# QUARTERLY REPORT NO. 3

## PRODUCTION ENGINEERING MEASURE
## ON
## 2N1708 SILICON PLANAR EPITAXIAL TRANSISTOR

CONTRACT NO. DA-36-039-SC-86729

FOR

U.S. ARMY ELECTRONICS MATERIEL AGENCY
PHILADELPHIA, PENNSYLVANIA

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ABSTRACT

Engineering Samples

The third group of twelve engineering samples was shipped to the Signal Corps on November 1, 1962. Electrical data for these units is provided.

Device Processing

Mechanically polished substrates, which have been subjected to a slight chemical etching, have been introduced into the process. This change has reduced striations which appear on the surface of epitaxial wafers.

An improved method of substrate cleaning has been developed which insures that the cleanest possible surface enters the system. Also, a technique of etching the wafers with anhydrous HCl in the epitaxial tube prior to epitaxial deposition was incorporated into the process. In addition, an improved method of measuring resistivity of the epitaxial layer is in operation.

The oxide on the surface of the wafer serves as a mask during the diffusion operation and generally serves to protect the silicon surfaces during wafer processing. In performing these functions, however, impurities may be diffused into the oxide at the high diffusion temperatures. A method was developed providing the cleanest possible oxide to improve the surface protection on the finished pellets. This was accomplished by removing the oxide after the final high temperature diffusion and replacing it with oxide grown under carefully controlled conditions. Experiments are also being made on baking of oxide to diffuse entrapped water molecules to the surface for subsequent evaporation.

Also, experiments are being run in the photoresist area to evaluate the use of hot concentrated sulphuric acid with chromic acid as a means

of removing polymerized photoresist. An additional photoresist step is being evaluated to form contacts which may reduce contamination. Filtering of chemicals under controlled conditions has been established as an important process improvement.

Investigations performed in the Advanced Development Laboratory on surface preparation and cleaning are completed. Important sources of impurities which are organic in nature are discussed as follows:

a. Lapping oils

b. Waxes and hydrocarbon

c. Vacuum grease

d. Teflon

e. Hard plastics

f. Polyvinyl chloride plastic

g. Solvents

An improved phosphorous diffusion technique is in use utilizing a $POCl_3$ source instead of $P_2O_5$.

Extensive experiments were conducted to develop a gold contact to replace aluminum. Results of initial tests indicated gold to be comparable or better than aluminum contacts. However, when units were subjected to high temperature and power tests, excessive failures occurred. Failure analysis disclosed that severe discoloration of the contacts was present and the connector wire had physically separated from the contact area.

In the mounting area, improved wetting, of a lower mounting temperature, has been achieved thru the use of a gold alloy which has been evaporated on the back of the wafers. A temperature profile for mounting has been established and process control procedures have been incorporated to insure that control is maintained. An evaluation is currently being

performed on devices mounted on ceramic stems.

Effort in bonding was curtailed awaiting results of the gold contact experiments. Due to the poor results obtained with these experiments, increased emphasis will be placed on bonding with gold alloy wire.

In the sealing area, initial experiments have been performed to control moisture in the shell using a desiccant. Results were poor. However, experiments are continuing with other types of desiccants.

Process control procedures are in effect for the automatic test equipment. Experiments have indicated, with a high degree of confidence, that the equipment itself does not cause unit failures.

Inspection and Quality Control

An Inspection and Quality Control Manual is being prepared. The format being followed is outlined in the report.

## I. PURPOSE

The objective of this contract is to improve the reliability of the RCA 2N1708 epitaxial, planar, silicon switching transistor. The failure rate goal, at a 90 percent confidence level, is 0.0011 percent per thousand hours at a junction temperature of 50°C.

To attain this objective, design and process improvements will be evaluated and put into effect. A system of process controls will be established.

Various reliability tests will be performed. These will include: step stress tests; matrix tests; temperature and/or power aging tests; life tests; and mechanical and environmental tests.

A 9 to 12 month development period followed by a 6-month production phase will be undertaken. During this latter period, life testing will be employed to establish the actual failure rate of the improved 2N1708.

## II. DELIVERY OF ENGINEERING SAMPLES

State-of-the-art samples were shipped to the U.S. Army Supply Agency as indicated below:

| NUMBER | DATE OF SHIPMENT |
|---|---|
| 12 | 1 June 1962 |
| 12 | 15 August 1962 |
| 12 | 1 November 1962 |

The electrical characteristics of the 12 samples shipped 1 November 1962 are shown in Table I. The electrical characteristics of the 12 samples shipped 1 June 1962 were provided in Quarterly Report No. 1 and the 12 samples shipped 15 August 1962 were provided in Quarterly Report No. 2. The 2N1708 Electrical Specifications are shown in Table II.

TABLE I

RCA 2N1708 STATE OF THE ART SAMPLES, NOVEMBER 1, 1962

| | $I_{CBO}$ | | $I_{CEX}$ | $BV_{CBO}$ | $BV_{EBO}$ | $BV_{CEO}$ | $V_{BE}$ | $V_{CE}$ | | $C_{OB}$ | $h_{FE}$ | $h_{fe}$ | $T_S$ | $T_{ON}$ | $T_{Off}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 25°C μa | 150°C μa | μa | v | v | v | 10ma v | 10ma v | 50ma v | $p_f$ | 10ma | 100mc | nsec | nsec | nsec |
| 2. | .0019 | 010.0 | 10.3 | 76.7 | 7.49 | 34.7 | .798 | .171 | .320 | 3.5 | 49.5 | 3.5 | 16.5 | 22.0 | 51.0 |
| 6. | .0022 | 005.4 | 09.4 | 61.8 | 7.80 | 23.7 | .808 | .164 | .317 | 4.3 | 49.5 | 3.88 | 12.0 | 24.5 | 42.0 |
| 9. | .0017 | 005.2 | 09.7 | 65.3 | 6.89 | 25.8 | .774 | .157 | .254 | 4.1 | 50.0 | 3.64 | 12.5 | 23.0 | 41.0 |
| 10. | .0013 | 003.7 | 06.6 | 52.2 | 7.20 | 30.8 | .762 | .161 | .240 | 3.8 | 39.8 | 3.16 | 15.0 | 22.5 | 45.0 |
| 12. | .0025 | 005.0 | 09.5 | 83.1 | 7.65 | 34.7 | .799 | .179 | .334 | 3.4 | 53.6 | 3.46 | 13.5 | 21.5 | 41.0 |
| 13. | .0015 | 003.0 | 07.1 | 78.3 | 7.38 | 34.4 | .796 | .180 | .316 | 3.6 | 40.0 | 3.34 | 14.5 | 23.0 | 42.0 |
| 14. | .0014 | 003.1 | 07.1 | 68.3 | 6.83 | 27.4 | .761 | .162 | .245 | 4.0 | 42.0 | 3.36 | 12.5 | 23.0 | 41.0 |
| 20. | .0013 | 007.8 | 08.0 | 73.4 | 7.31 | 31.8 | .798 | .176 | .310 | 3.7 | 37.6 | 3.15 | 16.0 | 23.5 | 46.0 |
| 21. | .0011 | .007.4 | 08.0 | 70.8 | 7.21 | 29.7 | .800 | .174 | .318 | 4.0 | 41.5 | 3.30 | 15.0 | 23.5 | 44.0 |
| 22. | .0024 | 011.0 | 06.6 | 62.0 | 7.29 | 29.8 | .76 | .168 | .254 | 3.9 | 33.2 | 3.2 | 13.5 | 22.5 | 42.0 |
| 24 | .0012 | 006.2 | 09.8 | 69.0 | 6.69 | 27.4 | .777 | .161 | .261 | 4.0 | 47.0 | 3.3 | 15.0 | 23.0 | 45.0 |
| 28 | .0020 | 006.6 | 10.4 | 73.7 | 7.72 | 32.3 | .796 | .176 | .332 | 3.7 | 52.5 | 3.75 | 15.5 | 23.0 | 45.0 |

TABLE II

2N1708 ELECTRICAL SPECIFICATIONS

| $T_J$ | $V_{CBO}$ | $V_{CEO}$ | $V_{EBO}$ | $I_C$ | $P_d$ ($T_A$=25°C) |
|---|---|---|---|---|---|
| -65°C to 175°C | 25v (max) | 12v (max) | 3v (max.) | 200ma (max.) | 3 watts (max.) |

| Characteristics $T_A$ = 25°C + | Conditions | Limits | | Units |
|---|---|---|---|---|
| | | Min. | Max. | |
| $I_{CBO}$ | $V_{CB}$=15v, $I_E$ = 0 | | 0.025 | μa |
| $I_{CBO}$ | $V_{CB}$=15v, $I_E$ = 0, $T_A$ = 150°C | | 15.0 | μa |
| $I_{CEX}$ | $V_{CE}$=10v, $V_{BE}$ = .025v, $T_A$ = 100°C | | 15.0 | μa |
| $BV_{CBO}$ | $I_C$ = 100μa | 25.0 | | v |
| $BV_{EBO}$ | $I_E$ = 100μa | 3.0 | | v |
| $BV_{CEO}$ | $I_C$ = 10ma | 12.0 | | v |
| $V_{BE}$(Sat.) | $I_C$ = 10ma, $I_B$ = 1ma | 0.7 | 0.9 | v |
| $V_{CE}$(Sat.) | $I_C$ = 10ma, $I_B$ = 1ma | | 0.22 | v |
| $V_{CE}$(Sat.) | $I_C$ = 50ma, $I_B$ = 5ma | | 0.35 | v |
| $C_{ob}$ | $V_{CB}$ = 10v, $I_B$ = 0, f = 140kc | | 6.0 | pf |
| $h_{FE}$ | $I_C$ = 10ma, $V_{CE}$ = 1v | 20.0 | | |
| $h_{fe}$ | $I_C$ = 10ma, $V_{CE}$ = 10v, f = 100mc | 2.0 | | |
| $t_s$ | $I_C = I_{B1} = I_{B2}$ = 10ma<br>$R_C$ = 1000Ω, $V_{CE}$ = 10v | | 25.0 | n sec. |
| $t_{on}$ | $I_C$ = 10ma, $I_{B1}$ = 3ma, $I_{B2}$ = 1ma<br>$V_{CC}$ = 3v, $R_C$ = 270Ω | | 40.0 | n sec. |
| $t_{off}$ | $I_C$ = 10ma, $I_{B1}$ = 3ma, $I_{B2}$ = 1ma<br>$V_{CC}$ = 3v, $R_C$ = 270Ω | | 75.0 | n sec. |

+Unless otherwise specified

## III. DEVICE PROCESSING

### A. Crystal and Substrate Preparation (B. Czorny, P. Grenier)

Standards are being revised to include the use of mechanically polished substrates. The reliability goals will be completed in this area with the introduction of this change in the process.

#### 1. Items Completed

Mechanically polished substrates have been intrcduced, because of the difficulties experienced with striations on the chemically polished wafers. Superior surfaces are obtained after epitaxial growth on mechanically polished substrates, with minimum chemical polishing, which has improved photoresist definition.

#### 2. Item Under Investigation

Antimony doped substrates in the 0.008 - 0.015 ohm cm range are being investigated. These substrates considerably reduce downstream contamination in epitaxial layer growing and therefore, improve uniformity of product. Equipment which will permit infrared thickness measurement on more lightly doped substrates is being investigated.

### B. Epitaxial Layer-Preparation (U. Roundtree, P. Grenier)

The procedures and process controls for this operation have been standardized. The process improvements which have been adopted are:

#### 1. Improved Method of Substrate Cleaning

This procedure insures that the cleanest possible substrate surface enters the system for exposure and deposition of the epitaxial layer. It is especially designed to remove materials that tend to cause layer erruptions, polycrystal formations and p-type contaminents.

2. Anhydrous HCl Etching of the Wafers at 1200°C (Removing about 0.2 mils of the substrate surface).

This added operation performs two important functions; first, it tends to reduce or eliminate surface imperfections caused by chemical polishing and secondly, it tends to seal off the back of the wafers and eliminate transport downstream doping.

3. Resistivity Measurements by Point Contact Diode Method

Measurement of the individual wafer resistivities is now performed by the three-point probe (point contact diode) method. Individual measurements allow grouping wafers very closely according to resistivity. This permits improved $\beta$ control by appropriate adjustment of the base and emitter diffusion operations.

These process improvements complete the engineering effort in this area. The epitaxial layer preparation operation has been improved to the extent necessary to meet the reliability goals.

C. Oxide Growth and Surface Passivation (S. Policastro)

As a result of the work previously reported, the oxide formation required at various stages during processing have been standardized. These procedures have been incorporated into our standard and their use has yielded excellent results. Further studies have been undertaken as described below:

1. Removal of Oxide on Wafer After Emitter Deposition and Replacing with "Clean" Oxide

In an effort to improve the reliability of the product, investigations have been undertaken to resolve the problem of high saturation current that is associated with silicon planar devices on operating

life tests. Some success has been achieved on similar types by carefully controlling the formation of the final oxide surface of the device, thereby reducing the possibility of contamination that may contribute to this problem. The procedure used is as follows:

After emitter deposition, the entire oxide is removed using hydrofluoric acid. The base silicon surface is now cleaned using a solution of hydrochloric acid and hydrogen peroxide and rinsed in an all quartz system. The wafer is finally dried with a stream of filtered nitrogen. The oxide is then regrown in an all quartz system. The tube and flask used in this operation are cleaned by a special procedure before each run is made.

2. Baking Wafers to Diffuse Wafer Molecules and Other Contaminants Out of Oxides

Another investigation has been undertaken which is related to the problem of surface cleanliness and passivation. This investigation was designed to show the effects of baking the oxides formed at the base and emitter diffusion at elevated temperatures.

One of the possible causes to the problem of high saturation current experienced on operating life tests is the migration of water molecules trapped in the silicon oxide layer or at the silicon - silicon oxide interface. The oxides formed at the various stages of the process are steam grown and there is a possibility of water molecules being present at the interface or in the oxide. Under operating life conditions a strong polar field exists at the junctions and can cause migration of water molecules as well as other polar materials. By baking the oxides at elevated temperature under dry conditions, it is anticipated that the water molecules can be diffused out of the system.

The tests that are under investigation contain wafers that have been baked after base diffusion, emitter diffusion, and both diffusions. This test has also been run on wafers whose oxides had been removed and then regrown.

Because of the excellent test results on wafers in which the oxide was removed and replaced as described and the reliability improvements achieved on other silicon planar devices, it has been decided to incorporate this method as standard procedure in improving the reliability of the 2N1708. Further, the improved procedures in oxide formation, as previously reported, complete the required process improvements to meet the reliability goals of this device.

Tests to determine the effect of baking oxides will be completed and if warrented it will be included in the procedures. However, should the results be inconclusive, further effort will be discontinued since insufficient time remains prior to the start of production.

D. Photoresist and Etching Technique

Efforts in this area have been directed towards the following problems:

1. Improved Methods of Removing Photoresist After Etching

As a result of investigations being conducted in the laboratories, it was decided to abandon the use of organic nitrogen compounds for the removal of photoresist. Results indicated that the present method of using methylene chloride spray and combustion was a superior production method. However, the use of hot concentrated sulfhuric acid with chromic acid added appeared to be a better system. An experimental run using these materials is now in process and the results are encouraging. The standards will be revised to the use of this method if no detrimental effects are observed on life tests.

2. Obtaining Contacts by Improved Photoresist Techniques

The present method of obtaining aluminum contacts consists of evaporating aluminum over an exposed, developed and etched photoresist mask in the surface of the wafer. The aluminum is removed, except in the contact opening developed in the photoresist, by scrubbing the aluminum with a solvent which dissolves photoresist. This method has undersirable features because contamination may be scrubbed into the aluminum-oxide interface at the periphery of the contact.

This problem may be eliminated by the addition of another photoresist step which will permit the removal of aluminum in the unwanted areas using Na OH. An investigation is currently being made to evaluate this procedure. If satisfactory electrical yields are obtained on the wafers, the procedure will be standardized.

3. Filtering of Chemicals Used in Photoresist Operations

It has been established that all organic chemicals used in the photoresist operations will be filtered.

E. Surface Preparation and Cleaning (W. Kern)

Basic work in this area has been performed by the Advanced Development Laboratory of RCA, Somerville. Work of this nature is considered to be of utmost importance in achieving the high reliability requirements and has led to a number of significant conclusions that have been standardized for this program. Recommendations are being evaluated continually for possible inclusion in the standards prior to the start of the production run.

Important sources of impurities which are organic in nature are discussed below. Investigations are in progress in the laboratory on inorganic sources of contamination.

1. Organic Materials as Sources of Semiconductor Surface Contamination

a. Lapping Oils

Commercial lubricating oils are used in the mechanical lapping of cut silicon wafers. These lubricating preparations are soluble in many organic solvents. The bulk of the adhering oil is normally washed from the wafers by rinses in trichlorethylene. However, oily residues remain sorbed on and in the lapped surface layers and could have undesirable effects in the subsequent etching. It was shown by radioautographic analysis that such residues, especially if present as non-uniform spots or rings, can act in certain etches as masking agents, and impede the etch rate in localized areas. Ultrasonic cleaning of the wafers in a hot aqueous solution of nonionic surface-active agents was found more effective than additional extended treatment with organic solvents, and has been recommended for wafer treatments for this program.

b. Waxes And Hydrocarbons

The use of solid surfactants as wax substitutes for mounting and masking of semiconductor wafers, as discussed in the previous report, is being employed extensively and with great success. Waxes are still needed in some instances where masks are required that are completely insoluble in aqueous etch systems. Following solvent rinses (trichlorethylene, xylene) to remove the gross amounts of the waxes, residual layers on silicon are effectively removed by wet oxidation in hydrogen peroxide solution. Electronic grade hydrogen peroxide at a concentration of 6% and at a temperature of 95°C was found by sensitive surface tests to remove such

residues invariably within 30 minutes, forming soluble or volatile oxidative breakdown products which are eliminated by rinses with hot deionized and distilled water. The waxes tested include paraffin, beeswax, hydrocarbon hard waxes, apiezon wax, and Kel-F wax. Preliminary tests indicate that the rate of oxidation can be increased by adding small amounts of ammonia to maintain an alkaline pH.

c. Vacuum Grease

A persistent p-type contamination of epitaxially grown silicon layers was traced to Dow-Corning Silicon vacuum grease that had been used to lubricate the joints of the deposition apparatus. This material contains boron, added deliberately by the manufacturer as a viscosity modifier, and also aluminum compounds in the 10 ppm range of concentration. Contamination of wafer surfaces can result from either direct transfer, by accidental contact, or by mass transport as a mist by a gas stream. This type of surface contamination was detected during heat treatment of silicon wafers in a stream of hydrogen and ranged up to $5 \times 10^{16}$ carrier atoms per $cm^3$. The contamination is avoided by the use of halocarbon grease (Halocarbon Products Corp., Hackensack, N.J.) for the glass and quartz joints. It is to be noted that Halocarbon must not come in contact with aluminum, since it is explosive.

d. Teflon

It was found by spectrographic analysis that Teflon contains boron compounds which can be leached out by solutions or can volatilize in steam. Surface contamination of concentrations

up to $10^{16}$ atoms per $cm^3$ could be traced to this source. It appears that the boron compounds originated from the moulding powder used by some manufacturers of Teflon laboratory ware. The use of Teflon ware has been discontinued in any operations where contamination may have deleterious effects. Fused quartz laboratory ware is now used wherever possible in the wafer processing operations.

e. Hard Plastics

Commonly used sample storage containers made of hard plastics (polystyrene, polymethyl, methacrylate, etc.) can cause surface contamination in a short time through gaseous transfer of trace impurities, as demonstrated by surface tests capable of detecting fractional monolayers. To prevent contamination, silicon wafers and devices are stored after cleaning treatments in Petri dishes with glass sub-dividers; these dishes are in turn stored in a second glass covered container. It was demonstrated that samples stored under these conditions remained clean for at least several days.

f. Polyvinyl Chloride Plastic

Investigations have shown that polyvinyl chloride (PVC) plastic sheets can be used advantageously for mounting and masking of semiconductor wafers and pellets in certain areas of device fabrication, if processed by a special technique. The mounting method is particularly useful for the 2N1708 pellet separation in the present project. The scribed wafers are placed between two PVC sheets, covered on both sides with cellophane, and separated into pellets by cracking. The pellets can then be inspected while on

the plastic support, followed by removal for further processing. It is very important to subject the pellets at this point to an effective surface treatment because of contamination by polyvinyl chloride and dioctylphtalate plasticizer introduced during this process. Rinsing with organic solvents was found insufficient to completely remove these residual contaminants, as demonstrated by water repellency tests and electrical analysis of device parameters. An effective clean-up treatment has been instituted with excellent results. The treatment consists essentially of extractions with hot metyl alcohol followed by an aqueous solution of a nonionic surface-active agent and distilled water rinsing. A porous quartz disc apparatus is used for collection of the pellets. All containers are of fused quartz and the processing is conducted under nearly dustfree conditions.

g. Solvents

Organic solvents are common sources of contamination, as shown by residue determination, UV fluorescence analysis, chemical analyses, and electrical tests with silicon diodes. A selection of 16 common solvents of reagent grade or electronic grade quality was chosen. Of those tested, only Varsol, Fisher Reagent Grade carbon tetrachloride and petroleum ether, and Baker Reagent grade chloroform were found acceptable for application where very high purity is required. Trichlorethylene (Nitine, electronic grade) was found to deteriorate rapidly when used at elevated temperature. For example, fractionation by distillation under normal pressure yielded 140 mg of residue per 1000 ml from which a crystalline

compound was isolated and identified by functional group analysis and x-ray diffraction as triethylamine hydrochloride. This substance apparently originates from triethylamine added as stabilizer and from hydrochloric acid formed as a decomposition product from trichlorethylene. Decomposition is accelerated by heat, energetic electromagnetic radiation (such as UV, x-rays, gamma radiation), and metal catalysts. While triethylamine has a boiling point close to that of the trichlorethylene, the hydrochloride derivative is a solid melting at 254°C which remains at lower temperature as residue. When usingtrichlorethylene, overheating by use of a heating bath should be avoided. The application of chloroform or carbon tetrachloride, in which ethyl alcohol is usedas a decomposition inhibitor, is recommended as a substitute for trichlorethylene to minimize contamination.

It should also be realized that storage vessels may contaminate originally clean liquids. For example, storage of methyl alcohol in borosilicate glass bottles, such as Pyrex, may give rise to contamination by boron by the reaction:

$$6\ CH_3OH + B_2O_3 = 2(CH_3)_3^0B + 3H_2O$$

over a storage period of several months. The methyl borate can build up to concentrations of a few hundredths of one percent. Exposure of methyl alcohol to light induces the formation of formaldehyde which oxidizes easily to formic acid or can give rise to a variety of polymerization products. Observed degradation of surface sensitive device parameters are likely to be caused by such contaminants. Final rinses should best be done with high purity

carbon tetrachloride or preferably with superpure low-conductivity water obtained by mixed-bed ion exchange deionization followed by two distillations in stills of fused quartz. Silicon wafers that received a final hot rinsing treatment in quartz beakers with this high purity water should be processed as follows:

Grip the wet wafer on its extreme edge with specially prepared tweezers, holding it up so that the liquid drains down the tweezers. Hold the wafer in this position inside a wide quartz tube while passing filtered nitrogen down the tube while directing a beam of light from a 500 watt (GE, DHJ, shielded) projection lamp onto the wafer. Drying takes just a few seconds. The wafer is stored in double petri dishes, cleaned in hot hydrogen peroxide and distilled water.(See Figure 1)

It should be mentioned in this connection that storage of chemically polished silicon wafers under water leads invariably to contamination by a micro-organism of the order thallophyta Microscopic examination of originally clean samples stored for several days in deionized and distilled water under a variety of conditions revealed the appearance of many randomly distributed particles, often grouped in clusters. At 200 to 2000 fold magnifications these particles were seen to consist of a filamentous growth of irregularly branching or widening fibers of up to 1 micron diameter. After 10 days the growth was quite dense and visible as a haze by the naked eye. Storage under sterile conditions did not lead to this contamination. The contaminant was quite resistant to chemicals and solvents except

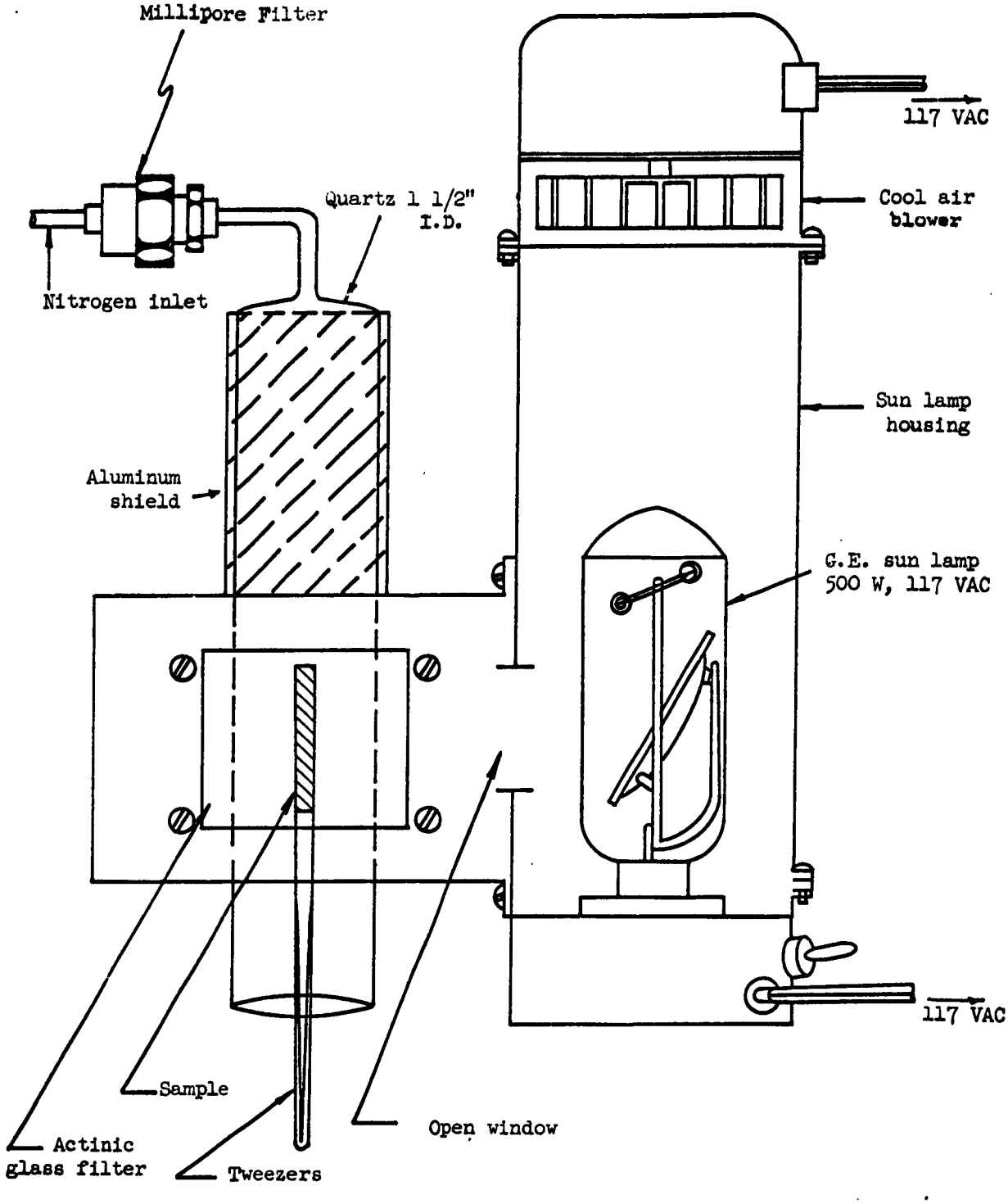


FIGURE I - WAFER DRYING APPARATUS

strong oxidizing agents, and might, therefore, give rise to trouble on subsequent processing. The problem is eliminated by applying the rinsing and drying technique described above.

2. Inorganic Reagent Materials As Sources Of Semiconductor Surface Contamination

Systematic radiochemical investigations are in progress concerning the contamination of silicon surfaces by trace cations and anions during etching. A variety of acid and alkaline etch systems is being studied with the following radionuclides as tracers:

| Cations | Anions |
|---|---|
| iron - 59 | fluorine - 18 |
| gold - 198 | chlorine - 36 |
| chromium - 51 | iodine - 131 |
| copper - 64 | carbon - 14 |
| cobalt - 60 | |
| zinc - 65 | |
| manganese - 54 | |
| antimony - 122 | |
| molybdenum - 99 | |
| sodium - 22 | |
| sodium - 24 | |

The evaluation of quantitative radiochemical data is in progress and the results will be presented in the next report.

F. Diffussion Processing (S.Policastro, W.Grieg)

In the preceeding report, the results of a detailed study of the $P_2O_5$ carrier gas system for phosphorus diffusion were discussed. During the past period, however, an improved $POCl_3$ system was developed in the Materials Service Laboratory of RCA, Somerville. Phosphorous Oxychloride

($POCl_3$) is a liquid at room temperature and is obtainable in reagent grade quality essential for high reliability device fabrication.

Several experiments were undertaken to evaluate the use of this material as a diffusion source for the high reliability 2N1708 program. It has been determined that the $POCl_3$ system is at least equal to the $P_2O_5$ carrier gas system. In addition, several advantages were realized with the use of this system as listed below:

(1) The need for a source furnace was eliminated.

(2) The source can be used repeatedly without change.

(3) The source exposure to the atmosphere is minimized.

As a result of the inherent reliability improvements, process standards have been written incorporating this procedure. This process improvement is considered to complete the work necessary in this area to achieve the high reliability goals of the program.

G. Contact Evaporation (G. Granger, W. Triggs)

Two designed experiments were made to evaluate gold contacts in comparison to aluminum contacts. The design of these tests is shown below.

DESIGN OF EXPERIMENT

| | |
|---|---|
| Cell# 1<br>Aluminum Contacts<br>No Encapsulant | Cell# 2<br>Aluminum Contacts<br>Encapsulant |
| Cell# 3<br>Gold Contacts<br>No Encapsulant | Cell# 4<br>Gold Contacts<br>Encapsulant |

All possible variation in the experiment were minimized by randomization of the processing, e.g., the wafers were divided in two immediately prior

to evaporation and half of the wafer was evaporated with aluminum and half with gold. The pellets were recombined into one lot for processing through assembly and separated into the four cells prior to the pre-seal bake by visual examination.

After sealing, the units were tested for Group A requirements and subjected to life tests as follows:

1. 300°C Shelf life for 96 Hours - All units in each cell
2. 300 mw Operating life for 96 Hours - All units in each cell
3. Extended Operating and Shelf Life Tests - 25 units on each test from each cell.

Only two cells (Nos. 2 and 3) were processed on the second test because of accidental loss of half the units started on this experiment.

Results

No significant difference was noted between gold and aluminum contacts on the first designed experiment in the processing area until after connector bonding. Evaluations were made and the bond strength of units with gold contacts was lower than the bond strength on units with aluminum contacts by a factor of two-thirds. This difference was attributed to lack of control of the amount of metal evaporated, because it was not found on the second test where complete control of the evaporation was achieved. (Measurement equipment for control of evaporated material was received and installed on the evaporator before the second test was started).

All of the units on the second designed experiment and a sample from cell #2 and #4 on the first were subjected to all of the electrical tests on the 2N1708 electrical specification. An analysis of $V_{CE}$ and $V_{BE}$ was made to determine if a difference existed between gold and aluminum

contacts due to variation in contact resistance. There was a significant difference between the distribution of $V_{CE}$ and $V_{BE}$ on gold and aluminum contacts as shown in Figures (2) and (3). However, this is due primarily to a difference in current gain between units with gold and aluminum contacts. The difference in the distribution of current gain is shown in Figure (4).

To further substantiate the fact that gold contacts have equivalent or lower saturation voltage than aluminum contacts with the same current gain, a detailed analysis was made of all the units from the second designed experiment. The results of the analysis is as follows:

1. There is a significant difference, at the 90% confidence level, between the current gain distribution on units made with gold contacts and units made with aluminum contacts.
2. There is a significant difference, at the 90% confidence level, between saturation voltage on units made with gold contacts and units made with aluminum contacts.
3. There is a high correlation between current gain and $V_{CE}$ for units with both gold and aluminum contacts as shown in Figure (5).
4. Given the same current gain values for units with gold contacts and aluminum contacts, there is a 90% probability that the saturation voltage will be higher for units with aluminum contacts.

The difference in distribution of current gain on units with gold and aluminum contacts is attributed to variations in processing which occurred on the wafer after evaporation. The distribution of current gain was higher on units with gold contacts on the first test and lower

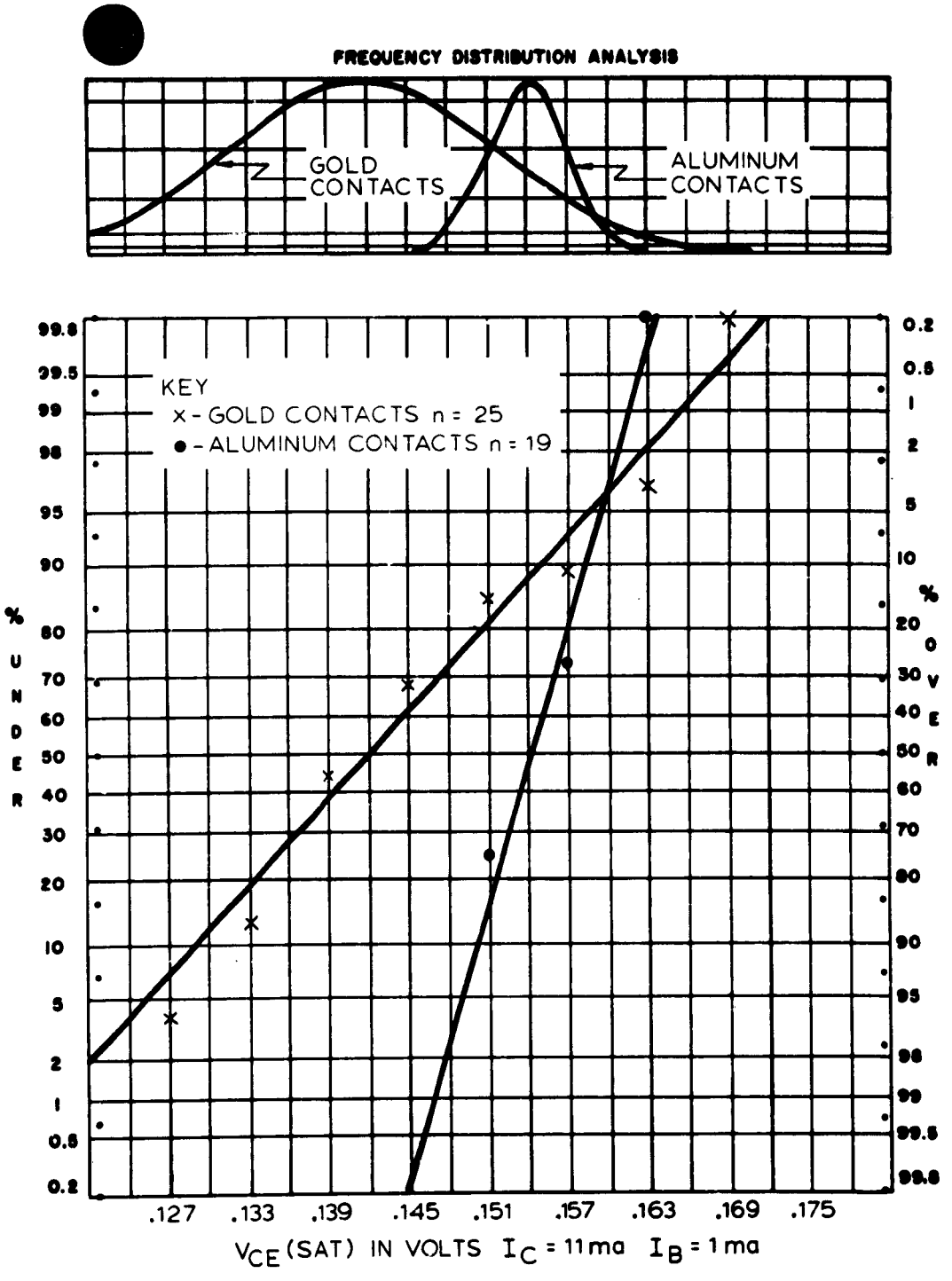


FIGURE 2 FIRST DESIGNED EXPERIMENT DISTRIBUTION OF $V_{CE}$ (SATURATION)

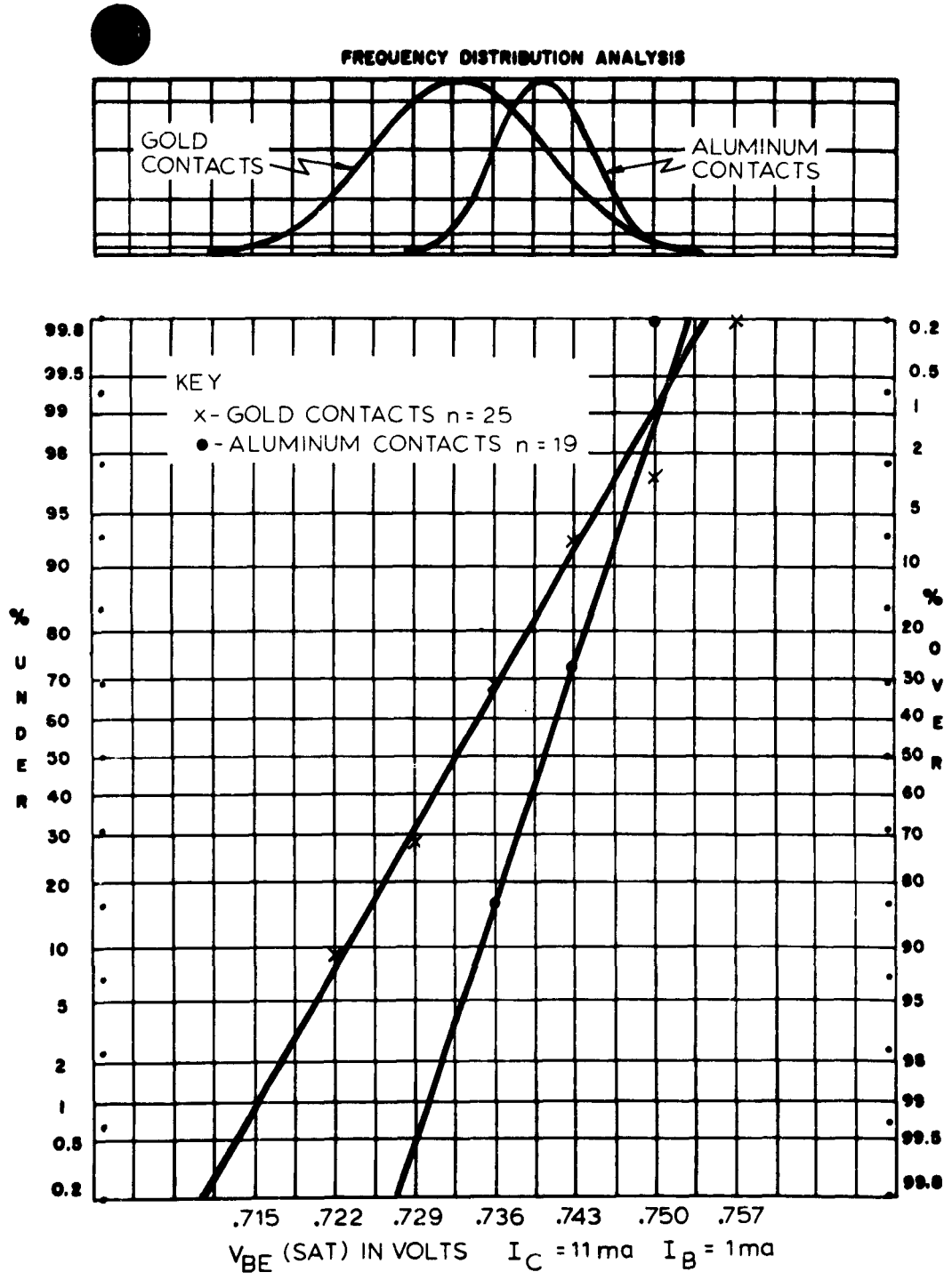


FIGURE 3 FIRST DESIGNED EXPERIMENT DISTRIBUTION OF $V_{BE}$ (SATURATION)

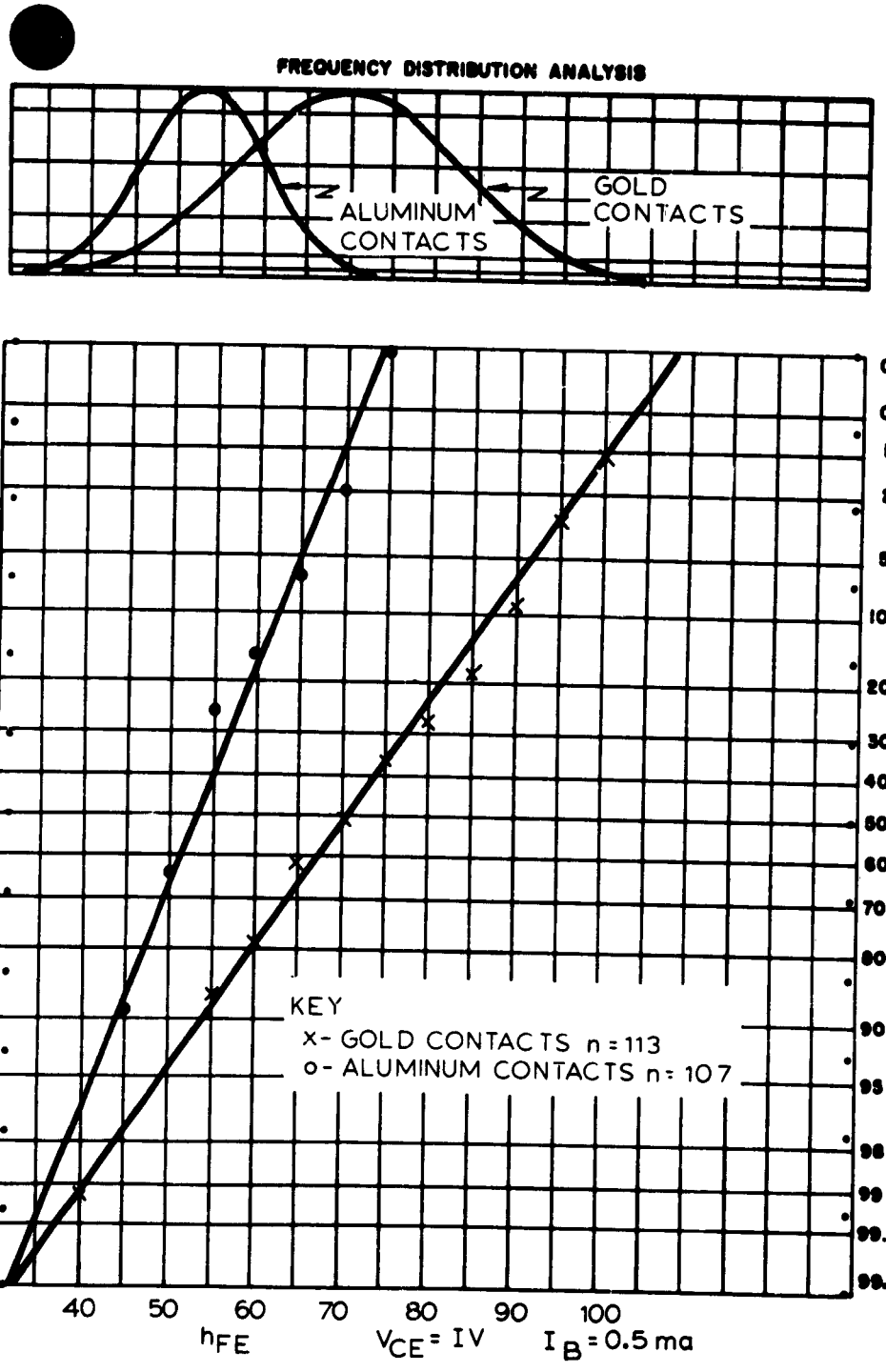


FIGURE 4 FIRST DESIGNED EXPERIMENT DISTRIBUTION OF $h_{FE}$

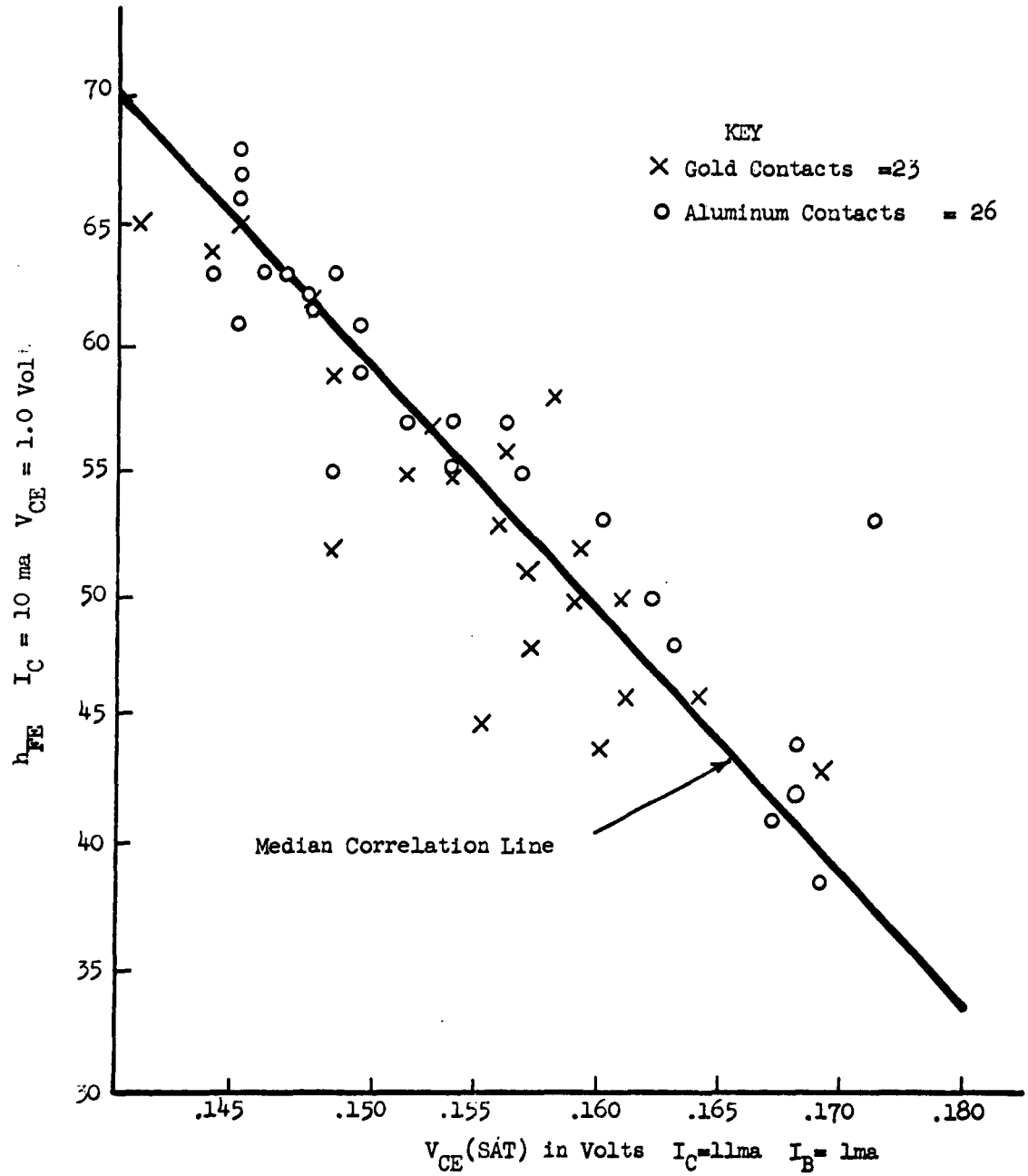


FIGURE 5 SECOND DESIGNED EXPERIMENT GRAPH OF $V_{CE}$ vs. $h_{FE}$

on the second test.

The results of short term and extended life test are as follows:

First Designed Experiment

| # Cell | No. of Units | Failures* Prior To 300°C 96 Hr | Failures* After 300°C 96 Hr. | Failures* After 300mw 96 Hr. | Type of Test | 100 Hr. Down Period | 250 Hr Down Period |
|---|---|---|---|---|---|---|---|
| 1 | 116 | 9 | 3 | 1 | 300°C<br>300mw | 0<br>0 | 3<br>0 |
| 2 | 111 | 3 | 0 | 4 | 300°C<br>300mw | 0<br>0 | 0<br>0 |
| 3 | 104 | 8 | 5 | 33 | 300°C<br>300mw | 9<br>2 | 2<br>2 |
| 4 | 125 | 12 | 2 | 4 | 300°C<br>300mw | 3<br>0 | 0<br>0 |

* Units were criticized to a tightened criteria at these test points.

Second Designed Experiment

No life test results available on second designed test to date.

The majority of the failures from cell #3 were open units. When failure analysis was performed on these units it was found that severe discoloration of the contacts was present on all of the failing units and the connector wire had physically separated from the contact area.

There is no explanation for the formation of this discoloration at the present time. Tests are being made to determine the materials involved so that corrective action may be taken. No further designed experiments on gold contacts will be performed unless this problem is corrected. Engineering effort will be devoted to improvement of the

aluminum contact to meet the reliability goal.

H. Mounting Pellet to Stem - Stem Design (G. Granger)

Process improvements in the mounting operation and stem design are considered complete and satisfactory to meet the reliability goals of the contract.

(1) Metal Evaporation on Back of Pellet

Since the last report, further improvement in metal backing was made by the use of a gold alloy in place of pure gold. The benefits derived from this improvement are as follows:

(a) Substantial improvement in wetting

Every unit which was examined after removal of the pellet by etching showed complete wetting of the gold to the back of the pellet. There were no voids or holes apparent in the remaining gold as there was on the control.

(b) Reduction in stress on stem

The new gold backing process permitted a 50°C reduction in peak temperature on mounting, reducing the stress on the package due to thermal shock.

(2) K & S Mounter

The temperature profile for mounting of the pellets to the stem has been established and a procedure for monitoring the profile has been included in the Process Control procedure.

(3) Continuous Furnace Mounting

No further work has been done in this area.

(4) Stem Design

Modification of the ceramic stem design to reduce production

problems has been completed and initial 300 samples have been received for evaluation. One hundred samples were subjected to thermal shock testing without a failure. Devices are currently being assembled on some of these samples stems for electrical evaluations.

Because of insufficient time remaining in the development phase of the program, it is not planned to change from the present stem design to the ceramic stem.

I. Wire Bonding (G. Granger)

Engineering effort in this area was reduced awaiting results of the gold contact experiments (see III-G). However, with the poor results obtained on the experiments near the end of the quarter, increased emphasis was placed on bonding with gold alloy wire to aluminum contacts by thermal compression bonding techniques. Aluminum thickness was increased to 4,000 Å but it was found that the force required to obtain a satisfactory bond caused fracture of the silicon under the aluminum.

Major effort will be expended during the next quarter to develop this method. Experiments on similar devices have shown a significant reduction in the rate of formation of intermetallic compounds such as that described as "purple plague". Experiments will be performed as follows:

(1) increased aluminum contact thickness - thermal compression bonding.

(2) increased aluminum contact thickness - nail head bonding (new equipment permitting the use of 0.7 mil wire will be utilized).

Investigations to develop methods of bonding aluminum contacts to aluminum wire will be continued.

J. Sealing (G. Granger)

Life test failures which have been analyzed show characteristics which are attributable to moisture in the package. Two approaches have been taken to eliminate this problem: (1) inclusion of a moisture getter in the package; and (2) improved baking procedures before sealing.

Initial test run on units made with a desiccant in the package show a rapid degradation on life. (There were 14 failures in a sample of 20 on the test after 96 hours of operating life and no failures on the control for the same period.) The difference in failures is attributed to the improper choice of the desiccant. The desiccant selected was not one which has irreversible desiccant properties and, therefore, the dew point in the package on the test is probably higher than the control.

A designed experiment has been run to further evaluate another desiccant which should be satisfactory. The units which were made on this test are now on life.

Equipment is on order to evaluate the inproved baking procedure before sealing.

K. Automatic Test Set (P. Grenier)

The automatic test equipment has been used continuously throughout the past quarter as described in the preceding Quarterly Report. Process controls have been established using a standard resistor to check the digital voltmeter prior to every run. In addition, a standard correlation unit is run with each board of units to detect reading variation or punching error.

Experiments have been made to determine whether the automatic test equipment has caused occasional unit failures due to voltage surges, etc. On completion of these experiments, it has been concluded with a high degree of confidence that the equipment does not cause unit failures.

With the implementation of the automatic test set and the establishment of appropriate process controls, the process improvements in this area are considered completed.

IV. INSPECTION AND QUALITY CONTROL (P. Grenier)

Inspection and Quality Control is being performed on incoming parts and in all phases of the manufacturing operation. In addition, the final units are being tested in accordance with the requirements of the program. As part of the program, an Inspection and Quality Control Plan manual is required and after further formalization of the necessary procedures, the manual will be completed.

The manual is in preparation as follows:

1. A set of flow charts has been prepared detailing the flow of material into and through the process from receiving to shipping. Each inspection station has been noted on the charts and will be described in the plan.

2. A list is being prepared of each inspection/test, including purchased material, in-process and end item inspection and quality control as follows:

| Inspection/Test | Instrument or Equipment Manufacturing Name and Model Number | Characteristics and Specification Limit | Sampling Plan | Inspection and Process Control Procedures (See 3 Below) |
|---|---|---|---|---|

3. A narrative description of the procedures to be used for inspections and process control is being prepared. Samples of data sheets will be provided.

4. A proposed military specification for a high reliability version of the 2N1708 has been prepared. This specification will be reviewed and revised as additional data is obtained.

## V. CONCLUSIONS

Process improvements have been completed in certain areas, others are still under investigation. These are as follows:

### A. Process improvements completed

#### 1. Crystal and substrate preparation

An improved method of polishing substrates has been developed to reduce striations which appear on the surface of the epitaxial wafer.

#### 2. Epitaxial layer preparation

Techniques improving substrate cleaning and preparation have been introduced. An improved method of measuring the resistivity of the epitaxial layer has been established.

#### 3. Oxide Growth and Surface Passivation

Oxide thicknesses have been optimized. A technique of removing the oxide after phosphorous diffusion and replacing it with a "clean oxide" has been introduced.

#### 4. Diffusion Processing

An improved phosphorous diffusion technique is in use utilizing a $POCl_3$ source instead of $P_2O_5$.

#### 5. Mounting

Improved wetting at lower mounting temperatures has been achieved and included in the process. This has been accomplished by evaporation of a gold alloy on the back of the wafers.

#### 6. Automatic Test Set

An automatic test set is in use. Process controls have been established to insure high reliability.

### B. Process improvements to be completed

#### 1. Photoresist

2. Surface Preparation and Cleaning
3. Contact Preparation
4. Bonding
5. Sealing
6. Ceramic Stem

## VI. PROGRAM FOR NEXT INTERVAL

A. Complete process improvements in the following areas:

1. Photoresist
2. Surface Preparation and Cleaning
3. Contact Preparation
4. Bonding
5. Sealing
6. Ceramic Stem

B. Begin a production run to demonstrate the reliability level which has been achieved.

## VII. PERSONNEL AND MAN-HOURS

The engineering personnel contributing to this program during this quarter are listed in Table III. Also indicated is a tabulation of the man hours expended on the contract. Figure (6) depicts the 2N1708 Reliability Improvement Project Organization.

TABLE III

MANHOURS FOR THE THIRD QUARTER

| Name | November | December | January | Total |
|---|---|---|---|---|
| | | ENGINEERS | | |
| W. L. Totten | 168 | 152 | 176 | 496 |
| G. F. Granger | 168 | 152 | 176 | 496 |
| A. A. Dunham | 12 | 20 | 35 | 67 |
| J. F. Vance | 15 | 30 | 30 | 75 |
| P. J. Grenier | 168 | 152 | 176 | 496 |
| W. J. Grieg | 95 | 6 | - | 101 |
| L. P. Fox | 5 | 3 | - | 8 |
| E. L. Williams | - | 7 | - | 7 |
| E. G. Buckley | - | 4 | - | 4 |
| W. M. Triggs | 19 | 29 | 56 | 104 |
| J. A. Emanuel | 26 | 39 | 20 | 85 |
| L. N. Pomante | 6 | 12 | 12 | 30 |
| S. Policastro | 160 | 156 | 184 | 500 |
| P. A. Hoff | - | - | 6 | 6 |
| Total Engineers | 842 | 762 | 871 | 2475 |
| | | TECHNICIANS | | |
| Manufacturing Services | 1014 | 942 | 1086 | 3042 |
| Testing Services | 676 | 628 | 724 | 2028 |
| Total Technicians | 1690 | 1570 | 1810 | 5070 |
| Total | 2532 | 2332 | 2681 | 7545 |

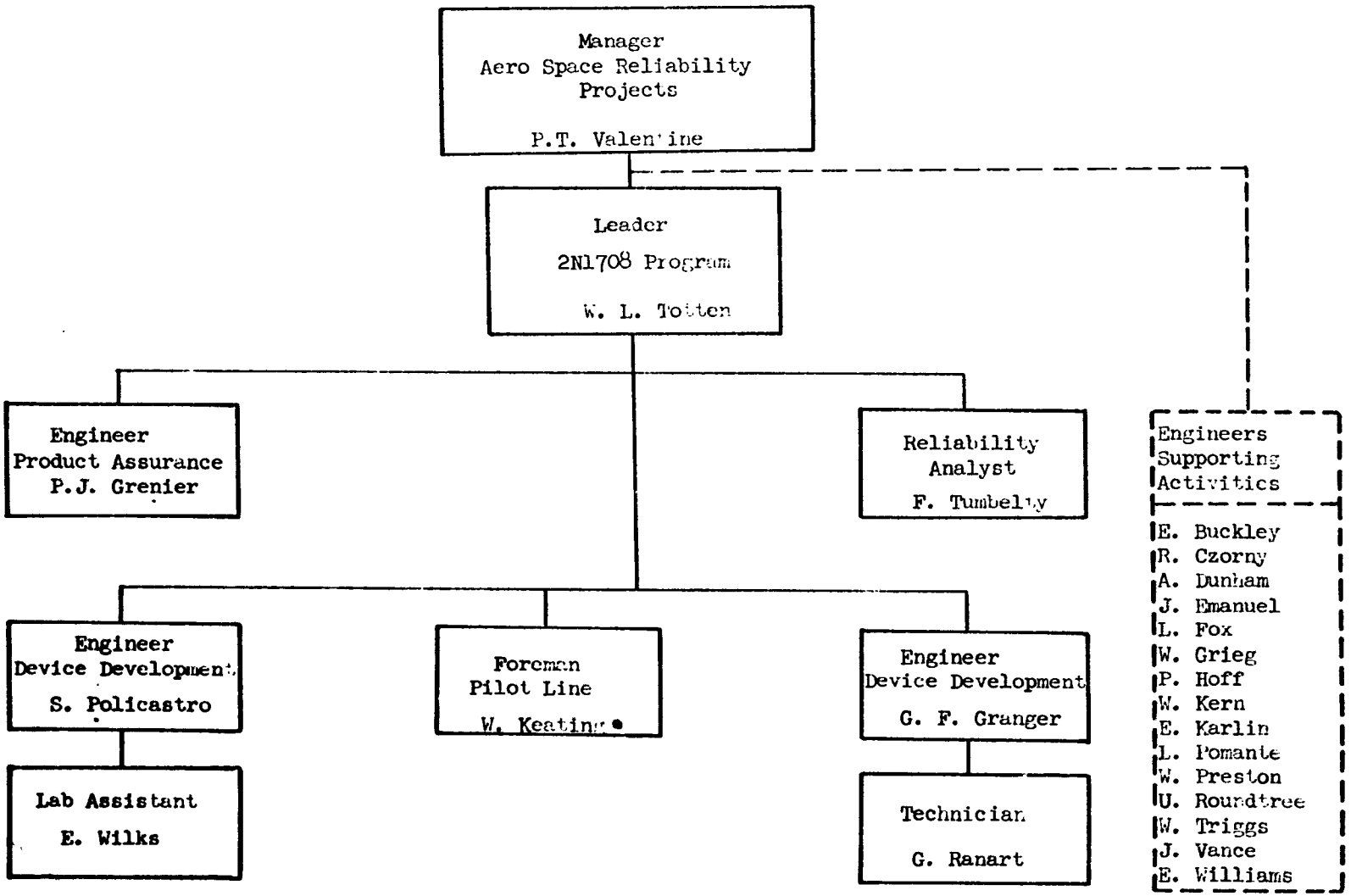


FIGURE 8 2N1708 RELIABILITY IMPROVEMENT PROJECT ORGANIZATION CHART