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PRODUCTION ENGINEERING MEASURES
TO INCREASE
TRANSISTOR RELIABILITY
QUARTERLY PROGRESS REPORT NO. 3
FOR THE PERIOD
NOVEMBER 1, 1962 TO JANUARY 31, 1963

CONTRACT NO. DA-36-039-SC-86730

PLACED BY
U.S. ARMY ELECTRONICS MATERIAL AGENCY
PHILADELPHIA, PENNSYLVANIA

TEXAS INSTRUMENTS INCORPORATED
SEMICONDUCTOR-COMPONENTS DIVISION
PRODUCTION ENGINEERING MEASURES

TO INCREASE

TRANSISTOR RELIABILITY

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PHILADELPHIA, PENNSYLVANIA

TEXAS INSTRUMENTS INCORPORATED

SEMICONDUCTOR-COMPONENTS DIVISION
OBJECT:

To establish engineering measures necessary for attainment of a maximum operating failure rate of 0.01% and 0.001% per 1000 hours at a 90% confidence level on the 2N960 series of Germanium Epitaxial Mesa Transistors, and the 2N744 Silicon Non-Planar Epitaxial type, respectively.

CONTRACT NO. DA-36093-SC-86730
ORDER NO. 19052-PP-62-81-81
CONTRACT ITEM 2 - 2N964
CONTRACT ITEM 3 - 2N744

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Silicon Mesa-Planar Transistor Dept.

Jim Lineback, Engineer
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Robert M. Shurley, Product Engineer
Germanium Small-Signal Department

Sam L. Carrell, Manager of Quality and Reliability Assurance Department

Albert T. Némecek, Contract Coordinator
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SECTION I - 2N964

ABSTRACT

This report covers all work performed during the third quarter of the contract.

During the report period one group of engineering test samples was shipped as per the contract requirements.

The major area of engineering activity during the report period was on the following process steps:

- Epitaxial Germanium Control
- Surface Preparation for Growth
- Cleaning Techniques to Obtain Optimum Surface Conditions
- Welding Techniques for Encapsulation
- Evaporation Control
- Mesa and Dice Cutting
- Piece Part Incoming Inspection
The purpose of the work provided in this contract is:

1. To establish process improvements necessary to attain improved operating failure rate.
2. To incorporate the process improvements into the production facilities.
3. To supply engineering test samples incorporating the improved processes.
4. To provide quarterly reports, final engineering report, bills of materials and parts, and general report.
SECTION III - 2N964

NARRATIVE AND DATA

This section covers the engineering process during the third quarterly period of the contract. A revision was made to the original process schedule for Item 2 (2N960 Series) in light of additional knowledge and planning during the contract work and is shown in Figure 1.

Additional work and all data are reported as separate sub-sections.
## Process to be Improved

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**FIGURE 1**
ENGINEERING PROGRESS (ITEM 2)

EPITAXIAL GROWTH CONTROL  (Bob Falt)

During this quarter a slight modification has been made to the cavity which holds the substrate during epitaxial film deposition. The depth of the cavity was reduced slightly to improve the gas flow over the surface area of the slice. The Perkin-Elmer 221 Infra Red Spectrophotometer has been used to check the film thickness on all slices produced. The capability of the epitaxial facility has shown a variation of approximately ± 15% from the normal, in film thickness for slices produced for the 2N960 series. This variation is from slice to slice and run to run, as well as reactor to reactor. The film thickness variation across the slice is considerably less than this figure.

SURFACE PREPARATION OF SUBSTRATES TO GROWTH  (Bob Falt)

Work has continued on electrochemical polishing of the substrates prior to epitaxial film deposition. This technique appears to provide a slightly superior surface to the epitaxial material. Indications are that some slight improvements in electrical parameter distributions may result also. Some work will continue on this item during the next quarter. However, it is felt that present surface preparation techniques are satisfactory for the goals of this contract.

EPITAXIAL CONTINUITY CONTROL  (Bob Falt)

Alterations discussed in the second quarterly report have continued to provide satisfactory resistivity control of the deposited epitaxial film deposited. This item has also been completed for the goals of this contract.
DIFFUSION CONTROL TO ELIMINATE RESISTIVITY PRODUCT PROBLEM

Diffusion control has remained satisfactory during the third quarter of this contract work. Diffusion runs have been made using 1000 mg of 1 mg Antimony pellets for the dope source. These diffusions have produced slices with sheet resistivities comparable to those from production diffusion runs. Electrical parameter distributions from finished devices also appear comparable to those using the standard production technique.

PROTECTIVE COATING TECHNIQUES FOR SURFACE PASSIVATION

Work has continued on the application of coating to germanium devices. However, as mentioned in the last quarterly report it is not expected that truly passivated coating techniques will be available for use on this product line by the close of the process work.

CLEANING TECHNIQUES TO OBTAIN OPTIMUM SURFACE CONDITIONS FOR ALL FABRICATION PROCESSES

The new automatic etch facility discussed in the second quarterly report has been placed in operation for the 2N960 series. The automatic unit loader is still not operating completely satisfactory and work will continue on this during the next quarter.

A pre-bonding cleaning operation has been installed in the operation. Also an out gassing procedure has been instituted for all mounting furnaces.

Presently investigations are under way on the use of a hot solvent ultrasonic cleaning for slices during the photo-resist removal steps of the process.
Investigation on header preparation resulted in improved header cleaning procedures prior to gold plating, that have been recommended to the Glass-to-Metal Seals department.

(Bob Eldridge)

WELDING TECHNIQUES FOR ENCAPSULATION AND SEALING

The use of a Nitrogen ambient is presently being investigated for the bake out medium in the final preparation of cans and desiccant, thus permitting the temperatures to be elevated without discoloration to the metal cans.

Initial samples of desiccant pre-form and other parts have been obtained to modify the encapsulation procedure as discussed in the last quarterly report.

Fabrication difficulty and the possibility of reducing the cost lead to an alternative method. In the later method a desiccant, of piece part nature, will be held in place within the transistor package by a formed piece of preforated metal. The earlier method did not call for forming the desiccant retainer and thereby needed an additional piece part. The desiccant retainer will be welded to the inside of the cap so that good mechanical rigidity will be maintained.

CONTROLLED FORMATION OF SURFACE OXIDES FOR SURFACE STABILIZATION (Bob Eldridge)

The pre-can bake temperature has been raised to 180°C to provide additional surface oxidation and improve parameter stability.

Investigations have been made on imporved humidity control for the pre-encapsulation baking operation. These results have indicated that sufficient control is being achieved in the present system.
PROTECTIVE INERT COATING FOR SURFACE STABILIZATION

Work has continued on the techniques for applying a protective coating to devices. However, as reported in the last quarterly report it is not expected that this work will have progressed to the realization of desirable production technique by the close of the process work for this contract.

EVAPORATION CONTROL  (Bob Eldridge)

Preliminary design work has been done on an evaporation mask and work holder arrangement which will provide the appropriate arrangement for best stripe definition. A considerable number of problems have been encountered on the geometrical positioning of the masks, shields, and evaporator filaments to provide the stripe sizes and tolerances over the area of the slice. Also it appears there may be some difficulty in obtaining the desired dimensions and tolerances on the mask itself. Work will continue on this during the next quarter.

Even though no specific specification change has been made in this area, the technique used has been improved. Evaporator operators have become more proficient because of the studies made under this item.

MESA AND DICE CUTTING  (Bob Eldridge)

Dice Cutting  - Operation continuing to be satisfactory.

Mesa Cutting  - Satisfactory mesa cutting is being experienced with the KMER process mentioned in the previous quarterly report. Some experimental work is still in process, however; and minor modifications may be made prior to the close out of the process work for this contract.
This work is concerned with the pre-conditioning of the photo-resist, whereby impurities are removed and resist is re-constituted to a specific viscosity. Investigations are being made on exposure light sources, curing cycles, development techniques, etchants and resist removal techniques.

PIECE PART INCOMING INSPECTION PROCEDURES (Dick Jensen)

Piece part incoming inspection procedures have been written for all parts currently being procured for the 2N960 series transistors.

Six of the Incoming Control Inspection Instructions have been improved through the use of Check Fixtures. The Check Fixtures are go-no go gages to be used in checking piece part dimensions.

Review of Piece Part Incoming Inspection Instructions will be continued to determine possible continued improvements in techniques, equipment or method.

PROCESS CONTROLS (Dick Jensen)

Process Control inspection procedures are 90% completed, and improvements in process control procedures are being incorporated.

All applicable Quality Assurance Specifications will be included in the Inspection and Quality Control Plan (manual) which will be prepared in accordance with the contract requirements.

POST CAN STRESSING (Bob Eldridge)

Power aging has been under consideration for this series as a means of detecting marginal devices prior to formation of a finished lot of devices. Factors revealed have made this somewhat questionable as to the possibility
of this truly reducing the number of failures under present testing conditions. An analysis of the failures for the most recent quarterly sample of this series indicates collector-emitter shorts to be the prime mode of failure. There are further indications that the $BV_{CEO}$ of the devices are being exceeded with the 7.5 volts used for the 150 mw operating life test. Operating step stress data has shown a larger number of failures result when the $BV_{CEO}$ of the devices is exceeded with the applied voltage. The failures from the devices run at 7.5 v and 20 ma were not of a degradation nature but rather a device abruptly changing from a good unit to a collector-emitter short.

Life tests run on a special device series taken from this product line, which do not require power aging, have shown 0 - failures from three separate samples of 150 devices (total of 450 units) at 500 hours when the life test is conducted at 6 v and 25 ma as opposed to the 7.5 v and 20 ma proposed in this work.

In view of the above comments careful consideration must be made as to merits of adding a power aging requirement to the processing of this series.

**SUB ITEMS**

The fourth group of engineering samples have been shipped. Test data on this group of units is shown in Figure 2.
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**FIGURE 2**
SECTION IV - 2N964

OTHER WORK

STRIPE ALLOYING  (Bob Eldridge)

The concept of "fast fire" alloying has been pursued during the last quarterly period of this contract work. Prototype equipment has been obtained and large scale tests have been run.

The prototype equipment permits the slices to be raised to the alloy temperature and cooled essentially to room temperature in less than one second.

The furnace is a resistance heater element type and is equipped with a quartz tube so that an ambient of forming gas may be provided during the alloy cycle. The furnace is inclined to provide a gravity feed for slices as they traverse through the hot zone of the furnace.

Test runs were made over a three weeks period, and data resulted which indicated the re-growth control over the entire slice area is difficult. The central portion of the slice appears to be alloyed at a lower temperature than the edges.

Devices fabricated from slices processed in this manner have shown favorable diode reverse characteristics. However; inconsistancy in forward diode drops across the slices indicates insufficient alloying over portions of the slices. Uniformity can be achieved through an additional, and more conventional, alloy cycle. Re-growth control during this latter step has been enhanced due to the high temperature, high speed exposure. Installation of "fast fire" alloying will occur for the 2N960 series when the system is
refined to such a degree where an additional alloy cycle is not required.

Work will continue on methods of accomplishing this type alloying. This work will be concerned with stripe composition and means of providing more uniform heating across the slice. Infrared heating has been tried in the past and will be further explored, also the feasibility of other concepts will be investigated.
Results of the life test evaluation on 300 units from each of the 1st and 2nd quarter contract periods are presented in this section of the quarterly report. First and 2nd quarter samples were from the production periods May - July 1962 and August - October 1962, respectively. Electrical parameters, test conditions and parameter limits are specified in Figures 2.01A and 2.01B of Texas Instruments Quarterly Progress Report Number 2.

**FAILURE RATE VERSUS TIME**

In Figure 3 life test results for both quarterly samples are presented in the form of cumulative percent failure versus time curves with each failure mode indicated by a separate curve. The top plot, May - July 1962 production, shows $I_{EBO}$ to be the predominant failure mode. In addition to the $I_{EBO}$ increases, $h_{FE}$ degraded severely from the initial reading. In the August - October sample $I_{CBO}$ was the only mode of failure. In contrast to the 1st quarter units, $h_{FE}$ showed an increase from the pretest values.

According to Production Engineering differences between the 1st and 2nd quarter test results were due, primarily, to major process changes made during the two production periods. Because of these process changes the 1st sample was subject to E-B shorts and the 2nd sample to C-E shorts. This is discussed further in the failure analysis section which follows.
Units going into the 3rd quarter sample will be from a process which differs from the two previous ones. Because of these changes in processes, no attempt will be made to fit the data to any of the possible failure distributions.

**FAILURE ANALYSIS**

Failure analysis was performed on a representative sample from each quarter. A copy of the report is included in the appendix. In both cases there was no evidence of hermetic seal problems or surface contamination. All failures were attributed to an electrical overstress. Three possible reasons for overstress have been considered: (1) Overstress caused by operating the devices above the collector-emitter breakdown voltage, (2) Overstress because the basic process produced devices susceptible to E-B or C-E shorting, and (3) Overstress due to RF in the life test circuits. To determine if the overstress in (1) above existed, samples from both production periods were measured on $BV_{CEO}$ at 25°C and 75°C. Results are shown in Figures 4 and 5. These plots show that more than 50% of the devices were operated above their breakdown voltage at $V_{CB} = 7.5$ v. This fact was discussed with the Signal Corps and the 3rd quarter sample will be tested at $V_{CB} = 6$ v, $P_C = 150$ mw. Overstress due to stray RF pickup in the life facility are also being considered as a possible cause of failure. Some positive steps are being taken. Filtering is being added to eliminate the stray RF which might be getting into the transistor circuit. Also, an extra 100 3rd quarter units will be placed on test in TI's battery operated life racks for comparison with results from the 300 units in the Centralized Automatic Reliability Testing (CART) facility.
PARAMETER CHANGE VERSUS TIME

In view of the new life test conditions and major changes in the process, these plots will be omitted until 3rd quarter results are available.
SECTION VI - 2N964

CONCLUSIONS

Control of the epitaxial deposition has remained satisfactory during the past quarter. The Perkin-Elmer 221 Infrared Spectrophotometer provides an excellent method for monitoring the epitaxial film thickness deposited.

Diffusion control has remained in satisfactory control throughout the past quarter. During the re-design of the evaporator tooling, geometric problems have arisen in the ability to hold acceptable dimensions and tolerances over the desired area of the slice. Present evaporation techniques have improved in that operators have become more proficient.

Mesa and Dice cutting were satisfactory at the close of this quarter.

Coating techniques for protective purposes and passivation still do not appear possible and practical for this device series by the close of this contract work. Work has continued on improving the encapsulation technique.

Steps have been taken to enhance the cleaning operations in the process steps, and the formation of an oxide on the finished devices.

Piece Part Incoming Inspection Procedures completed. Revision of the Process Control Procedures virtually completed during the last quarter.
The major areas of engineering effort will include:

- Monitoring of long term control of epitaxial deposition
- Encapsulation technique
- Evaporation control
- Revision of life testing procedures to eliminate over stressing
- Review and revision of Process Control
- Preparation of the required quality control manual
Two groups of 2N964 units which had failed 150 mw operating life at $V_{CB} = 7.5$ v were received for analysis.

The twelve units which failed $I_{CBO}$ and $BV_{CBO}$ were manufactured by the same process. Six of these units failed after 500 hours, and six failed these same tests after 1000 hours.

The 13 units which failed $I_{EBO}$ and $BV_{EBO}$ after 250 hours were manufactured by a process different from that used for the 12 units above.

The twelve $BV_{CBO}$ failures had no hermetic seal leaks. The units were checked electrically, and high $I_{CBO}$ and low $BV_{CBO}$ readings were confirmed. $BV_{CEO}$ measurement indicated that the units were practically shorted.

The units were decanned and inspected, and no manufacturing defects were apparent.

Electrical readings taken after the units had been etched, rinsed and baked showed no appreciable improvement.

It was concluded that the failure of these units was caused by collector-emitter punch through which was induced by electrical overstress.
C-E punch through was confirmed by microsectioning.

Thirteen $I_{EBO}$ and $BV_{EBO}$ failures also had no hermetic seal leaks.

Electrical parameter measurements showed that 12 units were out of specification limits on $I_{EBO}$, $BV_{EBO}$ and $h_{FE}$. The 13th unit, No. 26, was beyond limits on $I_{EBO}$, $I_{CBO}$ and $BV_{CBO}$. This unit and two others read practically shorted $BV_{CEO}$.

Microscope inspection after decanning revealed that 7 units had contact stripes extending to the mesa edge, so that failure might be expected from electrical stress at a level lower than normally required.

The remaining six units, however, had no apparent manufacturing defect, and five of these exhibited the same practically shorted emitter-base junction as did the seven misplaced stripe units. It appears that, in this case, the misplaced stripes, although not desirable, had no significant effect on failure of these units.

Etching, rinsing and baking after decanning failed to improve electrical readings.

Failure of these units is attributed to electrical overstress causing a punch through between emitter and base in twelve units, and between emitter and collector in the remaining one.

Since breakdown voltages of this device are somewhat low, it would be relatively easy for damage to be caused by transients in testing circuits, etc.
It is suggested that test equipment and procedure be evaluated for possible sources of damaging electrical stress and that wafer inspection be tightened to insure removal of units with physical defects such as improperly placed contact stripes.
ABSTRACT

This report covers all work performed during the third quarter after the effective date of the contract.

During this report period one group of engineering samples was shipped as per the contract requirements.

The major areas of engineering activity during this report period were on the following process steps:

1. Epitaxial material.
2. Cleanliness prior to high temperature diffusion.
3. Photo masking.
4. Lead attachment.
5. Contact materials.
The purpose of the work provided in this contract is:

1. To establish process improvements necessary to attain improved operating failure rate.
2. To incorporate the process improvements into the production facilities.
3. To supply engineering test samples incorporating the improved processes.
4. To provide quarterly reports, final engineering report, bills of materials and parts, and general report.
This section covers the engineering progress during the third quarterly report period of this contract. Tables 0.02 and 0.03 show the proposed schedule of work which was presented at the Pre-Production Planning Conference.
<p>| STUDY | 1962 | | | | | 1963 | | | | |
|-------|------|---|---|---|-----|---|---|---|---|---|---|
| 1. EPITAXIAL MATERIAL | | | | | | | | | | |
| a. Measurement techniques | | | | | | | | | | |
| b. Substrate surface | | | | | | | | | | |
| c. Layer uniformity | | | | | | | | | | |
| d. Crystal perfection | | | | | | | | | | |
| 2. CLEANLINESS PRIOR TO HIGH TEMPERATURE OPERATIONS | | | | | | | | | | |
| a. Improved electrical characteristics | | | | | | | | | | |
| b. Less spiking | | | | | | | | | | |
| 3. CONTROL OF DIFFUSION | | | | | | | | | | |
| a. New Sources | | | | | | | | | | |
| 1. Bo | | | | | | | | | | |
| 2. Doped gases | | | | | | | | | | |
| b. Control of conditions | | | | | | | | | | |</p>
<table>
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<tr>
<th>STUDY</th>
<th>1962</th>
<th>1963</th>
</tr>
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<tbody>
<tr>
<td>4. PHOTO-MASKING: ETCHING</td>
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<tr>
<td>a. Control of photo-resist properties</td>
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<tr>
<td>b. New photo-resist materials</td>
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<tr>
<td>c. Projection techniques</td>
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<tr>
<td>d. Developing methods</td>
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<td></td>
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<tr>
<td>e. Oxide etches</td>
<td></td>
<td></td>
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<tr>
<td>f. Photo-resist removal</td>
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<tr>
<td>5. HEADER IMPROVEMENT</td>
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<tr>
<td>a. Plating</td>
<td></td>
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<tr>
<td>b. Cleaning</td>
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<tr>
<td>6. LEAD ATTACHMENT</td>
<td></td>
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<tr>
<td>a. Ball bonding</td>
<td></td>
<td></td>
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<tr>
<td>7. CONTACT MATERIALS</td>
<td></td>
<td></td>
</tr>
<tr>
<td>a. New metals</td>
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</tr>
</tbody>
</table>
EPITAXIAL MATERIAL

(P. Shankle)

LAYER UNIFORMITY

Data taken by the infrared reflective technique indicates a variation in the epitaxial layer of approximately 5% across the slice and 5% from slice to slice and run to run. Since the measuring equipment has an accuracy of ± 2%, the controllability of layer thickness is excellent.

SUBSTRATE SURFACE

A high temperature vapor etching process is being investigated with initial results looking very promising. Stacking faults and etch-pit count levels have been lowered drastically. A large percentage of production material is being run utilizing this technique in order to fully evaluate the process.

RESISTIVITY MEASURING TECHNIQUES

Experiments to measure N on N+ resistivity 100% by probe methods have been unsuccessful. No reliable non-destructive method has been established as of yet. Experiments are continuing on this project.
CLEANLINESS PRIOR TO HIGH TEMPERATURE DIFFUSION
(P. Shankle)

As was stated in the previous report the introduction of a hot acid clean-up prior to high temperature diffusion was very effective in removing minute photo resist particles and other contaminants from the surface of the slice, thereby increasing diffusion yields by a factor of approximately 10%. The results of the hot acid wash resulted in a study of the feasibility of an automatic or semi-automatic slice cleaning machine. The machine has been determined feasible, design has been finished, and the machine is presently being assembled on the production floor.

The basic operation of the machine consists of a walking beam type mechanism which will carry the slices through two hot acid washes, two hot deionized water rinses and finally into a dry box system for storage in deionized water until the slices are ready to be baked dry. (See Figure 1.) The addition of this machine is expected to produce a considerable increase in yield and reliability.
WALKING BEAM ACID SLICE CLEANING
MACHINE AND DRY BOX SYSTEM

Operation Sequence:

1. Slices loaded from petri dish to acid carrier.
2. Carriers wait for removal from acid machine.
3. Carriers removed from machine and placed in D.I. bath.
4. Carriers stored in D.I. bath until loaded to furnace boat.
5. Slices loaded from carrier to furnace boat and placed in furnace drying tube.
6. Slices dried in 100°-150° C furnace prior to loading in diffusion furnace.

Figure 1
WALKING BEAM ACID SLICE CLEANING MACHINE AND DRY BOX SYSTEM

OPERATION SEQUENCE

1. Slices loaded from petri dish to acid carrier.
2. Carriers wait for removal from acid machine.
3. Carriers removed from machine and placed in D.I. bath.
4. Carriers stored in D.I. bath until loaded to furnace boat.
5. Slices loaded from carrier to furnace boat and placed in furnace drying tube.
6. Slices dried in 100°-150°C furnace prior to loading in diffusion furnace.

Figure 1
DOPED GAS
(P. Shankle)

Diborane gas was first experimented with as a possible "P" type dopant. Difficulty was experienced in obtaining uniform surface concentration and also in preventing excessive surface damage. Rather than using a doped gas, the use of a liquid source was investigated.

The two solutions investigated most extensively during this quarter were Phosphorus Tribromide (PBr₃) and Boron Tribromide (BBr₃). The problems encountered using the above mentioned dopants were similar to the diborane gas results.

Excessive surface stain and damage as well as uncontrollable doping levels were the general rule with both dopants. Due to the high degree of control over the present 2N744 base and emitter diffusion process, the doped gas and liquid dopant investigation has been terminated.
PHOTOMASKING / ETCHING (J. Peoples)

1. CONTROL OF PHOTO-RESIST PROPERTIES

This study has been completed. A pure (with respect to foreign particles) resist of known viscosity is now employed routinely in 2M744 photomasking processes.

2. NEW PHOTO-RESIST MATERIALS

Based on the results seen in controlling photo-resist properties together with the speed and quality of exposure now being obtained, it is felt that the fabrication of new photo resist materials is no longer necessary. This aspect of the program will be discontinued.

3. PROJECTION TECHNIQUES

An investigation of a commercial photo resist which is reportedly sensitive in the visible region of the spectrum has been discouraging. It is thought that such a resist system will be a pre-requisite to projection exposure techniques. To date experiments with the visible system indicate insufficient masking, both from the standpoint of etch resistance and pattern definition.

4. DEVELOPING METHODS

Completed.

5. OXIDE ETCHES

Experiments concerning the action of buffers in etch systems are being conducted. The object of these and other tests involving oxide etch systems is to try to understand the conditions which promote controlled etching of oxides. At this time no conclusions can be drawn.

6. PHOTO RESIST REMOVAL

Completed.
HEADER IMPROVEMENT
(P. Shankle)

An experiment to establish the identity of impurities that are outgassed from headers at elevated temperatures was made during this quarter.

The equipment for this experiment consisted of a vacuum system and a pyrex cylinder. The procedure for the experiment was as follows:

1. The header to be tested and the pyrex system were extensively cleaned.

2. The header was placed in the pyrex cylinder and a vacuum of approximately one micron was applied. A small amount of helium was introduced into the system.

3. The system was heated by R.F. heating to approximately 300°C in an attempt to have the outgassed impurities induce a glow discharge with the helium.

4. An emission spectrometer was then used to attempt to identify the outgassed impurities.

CONCLUSION

Sufficient cleaning of the pyrex system was never achieved to a degree that a good reference spectograph could be made. Without a reference point accurate measurement of the impurities is impossible. The system is being modified in an effort to overcome the difficulty.
Efforts to ball bond to the 2N744 geometry have been stymied by the small distance between the emitter and base contact area (1 mil) and the center to center distance from the emitter to base contact area of 3.0 mils. Due to the small distances involved a high volume ball bonding production process is extremely hard to control. (See 2nd Quarterly Report). In view of earlier experiments the approach was taken to change the present geometry in such a way to better facilitate a larger ball bond, but not change any electrical characteristics of the device. This was achieved by keeping the original base geometry and offsetting the emitter geometry. (See Figure 2). By using this geometry a successful and easily reproduced ball bond was achieved using one mil gold wire. The units built with the split contact geometry revealed no change in parameter distribution and after 500 hours of 300°C storage the ball bonded units revealed no failures or parameter distribution shift. The one adverse effect was the formation of AuAl₂ between the gold wire and the aluminum contact.

CONCLUSION:

While ball bonding to the revised 2N744 geometry has been proven feasible so far as the bonding operation is concerned, the formation of AuAl₂ (purple plague) is certainly undesirable. For this reason ball bonding will not be included into the process until a suitable contact material that does not form a dendritic growth between the wire and contact is found.
I. INTRODUCTION: As stated in the second quarterly report the advent of planar technology has intensified the need for an improved ohmic contact to silicon. Aluminum, which is the most widely used ohmic contact material now used in industry, has one major drawback, this being the formation of AuAl$_2$ (purple plague) which forms when the gold wire is brought into intimate contact with the aluminum contact. Since gold wire is the easiest material to use for the formation of ball bonds, the course being most intensively pursued is an improved contact which will give good ohmic contact, eliminate the dendritic AuAl$_2$ growth, and be a reproducible production process. Pd-Au systems were discussed in the second quarterly report and while showing some success the system appears to be undesirable so far as this device is concerned.

The chromium-gold system will be discussed in this report. This system was chosen for the following reasons:

1. The adhesion of chromium to silicon is remarkably good.
2. Chromium is reported not to react or dissolve appreciably in silicon at temperatures as high as 1000°C.\(^1\)
3. Gold is reported not to react with or dissolve in chromium at temperatures below 1000°C.\(^2\)
4. The chromium-gold ohmic contact is resistant to attack by chemical solutions containing HNO$_3$ acid (aqua regia excepted) and thus provides an opportunity for performing rigorous surface clean ups.
5. The chromium-gold ohmic contact is easily and inexpensively applied by evaporative techniques.

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2. Ibid., pp 196-197
II. **PROCEDURE:** The following flow chart describes in detail the procedure followed in applying the chromium-gold contact system in the fabrication of planar silicon transistors:

1. **Apply reverse contact KMER.**

2. **Remove oxide from emitter and base contact areas.**
   a. Oxide etch for 2 min., rinse with DI water, blot dry.
   b. Inspect KMER for adhesion and undercutting.
   c. Continue to remove oxide with oxide etch and inspect at regular intervals until all oxide is removed.
   d. Rinse with DI water, swab with methyl alcohol. **DO NOT REMOVE KMER.**

3. **Evaporate chromium.**
   a. Load chromium plated or unplated Tungsten filament with pure chromium.
   b. Evacuate to $10^{-5}$ mm pressure and then back-fill with He and re-evacuate.
   c. Degas the chromium source by heating filament with shutter over silicon slices.
   d. Heat substrate to 140°C and evaporate chromium on silicon.
   e. Control chromium thicknesses to 3-6 microinches.

4. **Evaporate gold.**

   Without breaking vacuum, evaporate gold in the same manner as described above. The final gold thickness is 7-12 microinches.

5. **Remove excess chromium gold and underlying KMER by the "blister peel" method.**
a. Heat slice on a hot plate until the KMER begins to blister.
b. Swab with hot trichloroethylene.
c. Repeat 5, steps a and b, until all KMER is removed.


III. RESULTS: The chromium-gold contacts were applied to devices of the 2N1893 series in the manner described in Section I. The results from several runs are summarized in Table IV. The 2N1893 was used in place of the 2N744 because a successful ball bond using gold wire is presently being used on this device. A successful ball bonding procedure had not been established for the 2N744 when these experiments were performed.

TABLE IV

V<sub>BE</sub> Measurements on 2N1893 Transistors with Cr-Au Contacts to Emitter and Base Region.

<table>
<thead>
<tr>
<th>Run #</th>
<th>Source</th>
<th>Cr Thickness</th>
<th>Au Thickness</th>
<th>I&lt;sub&gt;C&lt;/sub&gt; = 200 ma</th>
<th>I&lt;sub&gt;B&lt;/sub&gt; = 15 ma</th>
<th>Bondability</th>
</tr>
</thead>
<tbody>
<tr>
<td>29 A</td>
<td>Plated</td>
<td>4</td>
<td>10</td>
<td>1 - 1.2</td>
<td>100%</td>
<td></td>
</tr>
<tr>
<td>30 A</td>
<td>Plated</td>
<td>5</td>
<td>8</td>
<td>1 - 1.2</td>
<td>100%</td>
<td></td>
</tr>
<tr>
<td>32 A</td>
<td>Plated</td>
<td>6</td>
<td>7</td>
<td>1 - 1.1</td>
<td>100%</td>
<td></td>
</tr>
<tr>
<td>33 A</td>
<td>Plated</td>
<td>6</td>
<td>7</td>
<td>1 - 1.1</td>
<td>100%</td>
<td></td>
</tr>
<tr>
<td>34 A</td>
<td>Chunks</td>
<td>3</td>
<td>7</td>
<td>1 - 1.1</td>
<td>100%</td>
<td></td>
</tr>
<tr>
<td>35 A</td>
<td>Plated</td>
<td>5</td>
<td>7</td>
<td>1 - 1.1</td>
<td>100%</td>
<td></td>
</tr>
</tbody>
</table>

The completed 2N1893 Transistors were placed on 300°C storage and tested. The data are summarized in Table V.

TABLE V.

Life Test Data on 2N1893 with Chromium-Gold Contacts to Emitter & Base Region

<table>
<thead>
<tr>
<th>Initial</th>
<th>After Centrifuge</th>
<th>After 10 Hrs.</th>
<th>Heat Age @ 300°C</th>
<th>Heat Age @ 300°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>V&lt;sub&gt;BE&lt;/sub&gt;</td>
<td>V&lt;sub&gt;BE&lt;/sub&gt;</td>
<td>V&lt;sub&gt;BE&lt;/sub&gt;</td>
<td>V&lt;sub&gt;BE&lt;/sub&gt;</td>
<td>V&lt;sub&gt;BE&lt;/sub&gt;</td>
</tr>
<tr>
<td>I&lt;sub&gt;C&lt;/sub&gt; = 150 ma</td>
<td>I&lt;sub&gt;C&lt;/sub&gt; = 150 ma</td>
<td>I&lt;sub&gt;C&lt;/sub&gt; = 150 ma</td>
<td>I&lt;sub&gt;C&lt;/sub&gt; = 150 ma</td>
<td>I&lt;sub&gt;C&lt;/sub&gt; = 150 ma</td>
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<tr>
<td>I&lt;sub&gt;B&lt;/sub&gt; = 15 ma</td>
<td>I&lt;sub&gt;B&lt;/sub&gt; = 15 ma</td>
<td>I&lt;sub&gt;B&lt;/sub&gt; = 15 ma</td>
<td>I&lt;sub&gt;B&lt;/sub&gt; = 15 ma</td>
<td>I&lt;sub&gt;B&lt;/sub&gt; = 15 ma</td>
</tr>
<tr>
<td>0.96</td>
<td>0.96</td>
<td>0.96</td>
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<td>0.97</td>
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<td>0.96</td>
<td>0.97</td>
<td>1.0</td>
<td>0.97</td>
<td>1.0</td>
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</tbody>
</table>

43
IV. DISCUSSION: The results presented in this report suggest the possibility of successfully using the chromium-gold system for ohmic contact to planar transistors. Although the exact mechanism and metallurgy of bonding to the chromium-gold contact is still uncertain, it appears that the relatively porous chromium film sandwiched between the gold and the silicon controls and/or limits the alloying of the gold with silicon.

The controlled alloying of gold to the silicon is not only desirable but appears to be essential for bonding and good electrical properties. The evidence for the alloying mechanism is provided from the physical appearance of the contacts before and after sintering. Furthermore, there is a good correlation between bondability and physical appearance of the contacts. Successful bonding is accomplished only when there is direct visible evidence of alloying.

It has been observed that in some cases there is a pronounced difference in the bondability to the emitter and base region for the same chromium and gold thicknesses. This difference is attributed to differences in the alloying characteristics resulting from variation in surface concentration and type of impurities introduced in the emitter and base region during diffusion. The difference between the emitter and base behavior is further manifested in the appearance of stain films (etch burns) during the oxide removal step. It was observed that stain films formed much more readily in the emitter area. The formation of stain films was eliminated by modifying the etching procedure so as to include a quench in HNO₃ acid following the oxide etch.

In summary, the alloying mechanism for bonding, proposed above, requires strong dependence of bondability on chromium film thickness, temperature and duration of sintering, and substrate properties. The most successfully bondable slices are those that have a relatively thin chromium film, and have relatively lightly doped substrates.

Reproducible results could only be obtained by evaporating the chromium-gold film selectively to the contact areas using KMER as a mask. It was found that the alternate procedure involving reverse KMER and an acid leach step lead to bondability problems. The bondability problems resulted from undercutting of the contacts during the acid leach operation to remove excess chromium-gold. Severe undercutting of the chromium resulted in an uncontrolled alloying of gold and silicon and in some cases complete disappearance of the gold.
V. SUMMARY AND CONCLUSIONS:

1. The feasibility of using chromium-gold ohmic contacts to the emitter and base areas of silicon planar transistors is demonstrated.

2. A mechanism for bonding to the chromium-gold contact is suggested.

3. The chromium-gold ohmic contact is shown to possess desirable electrical and mechanical properties.

4. No dendritic growth was observed with this system.

5. Extreme control of all conditions is necessary if this system is to be used in production.
NEW PROJECTS
(P. Shankle)

HISTORY:
An important D.C. parameter on all switching transistors is $V_{CE}$(Sat), which is the minimum switch contact potential. Since collector resistance plays an important part in the final value of $V_{CE}$(Sat), some means must be used to lower it. Two processes now in use to lower the collector resistance are:

1. A N+ diffusion is put into the collector side of the slice during the emitter diffusion. This means that the slice must be lapped to its final thickness prior to emitter diffusion. The normal slice thickness is from .003 to .005 inches and a great deal of slice breakage is incurred if the slice is lapped this early in the manufacturing process.

2. A more desirable method of lowering collector resistance is to use a gold preform that has a small amount of "N" type material, such as antimony, added to it. This process works satisfactorily, but problems do arise such as tilted wafers and excess gold smearing on the glass eyelets of the header during the alloying process.

Experiments were run using evaporative techniques to apply gold-antimony to the collector side of the slice. Difficulty was experienced with the gold peeling off the slice during scribing and with some wafers not alloying properly to the header. While this process is feasible, it must be refined before it can be used in production.
SECTION IV
RELIABILITY ENGINEERING EVALUATION - 2N744
Wayne Murdock

This section of the report presents the 2000 hour life test results for 300 units from each of the 1st and 2nd quarter contract periods. The first sample came from May - July 1962 production and the second sample from August - October 1962 production. Devices were life tested at rated power as stated in Quarterly Progress Report Number 2, Figure 8.01B. Parameters measured, test conditions, initial limits and post test limits for the program are shown in Figure 8.01A of the above report.

In Quarterly Progress Report Number 2 several methods of data presentation were proposed for each group of 300 units. Some of these methods depend upon enough failures being generated to define the failure distribution graphically. Where too few failures occur to perform the indicated method of analysis, these particular plots will be omitted.

Failure Rate Versus Time

Three ICES failures occurred during the life tests, one from the 1st quarter sample and two from the second quarter sample. All failures were marginal; i.e., slightly over the 100 nanoamp post test limit. Weibull or Log - Normal plots would not be meaningful based on this number of failures. A plot of ICES versus time for these three units is given in Figure 3.

A calculation of failure rate, using the post test limits as previously noted, gives a point estimate of .25%/1000 hours at rated power. This is obtained by dividing 3 failures by 1.2 million test hours. Choosing a more realistic end point limit of > 1 uA for ICES gives a point estimate of 0%/1000 hours.

Parameter Change Versus Time

In Figures 4 - 5 the median, minimum and maximum values, for each of the 5 parameters,
are plotted against test time in hours. Median values are stable for all parameters except hFE which shows a slight increase the first 250 hours and then a decrease with time. The amount of decrease in hFE seems to be linear with time.

Plots like those above are presented in Figures 6-7 for the second quarter data. All parameters are stable including hFE.

Failure Analysis
The failed device from the first quarter sample will be sent to the Failure Analysis Laboratory at the end of 4000 hours on life test. Additional failures which occur between 2000 and 4000 hours will also be analyzed. Life tests were extended to 4000 hours on this sample so a direct comparison could be made between it and the final sample taken after completion of the 12-month product improvement program. The two failures from the 2nd quarter sample are awaiting laboratory analysis.

Results of the analyses will be included in the next quarterly report.

Failure Rate as a Function of Different Post Test Limits
Since all three failures were only slightly above 100 nanoamps, which is a very tight limit, a table of failure rate versus various post test limits would be of no value.
Figure 4
Figure 5
Figure 6
Figure 7
SECTION V
CONCLUSION

A non-destructive test to measure the resistivity of epitaxial layers has not been developed. While control of layer thickness and resistivity is excellent from empirical results, a non-destructive test must be developed before a satisfactory production process is established.

The automatic slice cleaning machine will be operation shortly. Yield and reliability are sure to be improved by the addition of this machine.

The split contact geometry to facilitate ball bonding has been proved feasible. This will not be incorporated into the process until a suitable contact material is found that will eliminate the AuAl₂ growth.

Photo resist techniques are under excellent control. Efforts to utilize projection type exposure methods have been unsuccessful due to present photo resist in use.
SECTION VI

PROGRAM FOR NEXT INTERVAL

1. Improve contact material.

2. Resistivity measuring techniques on epitaxial material.

3. Improve oxide etching technique.