1-bit shift register are shown in Figs. 14 and 15. The circuit consists of triodes, diodes, and diode-resistors. The diode resistors are formed by interconnecting the cathode, gate, and shield of the individual triodes. The symbol for a diode resistor is a resistor with an arrowhead pointing in the forward direction.

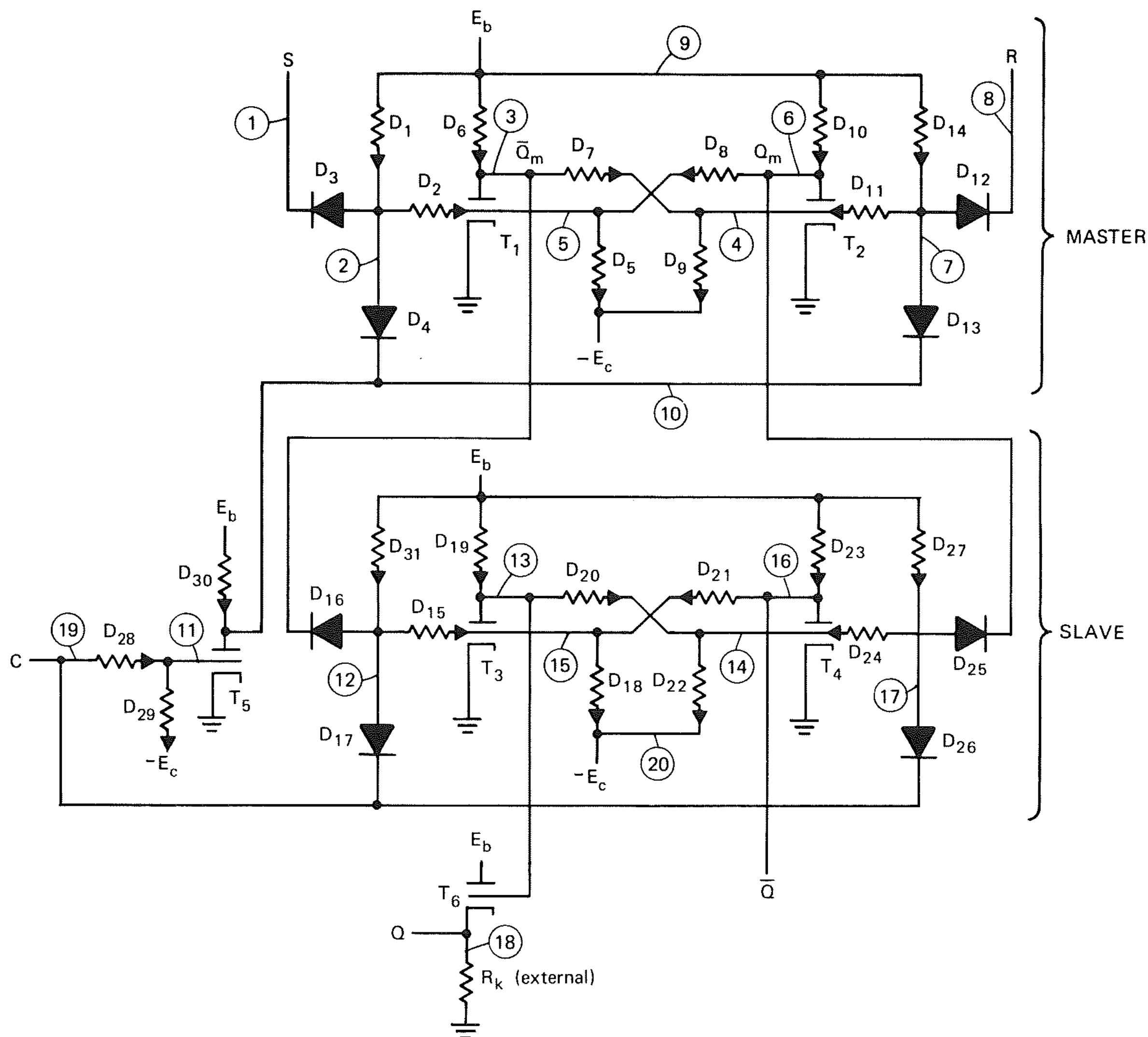


FIGURE 14 CIRCUIT DIAGRAM FOR 1-BIT REGISTER

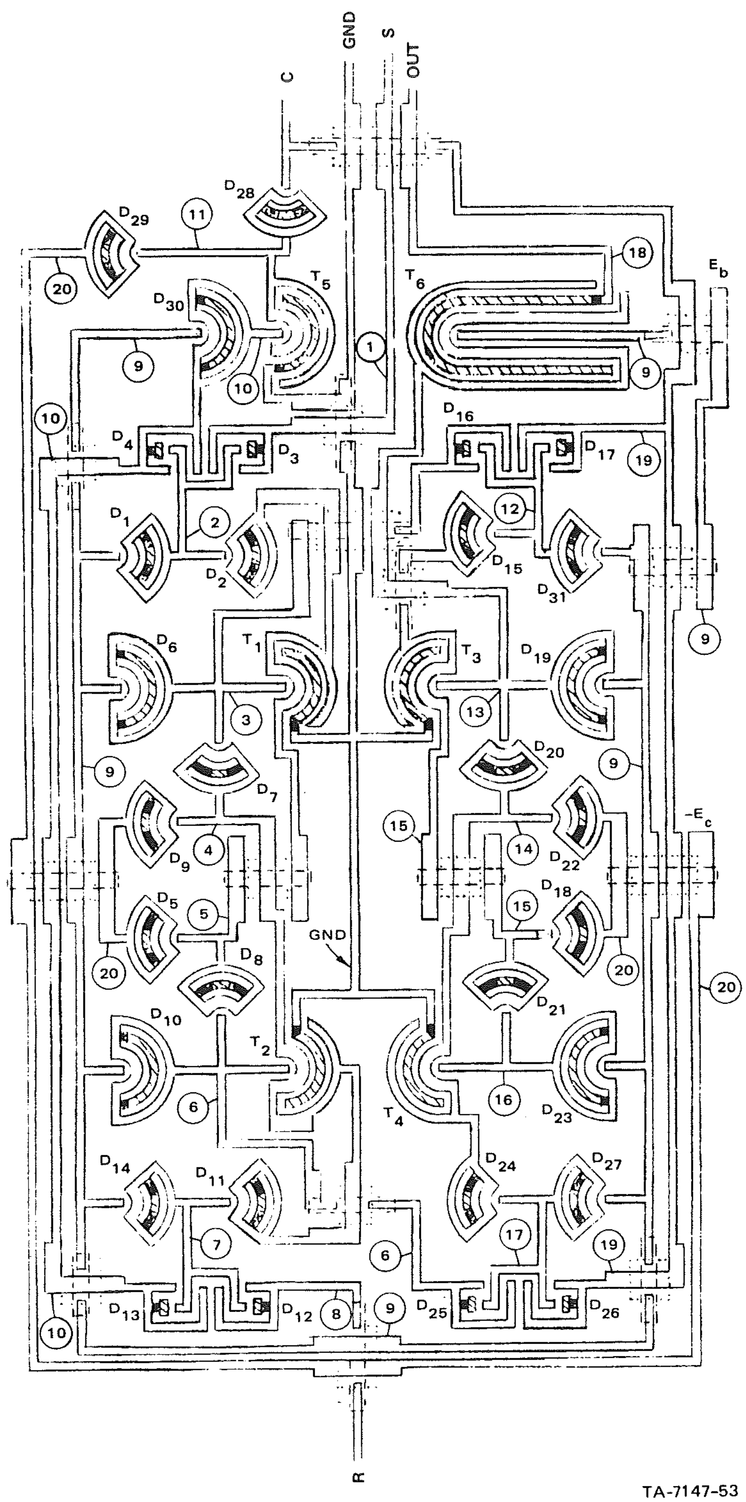


FIGURE 15 LAYOUT FOR 1-BIT SHIFT REGISTER. Cross-hatched areas: triple carbonate-photoresist cathode coating. Solid areas: uncoated tungsten film. Dotted-line areas: dielectric isolation. Dashed-line areas: crossover conductors.

At the time of design of the shift register, the fabrication of the IVCs was based on the use of nickel-titanium which, as a first step, requires the delineation of the whole circuit in tungsten. Figure 16 shows this pattern including the crossovers, but without the crossover metal strips that are sputtered or evaporated on in a subsequent step. Four patterns with connection pads are delineated on each wafer, as shown in Fig. 17, for encapsulation in a ceramic cup, which is the ultimate goal of packaging the IVCs.

The 1-bit shift register discussed above is a relatively complicated circuit for testing of a new technology, and the design of the circuit is based on characteristics of a single tube with very little information as to the reproducibility of the characteristics and the interaction of the tubes. The chances for such a circuit to work the first time it is tested are remote, considering the large number of active elements and crossovers. Thus, the question arises how to obtain enough information to evaluate the circuit in case of initial failure. A single 1-bit shift register requires seven connections, plus two for the heater. This leaves two pins of an eleven-pin tube for testing, which is inadequate. Clearly for this reason alone the 1-bit shift register must be broken down in smaller test circuits to allow evaluation but there are also other reasons as discussed below.

E. Test Circuits for the 1-Bit Shift Register

Ideally the testing of a circuit consists of methodically probing from input to output, disconnecting initially any complicating part of the circuit until the fundamental part is understood. The other parts are then reconnected step by step. Such an approach is not feasible here since it would be extremely difficult to test and connect parts of the circuit step by step. After activation of the cathodes, the circuits can no longer be exposed to air. We have therefore chosen to produce significant parts of the circuit separately for testing and analytical evaluation. If the reproducibility of the parts is good, it should then be possible by analysis to determine the operation of the circuit produced as a whole. In dividing the 1-bit shift register, we have selected parts that by themselves are circuits of practical importance. These parts are: an inverter, two inverters in cascade, and a NOR gate.

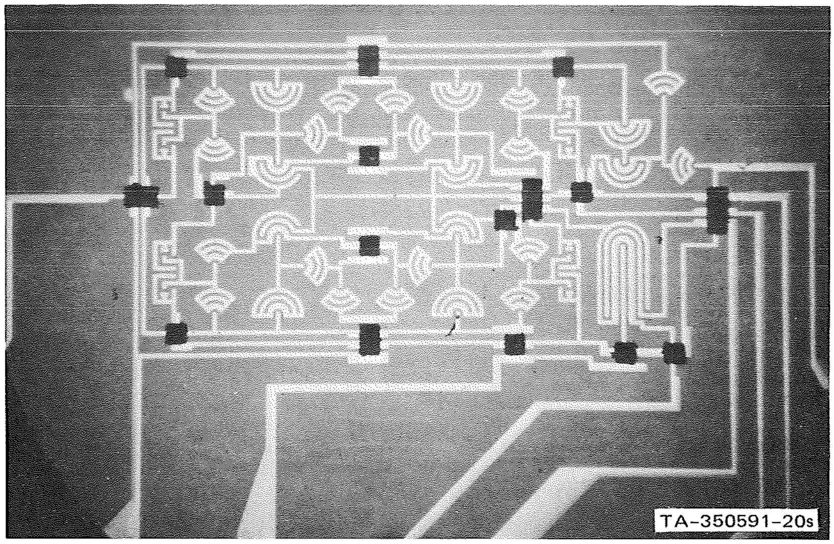


FIGURE 16 1-BIT SHIFT REGISTER OF 1-mil GEOMETRY DELINEATED IN TUNGSTEN. The dark areas are crossover material.

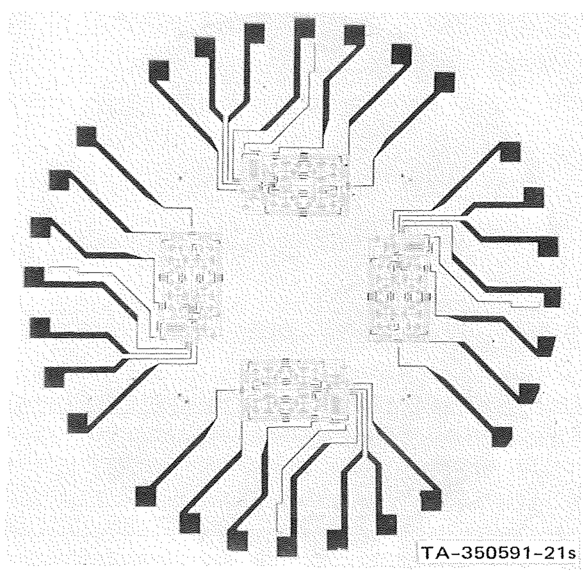


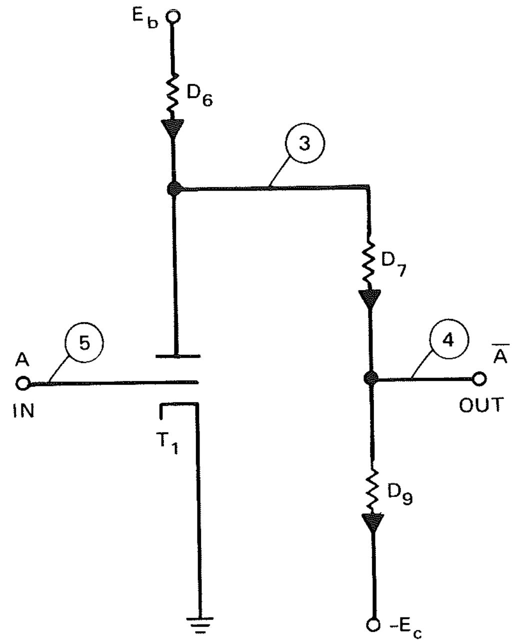
FIGURE 17 FOUR 1-BIT SHIFT REGISTERS ON A 3/4-inch SAPPHIRE WAFER

1. Analysis

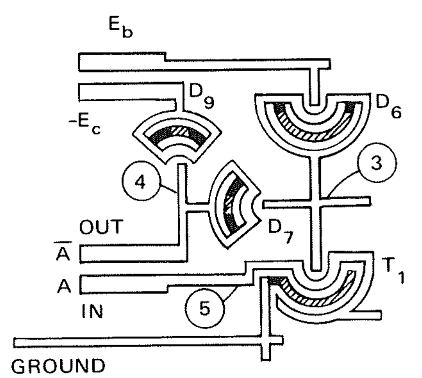
The test circuits are examined by graphical analysis.Such an analysis is adequate for simple circuits but for more complex circuits, the use of computers is necessary owing to the complexity of the nonlinearity of the IVCs.

a. Inverter

To start, we have chosen the inverter. The circuit diagram and layout of the inverter are shown in Fig. 18, which is a modular portion of the 1-bit shift register shown in Figs. 14 and 15. Before getting into the graphical analysis it should be noted from Fig. 18(b) that D_6 and T_1 have similar geometries, that the geometries of D_7 and D_9 are identical, and that the emitter area of D_7 (or D_9) is about 1/6 that of D_6 (or T_1). Consequently, the I-V characteristic of D_6 is the same as that of T_1 at a gate voltage $V_g = 0$, and the I-V characteristic of D_7 (or D_9) is obtained from that of D_6 by dividing each I value by 6. These characteristics are shown in Fig. 19(a). Referring to Fig. 18(a), we next construct in Fig. 19(b) the composite I-V characteristic of D_7 and D_9 in series (by doubling the voltage at each I value) and shift it to the left by E_c volts. The latter represents the I-V characteristic of D_7, D_9, and $-E_c$ in series. This characteristic is then added in parallel to T_1 by adding currents at each voltage value for each gate potential. The load line due to D_6 is now superimposed by flipping over the I-V characteristic of D_6 and shifting it to the right by E_b volts. Assuming, for example, that the input gate voltages are $V_g = +4V$ for A = 1 (T_1 is on) and $V_g = -4V$ for A = 0 (T_1 is off), the intersection points of the D_6 load line with the corresponding I-V curves yield the voltages and currents of D_6, the voltages of T_1, and the currents of D_7 and D_9 for the on and off states. Using these results in Fig. 19(a), we determine the currents of T_1 and the voltages of D_7 and D_9 for the two states. Note that, as expected, the output voltage $V_{out} = V_{T_1} - V_{D_7}$ is negative for $V_{in} = +4V$ (T_1 is on) and positive for $V_{in} = -4V$ (T_1 is off). Proper design requires that $V_{out,on} = V_{in,off}$ and $V_{out,off} = V_{in,on}$, and hence may require a few iterations.



(a) CIRCUIT DIAGRAM



(b) LAYOUT

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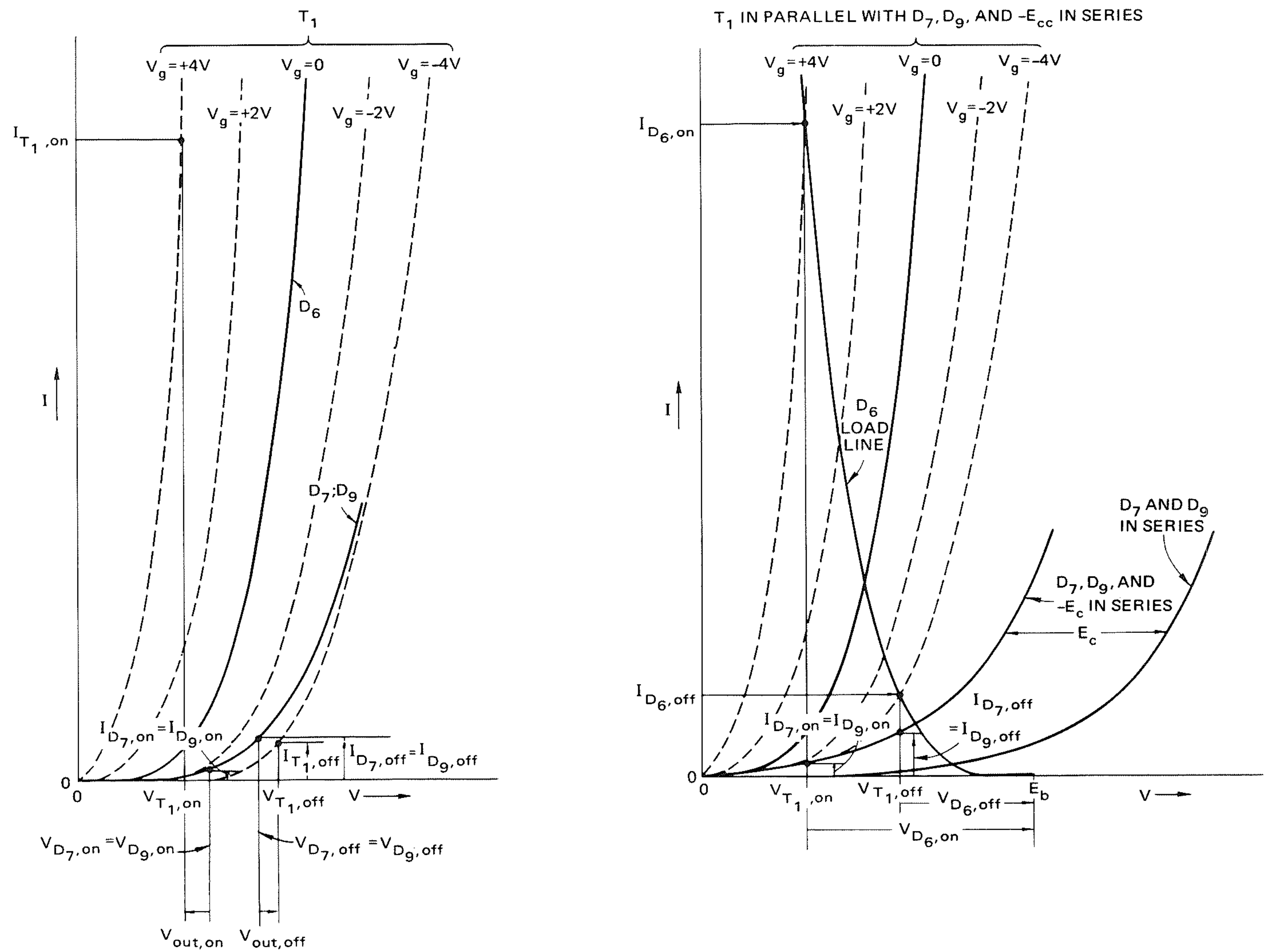
FIGURE 18 AN INVERTER



(a) I-V CHARACTERISTICS OF INDIVIDUAL TUBES

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(b) DETERMINATION OF OPERATING POINTS IN ON AND OFF STATES FROM I-V CHARACTERISTICS OF TUBES IN SERIES AND IN PARALLEL

TA-7147-52

FIGURE 19 GRAPHICAL DESIGN OF INVERTER

b. Two Inverters in Cascade

The next step is to connect two inverters in cascade, as shown in Fig. 20. The circuit and corresponding layout, Fig. 20(a) and (b) are extracted from Figs. 14 and 15, respectively. Crossover insulation and conductors are necessary for the $-E_c$ and output terminals as indicated in Fig. 20(b).

Assuming that the gates of T_1 and T_2 draw no current, the graphic analysis in Fig. 19 is also valid for each inverter stage in Fig. 20.

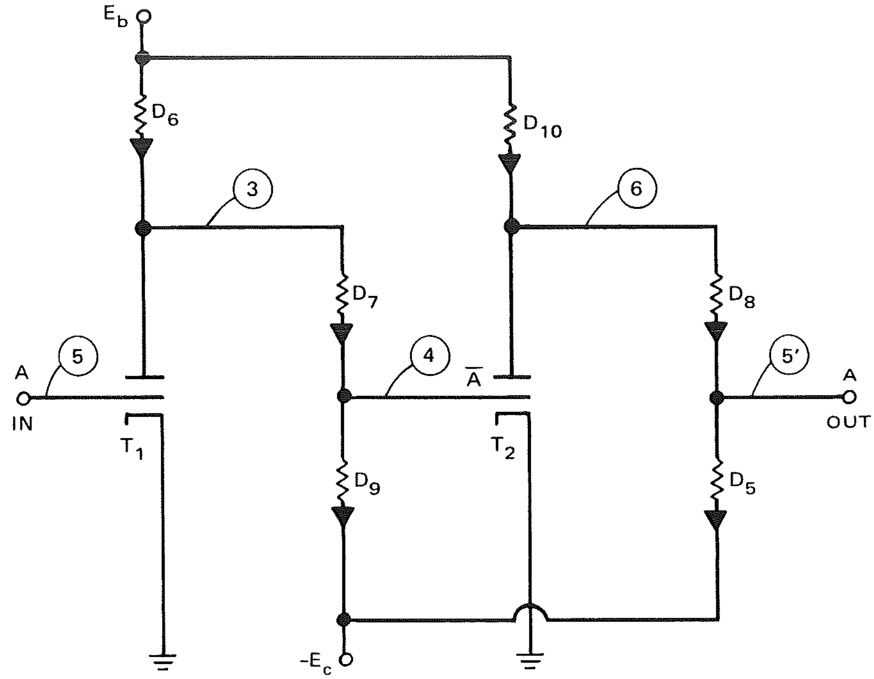
c. NOR Gate

The circuit diagram and layout for a NOR gate are shown in Figs. 21(a) and (b), respectively. Again, these may be identified as portions of the 1-bit shift register in Figs. 14 and 15. The terminal of triode T_3, designated by Node 13, requires an added connection to Node 3.

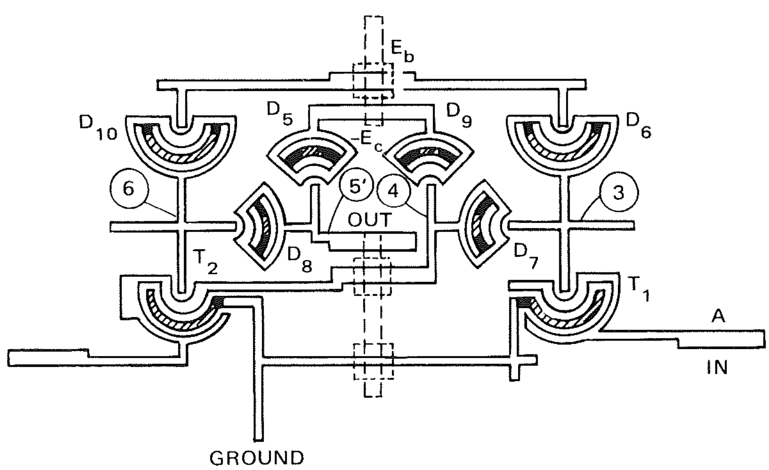
The graphic design of the NOR gate is identical with that of the inverter in Fig. 19, except that the I-V characteristics of T_1 are replaced by equivalent ones for T_1 and T_3 in parallel. The latter are obtained by adding the corresponding currents at each anode voltage, depending on the states of T_1 and T_3. If both are on, then the current of (for example) the + 4V characteristic is doubled at each value of anode voltage; if both are off, then the current of (for example) the - 4V characteristic is doubled; if one tube is on and the other off, then the current of the - 4V characteristic is added to that of the + 4V characteristic. In the example of Fig. 19, the contribution of the - 4V characteristic is negligible.

2. Experimental Results

Two inverters (tubes 244 and 247) and two NOR gates (tubes 248 and 249) were processed and examined. Figures 22 and 23 show the fabricated inverter and NOR gate of tubes 244 and 248. The masks for the photolithographic processing of these circuits were obtained from the mask for the 1-bit shift register by blanking out the unwanted parts of the circuit. At the time of fabrication of the masks, the effect of reducing



(a) CIRCUIT DIAGRAM



------ Dotted line area is dielectric insulation.

— — — Dashed line area is crossover conductors.

(b) LAYOUT

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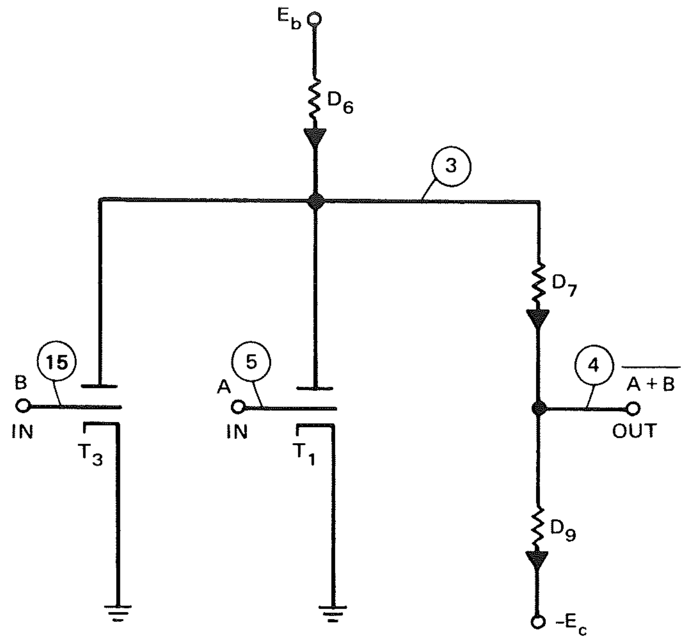
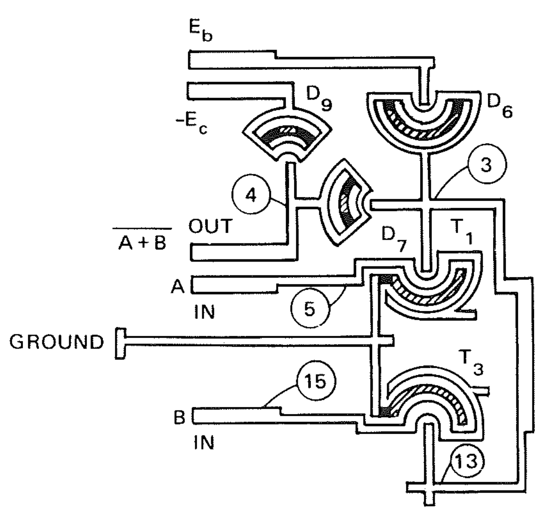
FIGURE 20 TWO INVERTERS IN CASCADE



(a) CIRCUIT DIAGRAM



(b) LAYOUT

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FIGURE 21 A NOR GATE

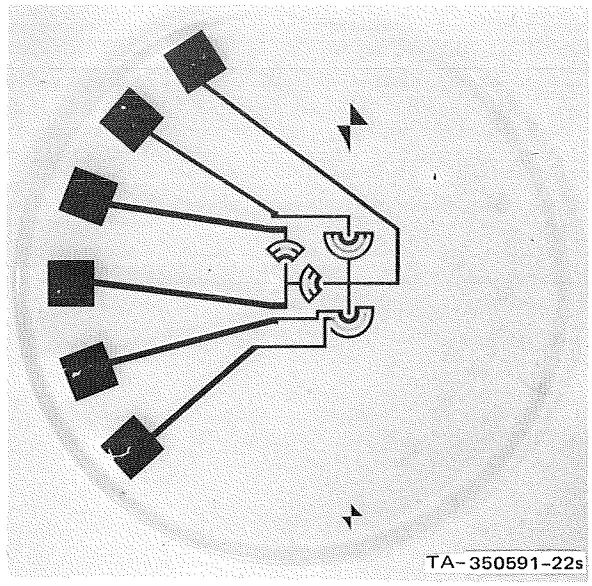

FIGURE 22 PICTURE OF AN INVERTER

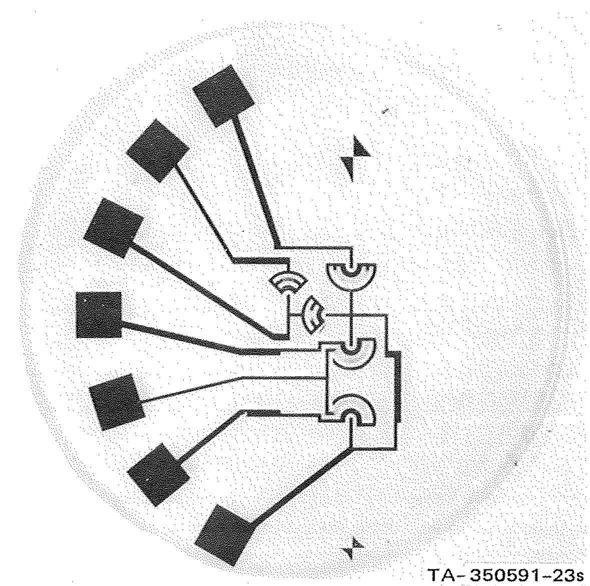

FIGURE 23 PICTURE OF A NOR GATE

the spacing between the cathode, grid, and collector was still not known and it was therefore decided to retain the 5-mil geometry.

The tubes were activated following normal activation procedures monitoring the forward and reverse current of one of the triodes or diodes. During activation of tube 244, the alumina wafer broke, causing an instantaneously high, local overheating. This caused evaporation of a thin film of barium over most of the circuit giving the metal films a yellow cast. The film of barium caused excessive leakage that disappeared when the tube was opened to air because of the oxidation of the barium. The activation of the other tubes proceeded without problems.

In general, the emission of the diodes and triodes was good. However, it was not possible to get any of the circuits completely operating because of a strong emission in the reverse direction of all elements except diode D_6. Figs. 24 and 25 show the forward and reverse current-voltage characteristics of diode D_9 of the NOR gate (tube 248). Both show improved emission with time which is normal for the cathodes during the first few days. Diode D_7 of the same circuit had similar characteristics except that, in this case, it was the forward and not the reverse current that showed a threshold below 140 μA.

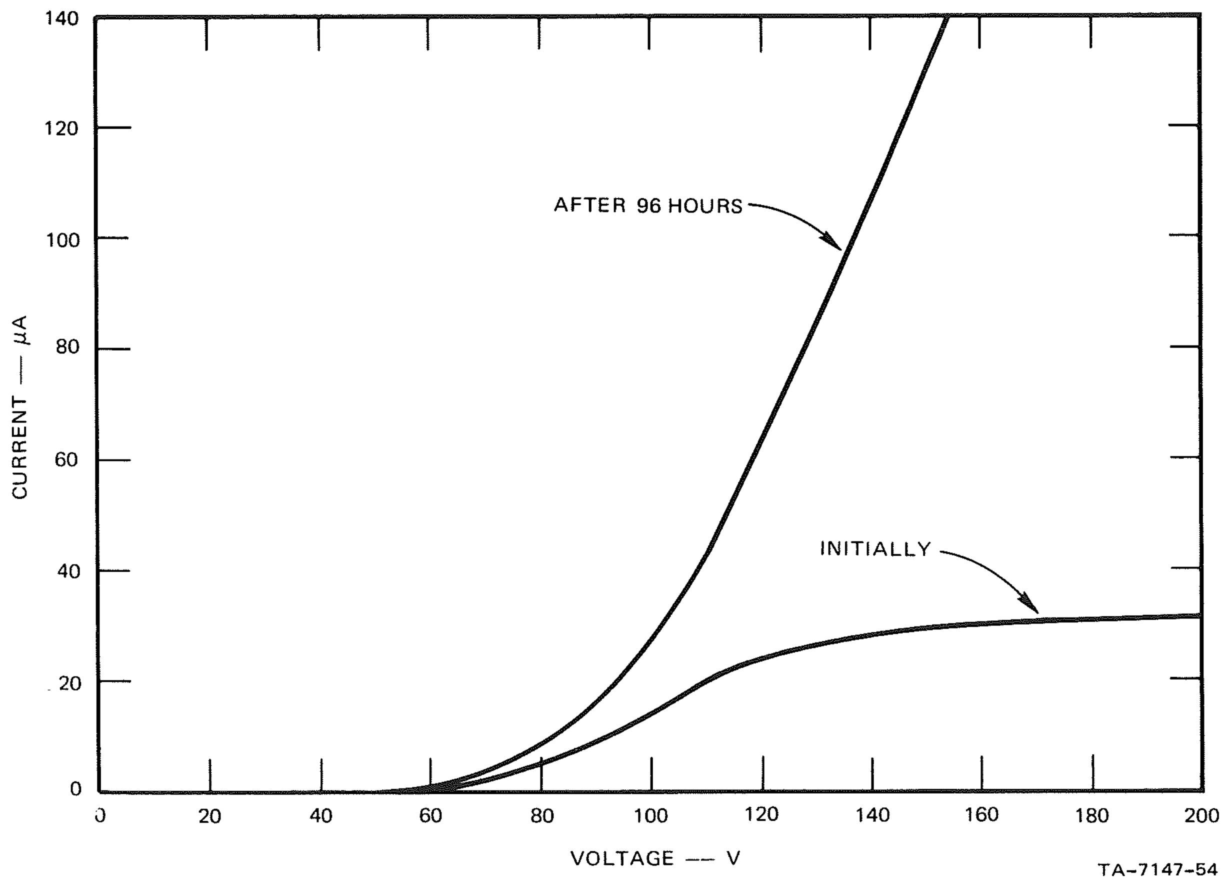

FIGURE 24 FORWARD CURRENT-VOLTAGE CHARACTERISTICS OF DIODE D_9 OF NOR GATE (Tube 248)

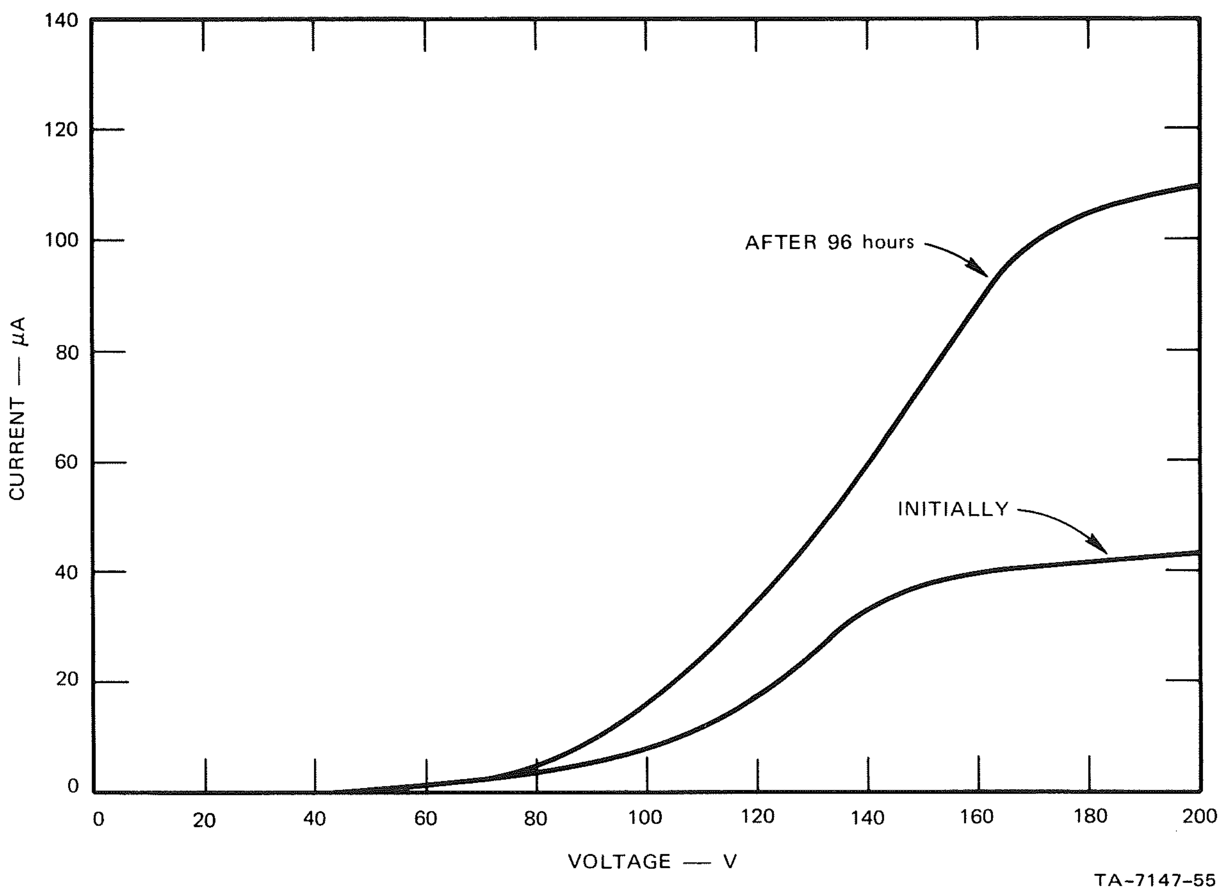

FIGURE 25 REVERSE CURRENT-VOLTAGE CHARACTERISTICS OF DIODE D_9 OF NOR GATE (Tube 248)

F. Discussion of Results

The two problems that have not been solved so far are the reverse emission and the short lifetime of the diodes and triodes. The reverse emission is associated with movement of the activation material (or part of it) during the activation process. The elements of the four circuits produced so far all have emission in the reverse direction with the exception of diode D_6, the only diode with a collector not connected to any cathode. Thus the activation material must spread along the connecting metal films. To verify this point the two triodes T_1 and T_3 were tested in parallel, first the reverse current between grid and cathode and then the reverse current between anode and cathode. Reverse emission was observed only in the second case, in agreement with the fact that only the anodes are connected by metal films to a cathode.

These results may be surprising at first glance because of the short period of time that the substrates are kept at activation temperature (approximately 900°) and the considerable distances the activation material must travel during this period. However, it should be kept in mind that at these temperatures the barium is molten and may run along the metal films at a relatively rapid speed. Evidence of molten barium is clearly seen in Fig. 26, which shows small droplets on the tungsten film next to the activation material. These droplets are presumably of barium since molten barium is known to exist during activation of a cathode, it being a very important part of the activation process as described earlier. However, there is no clear evidence of barium droplets on the molybdenum-zirconium films, but it may be there in form of a very thin invisible film or the barium may have run along the edges of the film.

The exact process responsible for the reverse emission is not known at this time but a few conjectures can be made. The molybdenum-zirconium film is exposed to air before the substrate is mounted in the tube so part of the zirconium is oxidized. When barium is present, part of the zirconium oxide (ZrO_2) is reduced and barium oxide (BaO) is formed until a certain equilibrium is reached. The barium oxide may then be

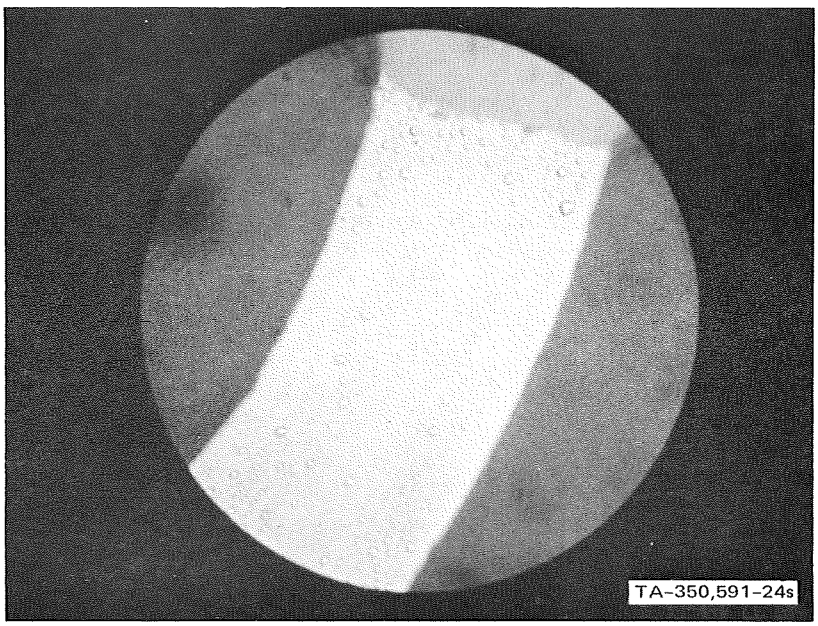

FIGURE 26 EVIDENCE OF MOLTEN BARIUM ON TUNGSTEN FILM NEXT TO CATHODE. The cathode is seen at the top.

coated with some of the excess barium forming a good emitter. The amount of barium necessary for such a process would be very small since only a coating of a few monolayers is necessary. In fact too much barium would reduce the emission and this may be the reason why the diode D_6 of tube 244 did not show any reverse emission. In this tube we had a film of more than 100 Å of barium over the entire circuit as a result of the overheating described previously. Unfortunately, the other diodes in tube 244 were not checked before the tube was opened to air because the excessive current leakage made evaluation difficult and because we were unaware of the problem at that time. These diodes should also have shown lack of reverse emission.

The amount of barium available for transport along the metal films is presumably proportional to the amount of activation material applied to the cathodes. Three layers of activation material were applied to the 5-mil circuits discussed so far. However, in some of the 1-mil dual triodes put on life test, only one layer was applied. Several of these tubes on life test were open after their emission was considerably reduced. There was no evidence of molten barium as shown in Fig. 26 but the cathodes had quite a different appearance. They were black compared to the white color of the cathodes of the 5-mil structure, which indicates a breakdown of the cathode material.

It is interesting to note that the reverse emission for most of the elements improved with time as did the emission from the cathodes, presumably due to migration of the barium. Since the entire wafer is heated to the temperature of the cathodes, the barium can slowly migrate along the metal films and thus cause the short lifetime. So far there is no evidence of migration of barium across the sapphire surface. Unfortunately, the results from the life test give little information about what goes on in the tube during operation. The value of the life tests made so far is limited because of the small number of tubes tested and the lack of information on reproducibility of the tubes. The results from the life test presented in Table II are confusing. Comparing tubes 237 and 238 with tubes 6 and 7 (not listed in the table) that were made from nickel-titanium and have been operating for more than 10,000 hours without serious deterioration, the test would indicate that the problem is associated with the use of molybdenum-zirconium. On the other hand, the use of nickel-titanium on sapphire, tubes 242 and 243, shows that nickel-titanium is no better than molybdenum-zirconium, at least not on sapphire. Whether the type substrate is also a factor to be considered is not known. Concerning the effect of the geometry, 1-mil versus 5-mil, the results in Table II show no marked effect except that the 1-mil structures appear to deteriorate a little more rapidly.

If the migration of barium on the metal film is the major problem associated with the short lifetime, one would expect that the ratio of cathode area to the area of metal film connected to the cathode is important. The smaller this ratio, the higher is the percentage of barium conducted away from the cathode. For tubes 6 and 7, the ratio is about one-half but for tube 189 it is much lower. However, for this particular tube only the area around the cathodes appears to be at operating temperature. Since the rate of migration is a function of temperature, the effective ratio would be higher. If there is any truth to this argument, one would expect the 1-mil configuration that has the lowest ratio of them all to deteriorate more rapidly than the 5-mil structure. The life tests made so far support this point of view.

G. Conclusions and Recommendations

The processes used for fabrication of the IVCs are all well developed individually. However, the combination of the thin film and photolithographic techniques with the production of coplanar cathodes is entirely new and the question arises to what degree these techniques are compatible. The activation of the cathodes, which follows the normal thin film technique, is crucial in that it exposes the thin films to quite high temperatures ($\sim 900^{\circ}C$) and possible chemical and physical interactions not normally encountered in other applications. The activation process and the conditions under normal operation in a regular tube are very localized, i.e., only the cathode is heated while the rest of the tube is at a much lower temperature. In the IVCs the cathodes and the rest of the circuit are all at the same temperature, not only at activation but also during the normal operation. This factor makes some severe demands on the adhesion of the thin films to the substrate and on their ability to withstand chemical or physical attacks during activation. By selecting metals that react with the substrate, the adhesion problem appears, at least at this point, to have been solved but the processes occurring during activation of the cathodes are still a problem. The results of this investigation indicate that the parts of the films connected with the cathode areas are all activated by a flow of molten barium along the metal films. The exact process occurring has not been completely identified but the experimental results show that any anode connected with a cathode through a metal film becomes an emitter. Thus it is clear that very detailed and simple experiments must be performed to solve this problem before any complicated circuits can be attempted.

In addition, there is a problem concerning the life time of the cathodes. While cathodes of conventional tubes, made from the mixture of triple carbonate and photoresists used in this investigation, show no deterioration after more than 12,000 hours, the coplanar cathodes associated with the thin film circuitry have a very short lifetime. This may be due to a slow migration of barium across the metal films but, just as above, detailed measurements are needed for identification and solution of this problem. It is believed that this problem can be overcome by the proper selection of compatible materials and processing conditions.

Finally, to obtain complicated IVC circuits, crossovers of metal conductors are necessary to reduce the size of the circuits. It is believed from the experiments with the mixture of CaO, MgO, and Al_2O_3 that this problem can be solved without too much difficulty. Otherwise, the techniques of building metal bridges that now are employed frequently in microminiature semiconductor circuits should be considered.