FINAL REPORT

DEVELOPMENT OF IMPROVED TEST STANDARDS FOR MONOLITHIC CIRCUITS

6-5-69 - 6-5-70

TRW Report 13512.6001-T-0-00

Prepared for

George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama
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June 30, 1970

Prepared by Murray Bloom

James J. Egan
Microelectronic Arrays Department

TRW SYSTEMS
ABSTRACT

The influence of the metal deposition process, the profile of the oxide edge, and the sintering process on the probability of microcrack formation were examined. It was shown that as long as aluminum is deposited by the usual evaporation methods the potential for microcrack formation will always be present. The profile of the oxide edge was measured using a scanning electron microscope. It was found that, for a variety of types of silica masked with KTFR and etched with buffered oxide etch, the wall angle was not 45° as would be expected but rather 34° ±3°. Sintering experiments strongly suggested that microcrack formation proceeds by surface diffusion driven by crystallite regrowth. It was concluded that for any combination of edge profile and evaporation technique, failure is more likely to occur if sintering conditions are severe.

A distinction was drawn between intrinsic and extrinsic failure under electromigration conditions. For typical conductors the error contributed by dust or dirt on the mask or slice or in the photoresist, and the error contributed by assuming the stripe has a rectangular cross section were each about 20%. The experimental results obtained were in agreement with Black's model as were the energies of activation 0.44 eV and 0.41 eV for stripes of 1 mil and 0.5 mil nominal width, respectively. A new failure mechanism in which the failure of one conductor due to electromigration results in the destruction of a conductor running parallel to it was identified.

The usefulness of electromigration as a monitor of the totality of the influences of all the processes was stressed.

A post-etching treatment was found which, while not optimized, increased the lifetime of aluminum conductors: heating in nitrogen for three days at 350°C increased the expected lifetime by a factor of 4.7.

Methods Nos. 6070-5 of "Test Standards for Microcircuits" have been examined and errors and replacement of certain of the Methods by new ones related to thickness, uniformity, and adhesion of metallization and quality of ohmic contact were suggested and Proposed Test Methods written.
A new method of etching steps for use in the interferometric method of determining thickness was devised. A quantitative method for determining the adhesion of aluminum films which involved moving the sample under a fixed stylus carrying a variable load was described. The Proposed Test Methods were applied to three production lots and the results tabulated.
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1. INTRODUCTION

1.1 Purpose

The production of monolithic microcircuits involves a sequence of processing steps each of which demands a particular degree of control in order to realize a certain percentage yield. If this degree of control is not held, the result will be felt as a diminished yield. This type of lack of adequate control can be fairly quickly realized and corrective actions taken. The result of another type, more subtle, and to the user, more important, is related to the reliability of the device. If the degree of control necessary for a certain level of reliability is not met, the result may not be felt as diminished yield. In fact, it may not be felt at all by the manufacturer, and it may not be felt by the user until sometime after the device is placed in operation. What is needed is a collection of test procedures which can be applied during the processing sequence, or very soon afterward, which will indicate the degree of control of the various processing steps. To a certain extent, all manufacturers use such controls in order that the percentage yield does not falter. However, in general, these tests are not sensitive enough, or are not applied frequently enough, to detect changes which may affect the reliability of the device. It is the purpose of this study to improve this situation insofar as the quality of metallization is concerned.

The quality of metallization is not influenced solely by the metal. It is also influenced by the oxide, or more specifically, by how the oxide is etched.

1.1.1 Possible mechanisms for metallization failures include the following.

1.1.1.1 There may be residual oxide in the contact window.
1.1.1.2 The angle between the wall of the contact hole and the original oxide surface may be too great to allow evaporated metal to form a continuous path from the top of the oxide to the wall.
1.1.1.3 Pile-up of the oxide, where it is deposited over a metal stripe (as in a multi-level interconnection scheme), may be of such a form as to prevent the evaporated metal from forming a continuous path over the oxide.\(^{(1)}\)

From the standpoint of yield a test procedure which detects the lack of a continuous path of metal is sufficient; from the standpoint of reliability it is not. A metal path which has places of reduced cross-sectional area will be prone to failure in use as will be seen from the discussion which appears elsewhere in this report.

1.1.2 Metal-related factors which can influence the quality of metallization include:

1.1.2.1 Deposition Parameters - Factors 1.1.1.2 and 1.1.1.3 just listed can be rendered less important if the pressure during deposition is made high. On the other hand, high pressure can more easily give deposits with lower conductivity. Similarly, other deposition parameters such as substrate temperature, deposition rate, deposit thickness, purity of the aluminum source, background pressure prior to the deposition, or oxygen content of the deposition ambient can affect the quality of metallization mainly via influence on the grain size.

1.1.2.2 Photoresist - The quality of metallization is influenced by the photoresist mainly through variations in line width and pinholes. The line width in turn can be affected by the exposure time, type of development, and nature of the post-development bake.

1.1.2.3 Etching - Etching exerts its influence in the same two ways as photoresist: by influencing line width because of under or overexposure or by producing pinholes where defects exist in the photoresist.

Thus, this program is concerned with tests which can monitor the results of changes in oxide etching, deposition parameters, photoresist operations and etching procedures. Ideally, it should be possible to apply these tests at the same time, or immediately after, the process which is being monitored has been performed.
1.2 **Scope**

This project has been restricted to aluminum deposited by evaporation in spite of the facts that: (1) some other metals show greater resistance to failure via electromigration and (2) certain other deposition systems are much better than evaporation with respect to lack of sensitivity to the substrate contours. There are many advantages to the use of aluminum as compared to other metals and these have been reviewed by Schnable and Keen. (2)

We first studied Methods 6070-5 in "Test Standards for Microcircuits" (3) and evaluated them in order to determine the necessity for modifications or additions to them. We then examined the parameters involved in controlling the factors mentioned in the previous section and from this we developed new tests and modified some of the old ones.

We devoted special attention to certain aspects of the "microcrack problem" and to electromigration.

Finally, the tests which we either originated or modified were applied to three production lots and the results evaluated.

2. **EXISTING TEST METHODS**

2.1 **Brief Description**

The full text of the existing Test Methods 6070-5 can be found in Appendix I, to which appendix the page numbers refer. The following tabulation will be sufficient to display the scope of these methods.

2.2 **Comments**

In the following section the page numbers 264-315 refer to the Existing Test Methods which are to be found in Appendix I.

2.2.1 **Method 6070A: Evaluation of Metallization Thickness and Uniformity**

The first line of p. 264 reads as follows: "A control of ±20% of the design thickness is adequate unless otherwise specified." The second sentence of paragraph three on the same page reads as follows: "The precision with which the monitor cutoff defines the point at which the design thickness of aluminum is obtained and the uniformity thereof is determined by the displacement of the interference fringe pattern to
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+0.5 fringe of sodium light." Thus, if the thickness of the film is less than 7370 Å the error in the design thickness produced by reading the interference fringe pattern to +0.5 fringe will be more than 20%.

There is no reason for reading the fringe pattern to only +0.5 fringe. The pattern shown as Figure 3, p. 269, can easily be read to +0.2 fringe thus enabling the method to be used for films as thin as 2950 Å. If care is taken with the preparation of the surface, and if a reflective layer is deposited on the step, the method is capable of even greater precision. However, this involves certain techniques which are discussed in Test Method 6070B and will be treated later.

Figure 4 on p. 270 contains an equation which is incorrect and in addition gives the wave length of sodium light as 2946 Å instead of the correct value of 5890 Å.

It is felt that Test Method 6070A could be deleted without significant loss.

2.2.2 Method 6070B - Thickness of Metallization

Certain aspects of this Test Method are useful but the specification of the Fairchild design interferometer jig is an unnecessary limitation. It would appear better to specify an apparatus such as the Varian "A-Scope" or the Sloan "Angstrometer" which incorporate in one unit a microscope, camera, interferometer, monochromatic light source, and jig.

The chief disadvantage to the technique is the time which must elapse between receiving the samples to be evaluated and the actual evaluation. One must first etch a step in the sample. This involves masking part of the sample, etching, rinsing, and removal of the mask. (For the mask material specified in the Test Standard, namely, Apiezon wax, the step produced with most metals is so abrupt that the fringes cannot be followed up the step and hence the fringe order cannot be determined.) It appears that a better way is to produce the wedge shaped step during the deposition by means of a wire mask such as shown in Figure 2, p. 268 of Method 6070A.

After forming the step one must then apply a reflective layer, either aluminum or silver according to the Test Method. This means that one must tie up the evaporator, or at least be able to put the sample in an evaporation run which is going to deposit the required film. In
any event, this causes still more time to elapse. (We have found that, when sodium light is to be used, a thin layer of copper is quite satisfactory as a reflective film. Moreover, this layer can be applied by sputtering in which case, if the sputtering apparatus is available, only about an hour is required for this step.) After the reflective layer has been applied the actual measurement can begin.

The object of this recitation has been to illustrate that the interferometric method is a slow one. Furthermore, for best results, an optically flat substrate should be used. Thus, in general, the measurement is of the thickness of the metal on the test substrate and not the actual device wafers. It would be better to test the device wafers themselves in order to be able to assess slice-to-slice differences.

2.2.2.1 Other Possible Methods - Probably the simplest way is to employ a stylus-type instrument such as the Taylor-Hobson "Talysurf" or "Talystep" or the Sloan "Dektak". The measurement could be made using the areas masked by the clip used to fasten the slices to the substrate holder in the evaporator.

X-ray and beta particle back-scattering methods have been suggested. Of these methods, x-ray fluorescence appears to be by far the most sensitive: R. P. Beatty and J. E. Cline have claimed a sensitivity of $46 \text{ Å}$ of aluminum. However, the equipment is expensive and not likely to be found in the usual manufacturing, as opposed to research, facility.

Beta particle back-scattering requires less expensive equipment but lacks sufficient sensitivity in the particular case at hand. In general the sensitivity depends upon the difference of between the atomic numbers of the substrate and the film being measured, a difference of 5 units being the minimum. Aluminum, with an atomic number of 13, is only one unit removed from silicon with an atomic number of 14. Thus, the method cannot be used for measuring aluminum films on silicon substrates. There remains the possibility of using beta particle back-scattering to measure aluminum on Pyrex substrates. To determine the feasibility of the method we need to know the "effective atomic number" for Pyrex.

The effective atomic number $Z_{\text{eff}}$ for a compound $G_xH_y$ can be calculated from
where $A_G$ and $A_H$ are the atomic weights of G and H, respectively. (15)

In the case of Pyrex glass we have a somewhat different situation because a glass does not have a definite molecular weight. However, we can proceed as follows. Commercial Pyrex brand glasses have compositions which are similar to the following:

- $\text{SiO}_2$: 80%
- $\text{B}_2\text{O}_3$: 14%
- $\text{Na}_2\text{O}$: 4%
- $\text{Al}_2\text{O}_3$: 2%

By dividing the percentage composition by the molecular weight of the corresponding oxide we get four numbers which stand in the following ratios.

$$\text{SiO}_2 : \text{B}_2\text{O}_3 : \text{Na}_2\text{O} : \text{Al}_2\text{O}_3 = 67:10:3:1$$

Thus the "empirical formula" of Pyrex is

$$\left(\text{SiO}_2\right)^6 \left(\text{B}_2\text{O}_3\right)^{10} \left(\text{Na}_2\text{O}\right)^3 \text{Al}_2\text{O}_3$$

or $$\text{Si}_{67}\text{B}_{20}\text{Na}_6\text{Al}_2\text{O}_{103}$$

From this we can calculate the molecular weight and then the effective atomic number.

The effective atomic number of Pyrex thus turns out to be 9 units, which while somewhat greater than the minimum difference required, is probably still not great enough to give anything better than marginally accurate results.

2.2.3 Method 6070C - Thickness of Metallization

The equation which appears in Section 1, p. 278 and in paragraph 4.6, p. 279, is correct only for a film which extends infinitely in all directions. In practical measurements the term $\pi/\ln 2$, which has the value 4.53, should be replaced by a term whose value depends upon the size of the substrate and where on the substrate the measurement is being made. If it is not made in the center, and if an accuracy of $\pm 10\%$ is desired, as stated in Sec. 6, p. 280, and the slices being measured are one inch in diameter, then the measurements must be made within a circle of 3/8 inch radius whose center coincides with that of the slice.
An even greater source of error is in the choice of a value for $\rho$, the bulk resistivity of the metal. It is well known that the resistivity of evaporated aluminum films seldom approaches the bulk resistivity by less than ten or twenty percent (for films 1 $\mu$m thick). Because the resistivity of the evaporated film is so sensitive to impurities it appears to us to be far better to use the sheet resistance in conjunction with an accurate thickness determination to calculate the resistivity of the film. We propose that Test Method 6070C be deleted insofar as it applies to the thickness of the metallization and that it be rewritten as a means of evaluating the resistivity of the evaporated film.

2.2.4 Method 6071A - Metallization Adherence

This Test Method contains the germ of a possible quantitative method for evaluating film adhesion. In Sec. '4, p. 283, the suggestion is made that: "... a somewhat more rigorous test is performed by using a degreased new razor blade or sharp metal probe to inscribe a cross-hatch pattern in [the] metallization and then proceeding as in ...". It was our thought that if the cross-hatch pattern were applied regularly using a ruling engine it might be possible to assign a quantitative value to the adhesion by counting the number of squares which had been removed by the tape. In order to assess the probability that such would be the case we examined some of the literature published on the nature of adhesion.

2.2.4.1 The Possibility of a Quantitative Scotch-tape Test - A recent paper (4) has discussed the failure mechanisms involved in the peeling of pressure sensitive tapes. As the tape backing is pulled the force is transmitted to the adhesive layer and then to the metal film. If the metal film substrate interface is weaker than the metal film-adhesive or the adhesive-backing interfaces then the metal film will be removed and the sample can be said to have "failed" the test. The authors recognize at least three types of failure. The two of most importance to the present discussion are:

- adhesive - separation at one of the two adhesive interfaces
- cohesive - separation within the bulk of the adhesive.

If failure due to any of these mechanisms takes place, the metal film will be considered to have "passed" the test. Thus it is important to know what affects these mechanisms.
Of the various factors investigated the following are of great importance to the present discussions:

1. Peel rate
2. Adhesive type
3. Molecular weight
4. Peel angle
5. Pressure of application.

2.2.4.1.1 The authors cite seven references which have reported irregular variations of peel strength with peel rate. This is shown in Figure 1 which has been adapted from one in their paper. In addition, the occurrence of large amplitude, regular oscillations of the force of peel at a given rate, a phenomenon known as "slip-stick" peeling means that even with constant peel rate the force applied to the evaporated film is fluctuating. Figure 3 taken from this paper dramatically illustrates this phenomenon. Therefore, it appears that a constant peel rate is necessary even though unobtainable, in the usual pressure sensitive tape test of metallization adherence, and that, even if it were obtainable, there still might be large fluctuations in the force applied to the metal layer.

2.2.4.1.2 The necessity of standardizing on a particular adhesive type was recognized by the originators of Test Methods 6071A and 6071B. Figure 1 shows how important this can be.

2.2.4.1.3 Even if the same manufacturer's tape is used each time, there is no guarantee that the adhesive used will always have the same molecular weight. Aubrey, et al have shown how the peel strength is influenced by changes in the molecular weight. Figure 2, which also has been adapted from one of theirs, shows this diagramatically.

2.2.4.1.4 Aubrey, et al, found that the peel strength is a fairly strong function of the peel angle when that angle is near 90°. Test Method 6071A specifies an angle of 60-90°. Figure 4, again adapted from the paper cited, shows that this is a particularly bad choice. Changing from a 60° to a 90° peel angle changes the peel strength by nearly 300%. A better peel angle would be around 120°.

2.2.4.1.5 Very surprisingly these authors found that the pressure of application was without effect on the peel strength providing no air bubbles were visible.
Figure 1. Factors Which Affect Relationship Between Peel Strength and Rate of Jaw Separation

Figure 2. Factors Which Affect Relationship Between Peel Strength and Rate of Jaw Separation
Figure 3. "Slip-stick" Peeling at Constant Pull Rate
Figure 4. Relationship Between Peel Strength and Peel Angle
2.2.4.2 We thus conclude that, at best, the pressure sensitive tape test can only be qualitative and that there are so many variables, many of which cannot be controlled, that the test is about as meaningful and convenient as scratching the deposit with a tweezers. Having thus demolished this straw man we must now propose his replacement.

2.2.4.3 Methods which involve attaching weights to the film have been considered and rejected because of the unknown influence of the adhesive on the adhesion strength of the metal film. In addition this type of experiment is time consuming because of the necessity of allowing the adhesive to cure in order to develop maximum bond strength. The test which we have proposed involves moving the sample under a fixed stylus whose load can be changed.

2.2.5 **Method 6071A - Metallization Adherence**

The comments made on Method 6071A apply to this Method as well.

2.2.6 **Method 6072A - Metallization Stability**

In Section 3, p. 292, the test duration is 1000 hrs. This is too long for a production monitor but might be satisfactory for application to selected lots. In case of serious trouble it might be necessary to apply an electromigration test to each lot. In order to be useful in this case the test must be performed under conditions which enable the aluminum film to be characterized in a day or two. Enough data has been published to enable one to predict that most aluminum conductors will show a mean time before failure of about 40 hrs when stressed at a current density of $10^6$ amp/cm² and a temperature of 200°C. Because of the statistical nature of failures, dependence upon one sample, or a few samples, must be avoided. We had originally proposed to rewrite this Test Method but decided against it for a number of reasons which will be found in Section 5, Electromigration.

2.2.7 **Method 6072B - Stability of Metallization**

2.2.7.1 Electromigration - The comments on Method 6072A apply to this test standard as well. In general this test standard is better thought out than the previous one. However, we object to having the metal film in contact with silicon at the constriction where the current density is greatest. This is because it is well known that the presence of
silicon catalyzes the formation of intermetallics and especially those whose physical and chemical properties can lead to failures. The test device is thus not realistic in that the metal contacts silicon at the region of greatest current density whereas in a well designed integrated circuit the metal contacts silicon at the contact holes where the area is such that the current density is at a minimum. Since an elevated temperature is specified, 200°, useful results at reasonable current densities can be obtained in a shorter time than in Test Method 6027A. The specified temperature is still low enough to allow structural differences to influence the lifetime.

2.2.7.2 Intermetallic Formation - Paragraph 4.2, p. 297 contains the statement: "Any slope greater than 20 \( \mu \) will indicate intermetallic formation". This choice of words is regrettable because, on the face of it, they seem to imply that the formation of intermetallic compounds of gold and aluminum will necessarily lead to high resistance connections. This is now known not to be true.\(^{(5)}\) It has been shown that AuAl\(_2\) "purple plague", one of the common intermetallic compounds which form whenever gold and aluminum thermocompression bonds are stored at elevated temperatures, is a very good conductor having a specific conductivity of \(8 \times 10^{-6} \ \mu \text{-cm}^{(6)}\), less than three times that of pure aluminum. Such a low resistivity would not lead to a 20 \( \mu \) increase in the measured resistance.

2.2.8 Method 6074A - Quality of Ohmic Contact

This Test Standard, as it stands, is unnecessarily complicated. Much simpler, and more quantitative, methods have been published. We propose to delete Methods 6074A and 6074B and replace them by a single new test method which will provide both qualitative and quantitative data on the quality of ohmic contact.

2.2.9 Method 6074B - Quality of Ohmic Contact

The use of a transistor curve tracer, as specified in this test method, permits the rapid evaluation of ohmic contact quality. However, there is an important deficiency in the method as it now reads. In order to insure that the contact is truly ohmic the test should first be made using the most sensitive current and voltage settings of the curve tracer. Then, if a straight line trace is obtained, the current should
be increased until a current comparable to that flowing during operation of the device is obtained. Only if a straight line trace is obtained during all of these conditions should the contact be considered as ohmic.

As mentioned in the discussion of Method 6074A we propose to delete this method.

2.2.10 Method 6075A - Cross-Section Uniformity of Metallization

This is a routine examination which, as specified in Sec. 7 of Method 6075B, p. 312, should be applied to every wafer. In the absence of some mechanized method of inspection, perhaps based on holography, this Test Method might as well stand as written.

2.2.11 Method 6075B - Cross-Section Uniformity of Metallization.

The comments under Method 6075A, paragraphs 2.2.10, apply to this as well.

3. PROPOSED TEST METHODS

We shall first list the titles of each of the Proposed Test Methods and give a very brief description of each. Then each Proposed Test Method will be discussed and the Test Method itself will be presented.

3.1 Brief Description

<table>
<thead>
<tr>
<th>Title</th>
<th>Brief Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resistivity of Aluminum Metallization</td>
<td>Four point probe measurement of sheet resistance used with thickness to calculate resistivity</td>
</tr>
<tr>
<td>Thickness of Metallization</td>
<td>Multiple beam interferometry</td>
</tr>
<tr>
<td>Metallization Adherence</td>
<td>Film moved against fixed stylus having variable load</td>
</tr>
<tr>
<td>Quality of Ohmic Contact</td>
<td>Qualitative examination and quantitative determination of contact resistance</td>
</tr>
</tbody>
</table>

3.2 Proposed Methods

3.2.1 Resistivity of Aluminum Metallization

3.2.1.1 Discussion

3.2.1.1.1 Resistance measurements using a four point probe have been utilized for a large variety of purposes ranging from archeological surveys
to those of greater applicability to the semiconductor industry. For the present case we shall consider only their use in determining sheet resistance of aluminum metallization. Moreover, we shall consider only the linear array of probes with a separation of 0.050" and films whose thickness is less than half this value.

When a current is passed between the two outermost points the ratio of the voltage drop across the two innermost points to the current is proportional to the sheet resistance of the specimen:

\[ R_s = \frac{V}{I} \]

where \( C \) is a constant of proportionality.

If the specimen is of infinite extent the constant has the value of \( \pi \ln 2 \) or

\[ C = 4.5324 \] (for a specimen of infinite extent)

For any real specimen the value of \( C \) just given will have to be corrected; the amount of the correction depending upon the shape of the specimen and where on the specimen the measurement is to be made. In the succeeding Test Method measurements are to be made at the center of square specimens and at various points on circular specimens.

3.2.1.1.2 Strictly speaking the resistivity as calculated in the Test Method applies only to an infinitely thin film. However, F. M. Smits has shown \( ^7 \) that when the ratio of the film thickness to the probe spacing is less than 0.4, the error is less than 0.05%. For the 0.050 in. probe spacing the film thickness must be less than 0.020 in., which is a very thick film indeed.

3.2.1.1.3 Corrections for use on square specimens have been examined in detail by Smith \( ^7 \). For a one inch square substrate, such as specified in the Test Method, he shows that the constant has the value

\[ C = 4.4516 \] (Probes in center of a 1" sq. substrate, probe spacing - 0.050 in.)

Although they are not required in the Test Methods, measurements on square substrates can be made off center and corrected by an approximate method. \( ^8 \)

In the Proposed Test Method measurements on the square substrates
are only made at the center because a better picture of deposit uniformity can be had using silicon slices which, in general, are larger than the square glass substrates.

3.2.1.1.4 The vast majority of silicon slices used in the industry are prepared by slicing an ingot at right angles to its major axis. As a result, the slices are more or less circular in shape and the value to be used for the constant will depend upon the diameter of the slice.

M. A. Logan\(^{(9)}\) has tabulated values for the constant as has Smits (loc. cit.). From their tabulations we have selected a few values of particular interest. There are displayed in Table 2.

<table>
<thead>
<tr>
<th>Specimen Diameter</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>3/4 inch</td>
<td>4.3646</td>
</tr>
<tr>
<td>1</td>
<td>4.4364</td>
</tr>
<tr>
<td>2</td>
<td>4.5076</td>
</tr>
<tr>
<td>(\infty)</td>
<td>4.5324</td>
</tr>
</tbody>
</table>

3.2.1.1.5 Logan has tabulated\(^{(9)}\) values of the constant \(C\), to be used when the line of the probes is displaced from the center along a radius. However, for surveying a slice in order to assess its uniformity, it is much more desirable to displace the probes perpendicular to the radius. This case has been discussed by Swartzendruber\(^{(10)}\) and the results are displayed in the form of curves for various specimen diameters, the larger two of which are 0.5 in. and 1 in. For larger diameters one must either compute the corrections from his equations or use the extensive numerical tables of these correction factors which have been published \(^{(11)}\). We wish to alert the reader to an unfortunate typographical error in the equations in the paper cited\(^{(10)}\). The correct equation to be used for the case of the four probes arranged at right angles to a radius is:
\[ \eta_c = \frac{1}{2 \ln 2} \ln \frac{\alpha_1 \alpha_2}{4 \alpha_3 \alpha_4}, \]

where,

\[ \alpha_1 = (v_2 - v_1)^2 + (u_1 + u_2)^2 \]
\[ \alpha_2 = (v_2 + v_1)^2 + (u_1 + u_2)^2 \]
\[ \alpha_3 = (v_2 - v_1)^2 + (u_2 - u_1)^2 \]
\[ \alpha_4 = (v_2 + v_1)^2 + (u_2 - u_1)^2 \]

\[ U_1 = \frac{3s/r}{D_1} \]
\[ U_2 = \frac{s/r}{D_2} \]

\[ V_1 = \frac{(1 - (\Delta/r)^2 - (9/r)(s/r)^2)}{D_1} \]
\[ V_2 = \frac{(1 - (\Delta/r)^2 - (1/4)(s/r)^2)}{D_2} \]
\[ D_1 = (1 + \Delta/r)^2 + (9/4)(s/r)^2 \]
\[ D_2 = (1 + \Delta/r)^2 + (1/4)(s/r)^2 \]

In Swartzendruber's notation, \( \eta_c \) is the correction factor in the equation

\[ \rho = \frac{\rho_0}{1 + \eta_c} \]

where \( \rho \) and \( \rho_0 \) are the true and observed values, respectively, of the resistivity. It is related to the correction factor, \( C \), used in this report by the formula

\[ C = \frac{4.5324}{1 + \eta_c} \]

Thus, the equation for calculating the resistivity is

\[ \rho = w \frac{V}{I} \left( \frac{4.5324}{1 + \eta_c} \right) \]

Other symbols used by Swartzendruber in the above equations are:
\( \Delta \), the displacement of the line of probes from the center of the slice; \( r \), the radius of the slice; and \( s \), the probe spacing. We have computed \( \eta_c \) for several slice diameters which were not given in the above paper\(^{(10)}\). These results are plotted in Figure 5.
Figure 5. Correction Factor $\eta_c$ as a Function of Displacement of Probes
We have already referred to the extensive tables of correction factors. We have selected certain of these values and plotted them in Figure 6. Both figures contain similar information. The reason for including them both is that some sheet resistance measuring setups will read directly in ohms/square if the current is set according to the values shown in Figure 6.

3.2.1.1.6 The three most significant instrumental contributions to error in determining the sheet resistance come from the measurement of the voltage and current and from the uncertainty in the probe spacing. For the microvoltmeter suggested the error is ±3% of the range and for the milliammeter, ±1% of the range. This is quite acceptable since the instruments have ranges divided in a 1-3-10 sequence.

The uncertainty in the probe spacing in some commercially available probes is 0.5% of the spacing. In the case at hand this amounts to 0.00025 in. Using the error analysis of Hargreaves and Millard\(^\text{13}\) one can show that in the worst case this will produce an error of 1% in the sheet resistance. Thus, from the equation

\[
R_s = C \frac{V}{I}
\]

it can be seen that the total instrumental error from these three sources will amount to ±5%. Obviously this error can be reduced by using instruments having accuracies better than those assumed.

3.2.1.1.7 No calibration procedure is given since non-adjustable probes are preferred and specified. The probe spacing can be checked by examining it with a traveling microscope.

3.2.1.2 Proposed Test Method

3.2.1.2.1 Purpose - This procedure involves measuring the sheet resistance of the aluminum film and then, from the sheet resistance and the thickness, calculation of the resistivity of the aluminum metallization. Since the resistivity is sensitive to impurities a quantitative estimation of metal quality can be obtained by comparing the experimental and bulk values. In addition, by use of the correction factors the uniformity of metallization may also be determined.
Figure 6. Correction Factor $F_2$ as a Function of Displacement of Probes

-21-
The resistivity is calculated from

\[ \rho = wR_s \]

where

- \( \rho \) = resistivity in \( \Omega \)-cm
- \( w \) = film thickness in cm
- \( R_s \) = sheet resistance in \( \Omega/\square \)

The sheet resistance is determined from the experimentally determined values of the voltage, \( V \), and the current, \( I \), by means of

\[ R_s = \frac{V}{I} C \]

where \( C \) is a constant whose value will depend upon the shape of the slice and the position of the probes with respect to the center of the slice.

If the slice extends infinitely in all directions from the probes \( C \) has the value \( \pi/\ln 2 \) or,

\[ C = 4.5324 \] (for a film of infinite extent)

Otherwise, the value of \( C \) must be determined from the Appendix.

3.2.1.2.2 Apparatus

- a. Four-point probe, 0.050 in spacing (Signatron Corporation, Sunnyvale, California or equivalent)
- b. Power supply, constant current, 0.1, 1.0, 10 mA ranges (any source)
- c. Microvoltmeter (Hewlett-Packard Model 425A or equivalent)
- d. Milliammeter, \( \pm 1\% \) F.S. accuracy, ranges divided in a 1, 3, 10 sequence. (Sensitive Research Instrument Co., Mount Vernon, New York or equivalent)

3.2.1.2.3 Materials

- a. Mechanically polished silicon slices carrying a layer of silica, either grown or deposited, at least 2000\( \AA \) thick
- b. Optically polished Pyrex flats, 1" x 1" x 1/8"

3.2.1.2.4 Procedure

- a. Coat the substrates according to the process under evaluation
- b. Place the coated substrate upon the probe stage and lower the probe until it touches the substrate
- c. Record the current and voltage.
3.2.1.2.5 Calculations

a. Measurements at the center of square substrates

The value of the constant, C, to be used in calculating the resistivity from

\[ \rho = \omega \frac{V}{I} C \]

when the measurement is made at the center of a one-inch square substrate using a probe spacing of 0.050 inch is:

4.4516.

b. Measurement on circular substrates

Determine the diameter of the substrate and find the curve in the figure corresponding to that diameter. Then determine the displacement of the line of probes from the center of the slice. Determine from the curve the appropriate value of \( \eta_c \). If the slice diameter does not correspond to one of the curves, interpolate between two curves.

Calculate the resistivity from

\[ \rho = \omega \frac{V}{I} \left( \frac{4.5324}{1 + 0.0907} \right) = \omega \frac{V}{I} (4.155) \]

c. Sample calculations

Assume a slice of 2" in diameter and that the measurement was made with the probes displaced 7/8" from the center. Therefore:

\[ \rho = \omega \frac{V}{I} \left( \frac{4.5324}{1 + 0.0907} \right) = \omega \frac{V}{I} (4.155) \]

d. Compare the result calculated with the bulk resistivity of aluminum, 2.7 \( \times 10^{-6} \) \( \mu \)cm.

3.2.1.2.6 Accuracy. The accuracy of the sheet resistance measurement is \( \pm 5\% \). Thus, the accuracy of the resistivity measurement is the sum of this value and the corresponding value for the thickness determination.

3.2.1.2.7 Test Frequency. This test should be performed on at least one test substrate included in every evaporation. If the Pyrex substrates are used they can also serve as thickness monitors. The oxidized silicon substrates are of greater use in determining the uniformity of the deposition.
3.2.2 Thickness of Metallization

3.2.2.1 Discussion

3.2.2.1.1 Introduction. As we have indicated previously we feel that the best method of routinely determining the thickness of metallization is one based on the use of a stylus-type instrument. However, the cost of these instruments might preclude their use as production monitoring tools. Therefore, our proposed Test Method uses the Tolansky multiple beam interference method.

3.2.2.1.2 Microscopes with vertical illumination are generally readily available and, with the addition of a monochromatic light source, filar eyepiece, and interference objective, can be used as an interferometer. We have specified an integrated apparatus, such as the Varian "A-Scope" or Sloan "Angstrometer", solely on the basis of convenience. If an interference objective such as the Bausch and Lomb No. 33-16-02-04 is used, the test plate normally supplied should be replaced with a Fizeau plate in order to provide suitably sharp fringes. In addition, it will probably be necessary to modify the illuminator optics in order to provide parallel light.

The substrates called for are the same as specified in our Proposed Test Method: Resistivity of Aluminum Metallization. Mechanically polished, unoxidized silicon slices could be used but the object in using the ones specified is to enable the same samples to be used for a number of test methods. We have spent a considerable amount of time searching for a mask which would be undercut sufficiently by an aluminum etch to give a smooth non-abrupt step. We had only a limited amount of success and the procedure specified in the Proposed Test Method gives far better results than did any method using a mask.

There are probably other etches which will serve as well as the one specified which is our standard reagent for etching aluminum. It happens to be rather viscous and consequently gives an ideally non-abrupt step when used as specified.

3.2.2.1.3 The option is given of forming the step during the evaporation and three ways of obtaining a suitable step are given. However, forming the step in this manner means that the area of metal available for a sheet resistance measurement is reduced, and thus a larger correction factor must be used in that measurement. Therefore, it is better to
cover the entire substrate with metal, make the sheet resistance measurement, and then form the step by the etching procedure.

The etching procedure given will provide a step which is shallow enough to be used for aluminum of about 1μ thick and yet not so broad that the lines corresponding to the lower and upper levels cannot both be seen in the interferometer at the same time. A typical step produced in this way is shown in Photograph 1. The method owes its success to the fact that the contact angle between the etch and the aluminum is very shallow. Because of this, etch products are only slowly removed from, and fresh reagent is supplied only slowly to, that portion of the aluminum next to the meniscus of the air-etch interface. The result is that the etch rate gradually decreases as one goes from the etch up the meniscus. Therefore, after any length of time, the quantity of metal remaining at any point of the substrate along the meniscus will differ from that on the part of the substrate completely immersed in the etch.

The type of metal to be used as the reflective layer is not specified because it is not critical; nor is the method of application. We frequently use copper deposited by sputtering when we are using sodium light in the interferometer. An inexpensive sputtering apparatus for the specific purpose of applying reflective layers can easily be devised.

In spite of the fact that it has been shown that the reflective layer can be as thick as 2μ without introducing error into the determination, we have specified a thickness of around 1500Å. This is because under certain conditions of deposition the fringe pattern can be difficult to interpret if the reflective layer is very thick.

We have examined the possibility of using substrates which are coated with a reflective layer prior to the metal deposition. For example, a group of Pyrex flats could be coated with, say, a reflective layer of chromium and then used one at a time in the same way as the uncoated flats. After a deposition the thickness could be determined at once on the interferometer if a mask was used during the deposition to form a step, or immediately after etching a step if a mask was not used. (The chromium layer has been found not to be attacked by the aluminum etch.) The disadvantage of this technique is that, if the same flat is to be used for both the thickness determination and the sheet resistance measurement, an error is introduced due to the conductivity of the underlying
Photograph 1. Step Produced in Aluminum Film by Etching at the Meniscus of the Air-Etch Interface
reflective layer. We have found that the presence of 1000Å of chromium underneath a micron of aluminum decreases the sheet resistance by about 12% - an unacceptably high error. This error will be still greater if the aluminum is thinner than one micron.

The method specified for measuring the fringe displacement has been chosen to average any non-uniformity in the thickness of the film. This is necessary because usually the fringe space is less than the four point probe spacing.

3.2.2.1.4 If the fringe pattern looked like that shown in Figure 7 of the Proposed Test Method, and the optics used gave no distortion, the only source of error would be in reading the filar eyepiece. With the spacing specified this would probably be less than 2%. But, the fringe pattern seldom looks like that in the figure and the optics do give distortion.

Distortion is frequently evident when examining photographs of interference fringe patterns; generally it introduces an error of less than 5%. Much of this distortion is introduced by the extra optics associated with the camera. Optical distortion is less at the center of the field; thus, in general, it is better if the measurements of the fringes are restricted to this area. Therefore, for films thicker than 1μ, the fringe spacing should be less than that specified.

The method described is generally considered the most accurate method for determining the thickness of thin films. Since it is an absolute method the chief source of error is that introduced by the operator. For this reason an accurate estimate of the probable error is difficult. It is felt that, with care, the error should be less than ±10% of the measured value.

3.2.2.2 Proposed Test Method
3.2.2.2.1 Purpose. The thickness of metallization as determined by this method is used for two purposes. First the determination is needed in order to ascertain that the design requirements of the device are being met. Secondly, an accurate value of the thickness is needed in order to calculate the metallization resistivity from the sheet resistance.
Figure 7. Interference Pattern Showing a Displacement of 3.0 Fringes
3.2.2.2 Principle. A step is formed whose height is equal to the thickness of the metal film. Monochromatic light is reflected from the top and bottom of the step onto a reflecting, slightly transparent plate whose plane can be tilted with respect to that of the sample. The two are illuminated and viewed from a direction normal to the sample. When the distance between a point on the sample and the corresponding point on the reflecting plate is equal to an odd number of half wave lengths of the light used, destructive interference will take place and that point will appear dark. With the sample tilted properly, the locus of these points will form a system of parallel lines or fringes, which will show a displacement corresponding to the step in the sample. The height of the step is equal to the product of the displacement, as measured in units of fringes, and one half the wavelength of the light used. For example, Figure 7 shows the appearance of an interference pattern when the step height is such as to cause a displacement of 3.0 fringes. Thus, the step height must have been

\[ t = \frac{\lambda}{2} = 3 \times \frac{5890}{2} \text{ } \text{Å} \text{ (Na light)} \]

The displacement, \( n \), will not, in general, be a whole number. Its value can be determined to a sufficiently high precision using a filar eyepiece.

3.2.2.3 Apparatus. Interferometer - An integrated apparatus such as the Varian A-Scope or the Sloan Angstrometer.

3.2.2.4 Materials.

a. Substrates: optically polished Pyrex flats 1" x 1" x 1/8"

b. Aluminum Etch - A mixture of the following composition (parts by volume): 320 H₃PO₄, 16 HNO₃, 72 H₂O.

c. Masking materials as specified in paragraphs (1), (2) and (3) of 3.2.2.2.5 if these options are used.

3.2.2.5 Procedure

a. Form a step by one of the following methods

Before the evaporation arrange a mask in front of the substrate so that the substrate is partially covered. The mask can be one of the following.

(1) A wire 0.025-0.030 in. diam in contact with the substrate

(2) A piece of aluminum foil arranged so that its edge is about 1 mm above the surface of the substrate
(3) A razor blade arranged so that the sharp edge of the blade is the edge of the mask.

or

Form a step by etching after the deposition

(1) Pour a quantity of etch into a Petri dish so that the layer of etch is about 1/4" deep

(2) Place the substrate, on end, in the etch. The substrate will easily stand on its 1/8" wide edge.

(3) Remove the substrate when all of the metal has been removed from that part of the substrate which was completely immersed in the etch.

(4) Rinse in D.I. water and blow dry.

b. Apply a reflective layer of metal to the substrate. The thickness should be around 1500Å.

c. Measurement

(1) Place the substrate on the interferometer stage and adjust controls to give a pattern of fringes which run at right angles to the step and have a spacing about the same as a major filar unit.

(2) Using the filar eyepiece determine the spacing between two adjacent fringes, \( w \), by measuring between two non-adjacent fringes and dividing this distance by one more than the number of fringes between them. That is,

\[
w = \frac{1}{n+1}
\]

(3) In the same way determine the displacement, \( D \), of a particular fringe by following it down the step.

(4) Calculate the thickness from the formula.

\[
t = \frac{D \lambda}{2w}
\]

For sodium light \( \frac{\lambda}{2} = 2946 \AA \)

3.2.2.2.6 **Error.** The error should be less than \( \pm 10\% \) provided proper care is taken with the measurement.

3.2.2.2.7 **Test Frequency.** This test is to be performed for every evaporation.

3.2.3 **Metallization Adherence**

3.2.3.1 **Discussion**

3.2.3.1.1 In discussing the existing Test Methods we suggested the
possibility of developing a quantitative test for metallization adherence by means of a regularly applied crosshatch pattern of scribed lines. The idea was that the pattern would render the metal film more susceptible to peeling by the pressure sensitive tape. We have examined this possibility and have been unable to obtain any useful results, mainly because of the great tenacity with which our films adhere to the substrates. We have, however, been able to find a test which can give a semi-quantitative estimate of the adhesion strength of our films. This test has been used as the basis of a Proposed Test Method.

Twenty years ago O. S. Heavens (17) published a method of testing the adhesion of thin films which involved moving the sample under a fixed stylus whose load could be changed. Ten years later Benjamin and Weaver (18) gave a physical analysis of the method and recently it has been used in a study of the effect of ion bombardment on the adhesion of aluminum films to glass. (19) Apparently the method has not been used more frequently because, at the maximum load which can be used on a hardened steel point without causing deformation, a good aluminum film will not peel. We have found that the range of shearing forces (adhesive strengths) can be extended to encompass good aluminum films by using, instead of a steel stylus, one made of diamond. This increases the shearing force in two ways: (1) A greater load can be used without deforming the stylus and, more importantly, (2) a smaller stylus tip radius can be used. A similar approach has very recently been reported by workers at the National Bureau of Standards. (20) This paper should be consulted for a discussion of the limitations of the method.

3.2.3.1.2 Our apparatus, which is sketched in Figure 8, differs from that of Heavens mainly in the means by which the weights are attached. Heaven's design had the weights suspended from the beam at a known distance from the fulcrum. Our design uses cylindrical weights, each of which has a center hole enabling it to be installed concentric to the stylus. The stylus is a 0.7 mil radius diamond phonograph needle. The mechanical stage is used to select the portion of the metal film to be tested and to cause the needle to traverse the metal film.

The limitation on the maximum load comes from the glass substrate. With a load of 300g visible fracturing of the glass underneath the aluminum films can be seen with the unaided eye. Under the microscope
fracturing appears to commence with a load of 220g. After removing the aluminum with dilute HCl scratches were visible only where a load of 200g or greater had been used. However, with careful illumination faint scratches could be detected as the result of a 150g load but not of a 100g load.

The same polished Pyrex 1" x 1" x 1/8" substrates previously specified are used. In an actual production run a single substrate could first be used to measure the sheet resistance, then the adherence, and finally the thickness.

3.2.3.1.3 Benjamin and Weaver showed that the results are independent of the film thickness as long as it is greater than 1000Å.

3.2.3.1.4 This is the weakest part of the whole procedure. An elegant way of determining the end point, which we have not examined, might involve measuring, with a microdensitometer, the transmission of a series of tracks made with various loads and plotting the transmissions as a function of load. One might hope to find a discontinuity in such a plot which could be used as the end point. The procedure as specified in the Proposed Test Method can resolve the end point to a degree somewhat better than the reproducibility which, in our experience, is about ±10%.

3.2.3.1.5 According to Benjamin and Weaver the shearing force, F, produced by a load, w, applied by a stylus of radius, r, to a metal film deposited on a substrate of indentation hardness, P, is given by

\[ F = \left( \frac{a}{\sqrt{r^2 - a^2}} \right) P \]  

(Not \( F = \left( \frac{a}{\sqrt{r^2 - a^2}} \right) -P \) as given in their paper)

where \( a = \sqrt{\frac{w}{\pi P}} \)

As long as the same stylus radius and substrate material are used, the load is a satisfactory measure of the adherence of the metal. The calculation of the shearing force is only of value when comparisons are to be made between the results obtained with different stylus radii or different substrate materials. For example, if it were desired to compare the adherence of a metal film to an oxide surface with that to a silicon surface.
3.2.3.2 Proposed Test Method

3.2.3.2.1 Purpose. This method determines the force necessary to remove a metal film from the substrate. The force required is a measure of the adhesion of the film to the substrate. It thus serves as a monitor of the substrate surface condition just prior to the arrival of the stream of evaporated metal.

3.2.3.2.2 Apparatus.

a. Adhesion Tester - One particular model is shown in Figure 8. Another style can be seen in O. S. Heavens' paper.\(^{(17)}\) In any event, the apparatus is equipped with a diamond stylus of 0.7 mil radius and a set of weights capable of giving a total of 300g.

b. Microscope - Any microscope of about 80X having transillumination can be used.

3.2.3.2.3 Materials.

a. Double-sided masking tape

b. Microscope slide

c. Pyrex substrates with polished surfaces, 1" x 1" x 1/8"

3.2.3.2.4 Procedure.

a. Fasten the coated substrate to be tested to a microscope slide by use of the double sided masking tape.

b. Check that the balance arm is properly counterbalanced with no weights on the vertical rod and install the slide with the substrate in the mechanical stage.

c. Using a load of 100g on the balance arm make a traverse of the substrate, raise the arm and move the substrate 0.5 mm in the "Y" direction. Change the load to 200g and repeat the above procedure, similarly for 300g.

d. Remove the substrate and inspect under the microscope using transillumination. See below for the criterion to be used.

e. From the results of (d) repeat the procedure of (c) changing the loads appropriately.

f. Example - A sample was tested and found to resist a load of 100g but not 200g or more. It was then examined after using loads of 150, 180, and 190g. Only in the last case was the removal criterion met.

3.2.3.2.5 Criterion for Failure. A sample is considered to have failed when examination under the microscope indicates that at a line of removed metal at least 50% of the length of the traverse is visible. Do not confuse the faintly bluish track left by a thin residual layer of aluminum with the much yellower, dashed track due to incipient failure.
The end point is easier to see if a binocular microscope is used in a room with subdued illumination.

3.2.3.2.6 Calculations. The force required to shear the metal film from the substrate can be calculated from the formula where

\[ a = \sqrt{\frac{w}{r}} \]

\[ F = \text{shearing force} \]

\[ w = \text{the critical load needed to just meet the criterion for failure} \]

\[ P = \text{indentation hardness of the substrate (for Pyrex glass, 595 kg/mm}^2) \]

\[ r = \text{the radius of the stylus tip} \]

By means of the previous formula one can compare the adhesion of a metal on one substrate with that of the same or different metal on a different substrate.

3.2.3.2.7 Precision. It is seldom possible to resolve the end point to a precision of better than 10%. The reproducibility of the end point is about ±10%.

3.2.3.2.8 Test Frequency. This test should be performed for every evaporation run. The load necessary to cause failure should be recorded and compared with the values found previously.

3.2.4 Quality of Ohmic Contact

3.2.4.1 Discussion

3.2.4.1.1 As mentioned in Section 2.2.8 we are replacing Test Methods 6074A and 6074B with a single Test Method. Strictly speaking, the two test methods just mentioned do not duplicate one another and could stand as they now exist. However, the former method requires that certain assumptions be made about the various contributions made to the measured resistance; the Proposed Test Method does not. In addition, the Proposed Test Method is, we feel, easier to build into an integrated circuit chip. The procedure is based on recently published work. (22) The only essential change in Test Method 6074B is the emphasis on examination of the behavior of the contact using the most sensitive ranges of the transistor curve tracer and under reversed polarity.
3.2.4.1.2 The apparatus list is divided into two parts. The first gives the equipment needed to make the qualitative test and the second that for the quantitative test. Specific models are not mentioned because this information is not considered important. The power supply and voltmeter could be the same one used for the four point probe measurement of resistivity.

3.2.4.1.3 The nature of the test pattern is not critical as long as it consists of four equally spaced pads, all of the same, known, area, all of which are in contact with the same silicon region. Ideally, each integrated circuit die would have this pattern printed somewhere on it. The processing sequence would be such that the four test contacts would be made in the same way and at the same point in the process as the contacts of the integrated circuit. As an alternative a monitor slice could be processed with the integrated circuit slices in such a way as to provide four contacts which are accurate facsimiles of the contacts in the integrated circuit.

3.2.4.1.4 Whether a particular contact system gives a linear plot of current vs voltage (that is, is ohmic) will depend upon the test conditions. Contacts which appear ohmic at some moderate voltage often exhibit non-linear behavior at very low voltages. For this reason the Proposed Test Procedure calls for testing first at the most sensitive settings of the horizontal and vertical amplifiers. For most transistor curve tracers this will correspond to $10^{-5}$ amps/cm or better and to $10^{-2}$ volts/cm or better. As an additional check that the contacts are not rectifiers the Proposed Test Procedure requires reversing the polarity of the applied voltage. This can be very simply done with most transistor curve tracers. Finally, it is required that the load resistance be changed in order that the contact pass a current similar to the maximum current listed in the device specifications. Again there should be no visible deviation from linearity nor dependence upon the polarity of the applied voltage.

3.2.4.1.5 The quantitative test has been written so that the contact resistance, $R_c$, is determined at two current levels and in two directions of current flow. An ideal contact system would give identical (within experimental error) values under all conditions. For certain devices it may be desirable to change the current from the 10 and 100 mA values.
suggested. Figure 9 shows a schematic diagram of the test setup. All four values of $R_c$ and all three values of the spreading resistance, $R_s$, are assumed to be equal because of the geometry of the test pattern. When the current goes in through contact 1 the voltage measured, $V$, is given by

$$V = IR_s$$

When the current goes in through contact 2 the voltage measured, $V'$, is given by

$$V' = I(R_c + R_s)$$

Thus,

$$R_c = \frac{V' - V}{I}$$

In order to compare the contact resistance found in one test pattern with that found in a pattern of different geometry the contact resistance should be normalized by dividing by the contact area.

3.2.4.1.6 It is important that all tests be performed as soon as is possible in the processing sequence. This test, unfortunately, cannot be performed except towards the end. During processing it is possible that what should have been one "lot" could have been subdivided several times. For example, suppose one lot of slices is, for some reason or another, broken in two and each part diffused separately. Diagrammatically: $L \rightarrow D_1 + D_2$. Then perhaps one of these is again divided before, say, deposition of metal: $D_1 \rightarrow M_1 + M_2; D_2 \rightarrow M_3$. Now, perhaps $M_2$ and $M_3$ are sintered at the same time whereas $M_1$ is sintered by itself. Thus, what might be considered as one lot actually contains slices that have undergone, in this particular example, three different processing sequences: $L \rightarrow D_1 \rightarrow M_1 \rightarrow S_1, L \rightarrow D_1 \rightarrow M_2 \rightarrow S_2$, and $L \rightarrow D_2 \rightarrow M_3 \rightarrow S_2$. In order for this test to be an effective monitor each of these three sublots must be tested. It would not be sufficient to test one of them and assume that the others were the same.

3.2.4.2 Proposed Test Method

3.2.4.2.1 Purpose.

a. To determine qualitatively the nature of the contacts produced by a certain sequence of operations

b. To determine quantitatively the magnitude of the contact resistance.
Figure 9. Schematic Diagram for Quantitative Determination of Contact Resistance
3.2.4.2.2 **Apparatus**

a. Qualitative test
   (1) Transistor curve tracer
   (2) Two probes

b. Quantitative test
   (1) Constant current power supply
   (2) Voltmeter
   (3) Four probes

3.2.4.2.3 **Materials.** A contact test pattern which is part of the integrated circuit being processed or a special contact test pattern which has been applied to a monitor slice.

3.2.4.2.4 **Procedure**

a. Qualitative test
   (1) Place each probe on a different contact
   (2) Slowly increase the test voltage until full scale deflection of the spot is obtained
   (3) At the most sensitive settings of the vertical and horizontal amplifiers the trace should be a straight line with a finite slope
   (4) Reverse the polarity of the applied voltage and repeat steps (2) and (3).
   (5) Decrease the value of the load resistance in the transistor curve tracer so that the contact passes a current similar to the maximum current given in the device specifications. Check for any nonlinearity in the trace. Reverse polarity and repeat.

b. Quantitative test
   (1) Connect the voltmeter to the two innermost contacts by use of the probes.
   (2) Connect the power supply to the outermost contacts.
   (3) Set the current at 10 mA and measure the voltage (V)
   (4) Move one of the current probes to the contact next to it and again measure the voltage (V')
   (5) Calculate the contact resistance from $R_c = \frac{V'}{I} - \frac{V}{I}$
   (6) Reverse the power supply polarity and repeat steps (4) and (5).
   (7) Set the current to 100 mA and repeat steps (4), (5), and (6). All four values should be the same within experimental error.
3.2.4.2.5 **Error.** The values of the contact resistance are generally reproducible to better than ±25%.

3.2.4.2.6 **Test Frequency.** This test should be performed once on each group of slices which have been processed together.

4. **THE MICROCRACK PROBLEM**

4.1 The term "microcrack" is a name given to a failure mode characterized by an open circuit between the bottom of a contact hole and the conductor on the surface of the dielectric. The cause of the open circuit is a discontinuity located, generally, at the intersection of the wall of the contact hole and the upper surface of the dielectric. This discontinuity is usually not visible in an optical microscope and may not manifest itself electrically until the device has been subjected to cycling between temperature extremes. The movement of aluminum films on substrates of lower thermal expansion coefficient as a result of temperature cycling has been observed\(^{(25)}\) in the scanning electron microscope. Hillocks were seen to grow as the temperature was increased and then to shrink as the temperature fell.

While the publicity given to the microcrack problem has given much happiness to some apparatus manufacturers (through the increased sale of scanning electron microscopes) it has also caused considerable anxiety among those whose responsibility is the long term reliability of integrated circuits. Consequently, it is germane to this report to examine the effectiveness of the Test Methods and our Proposed Test Methods in reducing the probability of microcrack formation.

4.2 Three possible contributors to the formation of microcracks are: the metal deposition process; the profile of the oxide edge; and the sintering process.

4.2.1 **The Metal Deposition Process**

The metal deposition process can effect the probability of microcrack formation if it is a process which tends to deposit less metal on the walls of the contact holes than on the top surface. Thickness measurements such as might be made by employing Test Methods 6070A, B, C or the Proposed Test Method of Section 3.2.2.2 give no indication of the amount of metal deposited on the walls.
The amount of metal deposited on the walls of the contact holes will depend upon the angle between the wall and the original dielectric surface and also upon the method of deposition.

Films deposited by sputtering are far less sensitive to the angle of the wall of the contact holes than are films deposited by evaporation. However, as indicated in Section 1.2 we are restricting ourselves to a consideration only of aluminum films deposited by evaporation. The use of planetary substrate holders has become widespread because of the greater thickness uniformity exhibited by films deposited on plane substrates by this method as compared to those deposited on stationary substrates. It is a widely held belief that planetary substrate holders also lead to wall deposits whose thickness approaches that of the deposit on the plane surfaces. However, it has been very recently shown that this may not be the case. The workers cited used computer simulation of deposition from two sources (which, in turn, simulates a planetary substrate) and found that a discontinuity existed until the deposit thickness was twice the step height. This discontinuity was caused by the shadow cast by the growing deposit at the bottom of the step. The application to the problem under discussion is obvious as is the conclusion that, while planetary substrate holders offer an improvement, they are by no means a guarantee that the deposit on the wall of the contact hole will be the same, or even substantially the same, as the thickness on the top of the dielectric.

Since not much can be done about changing the deposit thickness on the walls we turn now to the profile of the oxide edge.

4.2.2 The Profile of the Oxide Edge

The microcrack problem is associated with the steepness of the wall of a contact hole: under any given set of process parameters microcrack formation seems to be less prevalent if the wall forms an obtuse angle to the surface of the silica. In this section we will discuss the measurement of this angle (or its supplement) and the factors which influence this angle.

4.2.2.1 A simple way to measure the angle between the wall and the surface of the silica is by examination under monochromatic light. The diagram in Figure 10 shows how this can be done. The values of $d_2$ and $d_1$ can be determined by use of a filar eyepiece. Unfortunately, for silica
Figure 10. Determination of Wall Angle by Use of an Optical Microscope

\[ \alpha = \tan^{-1} \frac{2t}{d_2 - d_1} \]

Table 3. Preparation of Silica Layers

<table>
<thead>
<tr>
<th>Slice No.</th>
<th>First Layer</th>
<th>Second Layer</th>
<th>Total*</th>
</tr>
</thead>
<tbody>
<tr>
<td>2A</td>
<td>0.55(\mu) thermal oxide</td>
<td>0.52(\mu) SiH(_4)-O(_2):SiO(_2)</td>
<td>1.0 (\mu)</td>
</tr>
<tr>
<td>2B</td>
<td>&quot;</td>
<td>0.65 SiH(_4)-CO(_2):SiO(_2)</td>
<td>1.2</td>
</tr>
<tr>
<td>2C</td>
<td>1.0(\mu) thermal oxide</td>
<td></td>
<td>1.0</td>
</tr>
</tbody>
</table>

*The thickness listed under total may not equal the sum of the thicknesses of the first and second layer because it was determined independently in order to reduce the amount of error.
films of thicknesses of interest in the present work, and etched using photoresists, the results are subject to a very large error. Photographs 2, 3, and 4 show three slices illuminated by sodium light. One millimeter on the photographs equals 2.4μ on the sample. The silica layers were prepared as shown in Table 3. From the photographs and the equation in Figure 10 it is possible to list the three samples in order of increasing steepness: A2 < B2 < C2

But more quantitative data is beyond the resolution of the optical microscope. We thus turned to the scanning electron microscope.

4.2.2.2 In the scanning electron microscope (SEM) the sample is customarily tilted with respect to the electron beam in order to provide an image, when displaying secondary electron emission, that is easily interpretable as surface topography by the eye. This image contains variations in brightness which are the result of the fact that secondary electron emission is roughly proportional to the area of the electron capture volume exposed by the surface. In the case of edges, which would thus be expected to generate large number of electrons, the electrical field of the detector enhances the production of electrons. The result is that edges which appear acute, as seen by the electron beam, will contrast strongly with edges which appear obtuse. In the SEM photos to be discussed, the sample was always oriented so that one edge of the trough cut in the oxide appeared acute. This is illustrated in Figure 11.

Whereas tilting the specimen makes the SEM photograph easier to interpret, it introduces an angular distortion. Thus angles, in general, are not directly measurable. We shall now derive a relationship between the angle between the wall of the trough and the surface of the silicon and the projection of the slant height of the trough and the tilt angle of the specimen.

In Figure 12 AB represents the plane of silicon surface, CD the original surface of the silica, and AC the wall of the trough which makes an angle to the surface of the silicon whose supplement is α = < CAB. The electron beam strikes the surface of the silicon at an angle whose compliment is β. The thickness of the silica layer is t = CB. From the SEM photographs one can measure the projection of the slant height of the trough, w. Thus, the slant height is
Photographs of Samples taken in Sodium Light

Scale: 1 mm - 1.96μ

Photograph 2. A2

Photograph 3. B2

Photograph 4. C2
Figure 11. Reason for Difference in SEM Photos in Relative Brightness of the Two Edges of a Trough in the Silica Layer
Figure 12. Calculation of the Angle Between the Wall of the Trough and the Surface of the Silicon
\[ AC = \frac{w}{\cos (\alpha - \beta)} \]

and also is
\[ AC = \frac{t}{\sin \alpha} \]

thus
\[ \frac{t}{\sin \alpha} = \frac{w}{\cos (\alpha - \beta)} \]

or
\[ \frac{t}{\sin \alpha} = \frac{w}{\cos \alpha \cos \beta + \sin \alpha \sin \beta} \]

thus
\[ \alpha = \cot^{-1} \left[ \frac{1}{\cos \beta} \left( \frac{w}{t} - \sin \beta \right) \right] \]

In the present series of photographs \( \beta \) always has the value of 30°. Therefore
\[ \alpha = \cot^{-1} \left[ \frac{1.15w}{t} - 0.577 \right] \]

A photograph taken with an SEM can best be interpreted considering that the viewer is looking down the electron beam at the sample which is being illuminated by a light located at the detector. As a result, when \( \alpha > \beta \), the wall will lie in a shadow. Thus, many SEM photographs will be easier to measure if the wall opposite to the one illustrated in Fig. 11 is examined. The geometry is analogous and the formula for the angle can similarly be shown to be
\[ \alpha = \cot^{-1} \left[ \frac{1.15w}{t} + 0.577 \right] \]

The value of \( w \) was calculated from the apparent slant height as measured from the SEM photograph and the magnification used in making the photograph. The magnification, in turn, was calculated from the measured width of the bottom of a trough and the actual width as determined using a filar eyepiece and monochromatic light. The trough used for this last measurement was one which ran at right angles to the one used for the estimation of the slant height. That is, the major axis of the trough was tilted 30° with respect to the plane normal to the electron beam.

Photographs 5, 6, and 7 show SEM photographs of the same three slices shown in Table 3. Table 4 gives the results obtained by using the above analysis.
TABLE 4. ANGLE BETWEEN THE WALL AND THE ORIGINAL SILICA SURFACE PRODUCED USING OXIDE ETCH AND KIFR ON VARIOUS COMBINATIONS OF OXIDE

<table>
<thead>
<tr>
<th>Slice No.</th>
<th>Upper Layer Thickness</th>
<th>Type</th>
<th>Lower Layer Thickness</th>
<th>Type</th>
<th>Overall Thickness*</th>
<th>α</th>
</tr>
</thead>
<tbody>
<tr>
<td>2A</td>
<td>0.52 μ</td>
<td>SiH₄-O₂</td>
<td>35°</td>
<td>0.55 μ</td>
<td>Thermal</td>
<td>1.0 μ</td>
</tr>
<tr>
<td>2B</td>
<td>0.65</td>
<td>SiH₄-CO₂</td>
<td>32</td>
<td>0.55 μ</td>
<td>Thermal</td>
<td>1.2</td>
</tr>
<tr>
<td>2C</td>
<td>1.0</td>
<td>Thermal</td>
<td>34</td>
<td></td>
<td></td>
<td>1.0</td>
</tr>
</tbody>
</table>

Average 34°
* See note in Table 2

TABLE 5. ETCH RATES OF VARIOUS OXIDES

<table>
<thead>
<tr>
<th>Silica Type</th>
<th>P-Etch Rate</th>
<th>Oxide Etch Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal</td>
<td>2.0 2/sec</td>
<td>6-17* 2/sec</td>
</tr>
<tr>
<td>(ETO)₄Si densified</td>
<td>2.0</td>
<td>11-12</td>
</tr>
<tr>
<td>RF sputt. 450°C</td>
<td>4.6</td>
<td>-</td>
</tr>
<tr>
<td>React. sputt.</td>
<td>3.8-5.2</td>
<td>8</td>
</tr>
<tr>
<td>(ETO)₄Si 835°C</td>
<td>6.7</td>
<td>20</td>
</tr>
<tr>
<td>P₂O₅-SiO₂ densified</td>
<td>18-35</td>
<td>9-10</td>
</tr>
<tr>
<td>Anodized</td>
<td>18-228</td>
<td>50-75</td>
</tr>
<tr>
<td>SiH₄-O₂ - 5% B₂O₃</td>
<td>-</td>
<td>30</td>
</tr>
<tr>
<td>SiH₄-O₂ - 20% B₂O₃</td>
<td>-</td>
<td>6</td>
</tr>
<tr>
<td>SiH₄-O₂ - 5% B₂O₃ densified</td>
<td>-</td>
<td>9</td>
</tr>
<tr>
<td>SiH₄-O₂ - 20% B₂O₃ densified</td>
<td>-</td>
<td>4</td>
</tr>
<tr>
<td>SiH₄-O₂</td>
<td>-</td>
<td>90-97*</td>
</tr>
<tr>
<td>SiH₄-O₂ densified</td>
<td>-</td>
<td>20</td>
</tr>
<tr>
<td>P₁₅-SiO₂</td>
<td>220-570</td>
<td>9-9.5</td>
</tr>
<tr>
<td>PbO cat. oxide</td>
<td>600</td>
<td>-</td>
</tr>
</tbody>
</table>

* Values determined in our laboratory
The first thing that strikes one on examining Table 4 is that the angle between the wall and the original surface of the silica is not 45°. Since it is different one is justified in postulating an influence of the photoresist layer. This influence might be merely mechanical (microscopic lifting of the photoresist just ahead of the advancing etch front) or it might be the result of acceleration of the etch rate by the resist.

The next noteworthy item in Table 4 is the constancy of the angle regardless of the type of oxide. It has been shown, (24) for a series of waxes used as etch masks on silica, that the angle depends upon the type of wax. The considerations discussed in the previous paragraph probably apply here as well. Explanations of differences in acceleration might involve differences in the polarity of the resists; even surface catalysis might play a part. One wonders if a resist other than KTR would produce a different angle. If so, the first step in avoiding microcrack formation might be the choice of the resist used. In the past resists were chosen to give a minimum of undercutting, that is, a maximum value of $\alpha$.

Table 4 shows also that the presence of a lower layer having an etch rate less than that of the upper layer results in an overall value of $\alpha$ which is less than that of the upper layer. (In the sequel it will be shown that the overall angle can be estimated from the relative etch rates of the two layers and the value of $\alpha$ for the upper layer). One of the ways in which the microcrack problem has been "solved" is by use of "P-etch" instead of oxide etch. These two etches differ in that a greater range of etch rates is produced with P-etch and various oxides than with oxide etch. This can be seen from the data in Table 5 which has been gleaned from various sources. Now, the use of an etch whose rates show a large difference between the upper and lower layers will produce a very shallow overall angle between the wall and the original silica surface. However, if the uppermost layer is too thick (when compared to the thickness of the metallization) this will have no effect whatever on the formation of microcracks because the value of $\alpha$ for the uppermost layer is uninfluenced by the presence of lower layers. Thus, we conclude that the choice of etch cannot alone guarantee the nonformation of microcracks, but that the combination of etch choice and metallization thickness and nature and thicknesses of the various
silica layers have to be considered together. Nevertheless it is obvious that a method of predicting the wall profile would be of great value.

4.2.2.3 It appears reasonable that one should be able to draw the wall profile if the initial value for \( \alpha \) and the relative etch rates are known. This will be done for samples 2A and 2B.

Example: No. 2B

Draw a line representing the interface between the two silica layers and then, 0.65 \( \mu \) above it, one representing the upper surface of the \( \text{SiH}_4 - \text{CO}_2 \).

Draw a third line 0.55 \( \mu \) below it to represent the silica-silicon interface. At some point, 0, on the middle line draw a third line at an angle, \( \alpha = 34^\circ \), which is the average value found. See Table 3. The point on the uppermost line directly above this point represents the boundary of the photoresist (See Figure 13).

Now, in the length of time in which the bottom 0.55 \( \mu \) of thermal oxide is etched, the upper layer will be etched a distance which depends on the relative etch rates of the two oxides. From Table 5 we see that the ratio is 20:17. Thus the upper oxide will be etched \( 0.65 \times \frac{20}{17} = 0.77 \mu \).

Draw an arc of this radius from the uppermost unmasked point of the upper oxide layer (Point A in Figure 13) and then draw a perpendicular to the wall at point A. The intersection of this perpendicular and the arc just drawn (point B) represents the bottom of the upper portion of the wall which can now be drawn so that it makes an angle of 34\(^\circ\) to the original uppermost silica surface. Connecting this point with one on the silicon-silica interface directly below point 0 will give the lower portion of the wall. The wall angles can now be measured.

Example No. 2A

See Table 4 for data. The construction is analogous to that in the previous example except that, since the thickness of the layer is smaller than the thickness of the lower layer multiplied by the ratio of the etch rates, the process is repeated until the sum of the segments parallel to \( AB \) is equal to the amount of upper layer etched. That is, \( \frac{97}{17} \times 0.55 = 3.1 \mu \). See Figure 14.
Figure 13. Graphical Estimation of Wall Profile for Slice 2B

Figure 14. Graphical Estimation of Wall Profile for Slice 2A
The values estimated for slice No. 2B are so close to those measured that the agreement must be fortuitous. In the case of slice 2A it is possible that the presence of a thin layer next to a very much more slowly etched layer results in depletion of the etch and the actual etch rate becomes diffusion controlled. This, of course, is not accounted for in the graphical method. The result obtained assumed an amount of silica removed which is greater than that which was actually removed and thus the estimated values for $\alpha$ are less than those measured. Nevertheless the agreement is close enough to be useful.

Even subject to the limitations just discussed the method can be of value in predicting wall profiles. As an example, we will draw the profiles produced on an integrated circuit slice by two different etches. We shall assume that the value of $\alpha$ for the first layer using P etch is the same as was found for oxide etch. The types of oxide, their thicknesses and etch rates (taken from Table 5) are shown in Table 7. The constructions are shown in Figure 15.

In the case of example 3 it can be seen that a slight amount of undercutting is predicted. That this actually happens is suggested by the scanning electron microscope photograph shown in Photograph 8. The undercutting is just barely visible, but the crack which it produced in the metal is quite easy to see. The graphical method also shows how this problem can be cured. Possible methods are (1) make the first layer of a silica type which etches at a rate of at least $23 \text{ \mu m/min}$. The data of Table 5 suggests the use of unconverted $(\text{ETO})_4\text{Si}$-silica or partially converted $\text{SiH}_4\cdot \text{O}_2\cdot \text{Si}$-silica; (2) make the bottom layer thinner. A reduction
Each div. = 1000 Å

Figure 15. Graphical Estimation of Wall Profiles, Examples 3 and 4
Photograph 8. Undercutting and Crack Formation Produced Thereby
of about 700 Å is estimated by this method.

### TABLE 7. DATA FOR EXAMPLES 3 AND 4

<table>
<thead>
<tr>
<th>Layer</th>
<th>Silica Type</th>
<th>Thickness</th>
<th>Oxide Etch</th>
<th>P Etch</th>
</tr>
</thead>
<tbody>
<tr>
<td>Upper</td>
<td>(ETO)$_4$Si</td>
<td>3000 Å</td>
<td>20 Å/min</td>
<td>7</td>
</tr>
<tr>
<td></td>
<td>conv. @ 900° 30 mm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Middle</td>
<td>$P_2O_5$-$SiO_2$</td>
<td>1000</td>
<td>10</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td>converted</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower</td>
<td>Thermal</td>
<td>3000</td>
<td>17</td>
<td>2</td>
</tr>
</tbody>
</table>

In example 5 we see the result of etching this same combination with P-etch. The differences in etch rates with this reagent are so great that extreme undercutting is predicted to take place. The overall angle cannot be predicted because of the obvious presence of diffusion controlled etching in this case.

#### 4.2.3 The Sintering Process

The only Test Methods which have any bearing on the contribution of the sintering process to the probability of microcrack formation are those which are concerned with the quality of the ohmic contact: 6074A, B and the Proposed Test Method of Section 3.2.4.2. Unfortunately the application of these Test Methods can increase the probability of microcrack formation rather than reduce it. This apparent paradox arises because if the Test Method indicates that the contacts are poor in a batch of devices, the usual "fix" is to increase the severity of the sintering process. As will be shown in the sequel, it is this treatment, more than any other, that increases the likelihood of device failure due to microcrack formation.

**4.2.3.1** The samples discussed in Section 4.2.2 were coated with a layer of aluminum, 1500 Å thick, using a stationary substrate holder. They were then subjected to a series of sintering conditions of increasing severity. The first sintering operation consisted of heating in argon
at $450^\circ C$ for 20 minutes. The second: $500^\circ C$ for 30 minutes. The third: $565^\circ C$ for 60 minutes.

In order to be able to identify more easily various features in the photographs to be discussed, the terms illustrated in Figure 16 will be used.

Slice 2A: The presence of the aluminum layer rendered more distinct the upper shoulder of the right wall (Photograph No. 9). Because of the strong emission of electrons, this feature is not visible on the upper wall. This was not the case before the deposition of the aluminum, as can be seen in Photograph No. 10. Note that the superimposed intensity scan shows the presence of the upper shoulder of the upper wall, even though it is scarcely visible in the photograph. Photograph No. 11, taken after sintering at $450^\circ C$ for 20 minutes, shows essentially no change. These sintering conditions are the ones normally used in the Microelectronics Center and have been found sufficient to give good contacts, provided that the contact holes have been etched properly. An additional heat treatment ($500^\circ C$ for 30 minutes) results in the loss of metal from the upper shoulder of all four walls, as can be seen in Photographs 12 and 13. (Note that Photograph No. 12 was taken at a magnification of 1000X; half of what was used in the other photographs.) This metal which has been lost from the shoulders must have gone somewhere and this somewhere is suggested by comparison of the surface topography as shown in Photographs Nos. 9, 11 and 13. Observe that the aluminum as deposited is smooth and featureless. After the first sintering operation, a definite surface structure is visible, evidently due to grain growth. This consists of two classes of crystallites: small and large. After further heating, it is seen that the large crystallites have grown at the expense of the small. This indicates the presence of surface diffusion and this same phenomenon is probably responsible for the loss of metal at the shoulders. This phenomenon is shown after having been carried to an extreme degree in Photograph No. 14. Note that now, after heating at $565^\circ C$ for one hour, there are large patches
Figure 16. Illustration of Terms Used in Describing Features in the SEM Photographs
Photo. No. 9. 2A After Aluminum Metallization

Photo. No. 10. 2A Before Aluminum Metallization

Photo. No. 11. 2A After First Sintering Operation

Photo. No. 12. 2A After Second Sintering Operation
Photo. No. 13. 2A After Second Sintering Operation

Photo. No. 14. 2A After Third Sintering Operation

Photo. No. 15. 2A After Second Sintering Operation
on the upper surface which are devoid of metal. While a temperature of 565°C may be considered as excessive, it has been used by some manufacturers. Even at the considerably lower temperature of 500°C, drastic metallurgical reactions can take place between aluminum and silicon. This is illustrated in Photograph No. 15 which was taken at 1000X after the second sintering operation. Note the well-defined etch pits at the bottom of two of the troughs. No such pits were observed to form as a result of the first sintering operation used.

Slice 2B: Photographs 16 and 17 were taken after the deposition of the aluminum and after the first sintering operation, respectively. Again, no voids have made their appearance, although surface diffusion has taken place as indicated by grain growth. After the second sintering operation (Photograph 18), little change has taken place. Surprisingly, no voids appear to have formed. After the third sintering operation (Photograph 19), there are obvious patches from which the metal has completely disappeared, as well as pits at the bottom of the trough.

Slice 2C: Photographs 20 and 21 are analogous to and appear similar to Photographs 16 and 17. Photograph 22, taken after the second sintering operation, again shows the growth of the large crystallites at the expense of the smaller with consequent smoothing of the surface. Voids first become visible as a result of the third sintering operation, as shown in Photograph 23.

4.2.3.2 The fact that voids were seen only on one of the three samples, even though all three had about the same value for the angle of the upper shoulder, may reflect the fact that a nonrotating substrate holder was used during the evaporation. Then, when the samples were each mounted in the SEM with the same orientation, it is possible that those edges on which microcrack formation took place because of the relative direction of the aluminum vapor stream might, or might not, be in a position to be observed. This would also account for the observation
Photo. No. 16. 2B After Aluminum Metallization

Photo. No. 17. 2B After First Sintering Operation

Photo. No. 18. 2B After Second Sintering Operation

Photo. No. 19. 2B After Third Sintering Operation
Photo No. 20. 2C After Aluminum Metallization

Photo No. 21. 2C After First Sintering Operation

Photo No. 22. 2C After Second Sintering Operation

Photo No. 23. 2C After Third Sintering Operation
in Sample 2A that microcracks were seen on the upper wall and the right wall very easily, but less easily on the lower wall or on the left wall (See Figure 16).

The experiments just described and those described in Section 4.2.2 strongly suggest that microcrack formation proceeds by surface diffusion driven by crystallite regrowth. Failure occurs where the metallization is thinnest and this in turn is determined by edge profile and evaporation technique. For any combination of edge profile and evaporation technique, failure is more likely to occur if sintering conditions are severe. Edge profile can be controlled by suitable choice of oxide type and thickness, and etch type but the angle of the upper shoulder is determined (probably) by the etch and resist used and is independent of oxide type.

4.2.3.3 Thus, the best way to avoid microcracks is to use moderate sintering conditions. The use of moderate sintering conditions demands, in turn, that the contact holes be completely and cleanly opened. This then requires assurance that all of the photoresist has been removed from the contact holes during the developing step.

There is a great need for a technique to monitor the bottoms of contact holes. The technique must be nondestructive and capable of detecting films thinner than 100 Å. It is possible that a micro-ellipsometer could be devised which would meet these requirements. If one had such an instrument one could check after developing to make certain that no photoresist remained in the contact hole just prior to etching. After etching one could establish that the contact hole was completely open. If they were not, the slice could be re-etched. Another check would be made after the photoresist removal just prior to placing the slice in the evaporator to ensure that all of the photoresist had been removed and that none had redeposited in the contact holes. At the same time the extent of regrowth of "native oxide" could be assessed.

Contacts made using the monitoring scheme described should be of high quality with only very moderate sintering conditions, and the use of moderate sintering conditions would greatly reduce the probability of microcrack formation. These comments apply as well to crossover processes.
The following quotation from a paper by E. E. Maiden and R. D. Engquist (26) will summarize the structurally unsound aspects of silicon semiconductor contacts as used in integrated circuits: "The idea of stretching a material with poor elevated temperature strength, good thermal conductivity, and fairly high specific heat over a ceramic which has good hot strength, very low thermal conductivity, and somewhat lower specific heat and firmly attaching it to a brittle metal with fairly high thermal conductivity and low specific heat is repugnant to most structural engineers. When the problem is further aggravated by a 1:5:10 ratio of coefficients of thermal expansion, inconsistent bonding conditions and etched oxide edges, it seems surprising that metallization cracks are the exception and not the rule."

5. ELECTROMIGRATION

5.1 The phenomenon of the transport of metal atoms by an electric current is called electromigration. It is of importance to the integrated circuit designer because the movement of metal atoms results in the formation of voids (which in turn can migrate). The voids result in a decrease in the effective cross sectional area of the conductor and hence to ever more rapid transport of atoms and creation of voids until finally failure occurs: usually by the formation of an open circuit although the accumulation of transported metal atoms can result in shorting.

The pioneer work on the electromigration of aluminum thin films was done by J. R. Black and a good guide to the literature on his work and that of others is a paper he published in 1969. (27) Penny (28) established that, in the case of bulk aluminum, the atoms are transported in the direction of the electron flow and this was confirmed by Black for the case of thin aluminum films.

5.2 In order to stress the influence of various processing parameters on failure under electromigration conditions, we wish to introduce the concept of "intrinsic failure" and "extrinsic failure". The latter is exhibited by a sample when the failure after passage of current can be ascribed to the presence of a fault imparted by the processing sequence, for example a tweezer scratch or a pinhole. Other examples will be cited later. Intrinsic failure is that which occurs when no such faults are present.
The causes for failure in such a case are called "primary factors".

5.3 Black proposed a model based on the work of Huntington (29):

\[ \frac{1}{M} = AJ^2 \exp \frac{q}{kT} \]

where

- \( M \) = median time to failure (hrs)
- \( A \) = a constant which contains a factor involving the cross sectional area of the film
- \( J \) = current density (A/cm\(^2\))
- \( q \) = an activation energy (eV)
- \( k \) = Boltzman's constant
- \( T \) = film temperature (°K)

Other models have been suggested (30, 31, 36) but Black's model has had by far the largest amount of data fitted to it. The chief point of dispute is the value of the exponent of \( J \), values as low as unity having been suggested (37) for current densities less than 10\(^6\) amp/cm\(^2\).

For dealing with the data Black found a useful parameter which we wish to identify with the letter \( B \) (for Black):

\[ B = \frac{we}{J^2M} \]

where

- \( w \) = width of the conductor
- \( t \) = thickness of the conductor

Since \( J = I/a \)

where

- \( I \) = current
- \( a \) = cross sectional area of the conductor

\[ B = \frac{a^3}{I^2M} \text{ (cm}^6, \text{ amp}^2, \text{ hrs}) \]

Typical values for \( B \) are in the range 10\(^{-23}\) to 10\(^{-20}\); the smaller values being associated with longer lifetimes at a given temperature.

5.4 The primary factors which have so far been identified, by some authors, with intrinsic failure of aluminum films under electromigration conditions are the size of the crystallites and the presence or absence of a dielectric overcoat. (We are not ignoring the effect of temperature gradients, only deferring them until later.)
5.4.1 The influence of grain size is by no means universally accepted. Thus we have Black in his series of papers\(^{27}\) saying that large grains lead to longer life as does Ghate\(^{32}\) and Oliver and Bower\(^{33}\). On the other hand, workers at Fairchild have found no effect of grain size.\(^{31, 34}\) A possible rationalization of this conflicting result might be found in the paper presented by M. J. Attardo\(^{35}\) in which he examined films having a preferred orientation and concluded that grain size divergences result in earlier failures. It must be borne in mind that Black's original work showed that differences in lifetimes became smaller as the ambient temperature increased and vanished at about 275\(^{\circ}\). 

5.4.2 The beneficial effect of a silica overcoat was pointed out by Black\(^{27}\) and received confirmation from Spitzer and Schwartz\(^{38}\), who explained the phenomena as being due to "filling [of] broken electron bonds at the aluminum surface". Later we shall suggest a mechanical mechanism by which a silica overcoat may increase lifetime. It should be noted that Sello\(^{34}\), in reporting a paper presented by Mutter\(^{39}\) wrote: "On the other hand, covering aluminum strips with r.f. sputtered silicon dioxide resulted in no appreciable change in the lifetime of these strips."

5.5 Investigators of electromigration in thin films have been aware of the statistical nature of the failures and generally have carried out their experiments on large numbers of samples. The reason for this statistical nature lies in the secondary factors which cause extrinsic failure. We will now examine the magnitudes of the influences produced by the following secondary factors:

a. Stripe width
b. Stripe cross section
c. Abrupt steps
d. Substrate asperities
e. Lead bonding
f. Adhesion to substrate
g. Frequency of faults

5.5.1 Black's equation, shown at the beginning of Section 5.3 can be expressed as

\[
\frac{wt}{J^2M} = c[f(T)]
\]
where $C$ is some constant

Then

$$M = \frac{w^2t^2}{I^2c[f(T)]}$$

Or, for a definite current, temperature, and thickness

$$M = c'w^2$$

If some fault has been introduced such that the width of the stripe is irregular to an amount $\Delta w$ then the change in the mean time to failure will be

$$\Delta M = \frac{c'(w + \Delta w)^2 - c'w^2}{c'w^2}$$

Typical faults might be caused by dirt on the photomask, on the slice, or in the photoresist, or from improper exposure or development of the photoresist or from over etching. Photograph 24 shows two types of faults. The more obvious type appears as more or less semicircular notches in the edges of the stripe. Less evident is the irregularity in the width of the stripe caused by "necking-down" where the stripe joins the contact pad. This may have been due to improper exposure of the photoresist. Similar faults can even be caused by improper mask making technique. If we assume that these faults are 1µ in size then the equation above becomes

$$\Delta M = \frac{2w + 1}{w^2}$$

This is shown plotted in Figure 17. The graph lucidly demonstrates that the test stripes should be nearly one mil wide if it is desired to reduce to 10% the contribution made by these small faults to error in the median time to failure.

5.5.2 The assumption that the metallization stripes have a rectangular cross section instead of a trapezoidal one is another contributor to error. We have shown in Section 4.2.2 that various types of silica, masked with KTFR and etched with oxide etch exhibit a remarkable constancy in the angle made by the wall of the trough and the original surface of the silica. While we do not know that the angle found, 34°, is applicable to the case of aluminum films it is certain that the angle is not 90°.
Photograph 24. Aluminum Conductor, 0.15 Mil Wide, Showing Two Kinds of Faults Which Can Lead to Premature Electromigration-Induced Failure.
Figure 17. Error Produced in Mean Time to Failure by One Micron Irregularities in Width of Stripe
Figure 18 shows the cross section of a conductor in which the wall angle is $\alpha$. The nominal width of the stripe is shown as $w$, and the thickness as $t$. It can be shown that the area of the figure is

$$a = t(w - t\cos\alpha)$$

If $\alpha$ is $90^\circ$, then $a = tw$, but if $\alpha$ is assumed to be $34^\circ$

$$a = t(w - .829t)$$

Thus, the error introduced into $a$ by the assumption that $\alpha$ is $90^\circ$ instead of $34^\circ$ is

$$\frac{tw - t(w - .829t)}{t(t(w - .829t))} = \frac{.829t}{w - .829t}$$

In the previous Section we showed the relation

$$M = \frac{22}{w^2} \frac{2}{1^2} c'[f(T)]$$

Thus, for a definite current and temperature

$$M = c't^2w^2$$

Therefore, the change in the median time to failure produced by the assumption that $\alpha = 90^\circ$ when it is actually $34^\circ$ would be

$$\Delta M = c'[t(w - .829t)]^2 - c'[tw]^2$$

Or, neglecting the sign, the percentage error is, approximately, $\frac{164t}{w - 1.64t}$

This is shown plotted, for two values of the thickness, in Figure 19. Note the circles which indicate a cross sectional area of $10^{-7}$ cm$^2$, a common value. For a 1$\mu$m thick film the error contribution to the median time to failure made solely by the assumption being discussed is almost 20% and is even greater if the angle made by the wall of the trough and the original surface of the silica is less than the $34^\circ$ being assumed. That this angle may intentionally be made less than this is shown by a recent paper by Devaney, et al. \cite{40} The purpose in producing metal stripes with a nonrectangular cross section is that such stripes, after coating with dielectric, make less abrupt steps in a multi-level crossover process. 5.5.3 Abrupt steps, as just mentioned are the result of having a lower level metallization stripe of ideal, or nearly ideal, cross section from
Figure 18. Cross Section of a Metallization Stripe
If There Had Been No Undercutting, the Wall Angle, $\alpha$, Would Have Been 90°.
Figure 19. Error Produced in Mean Time to Failure by Assuming That the Stripe Has a Rectangular Cross Section Instead of a Trapezoidal One ($\alpha$ assumed to be 34°). Circles indicate cross section area of $10^{-7}$ cm$^2$. 
the standpoint of the previous Section. And abrupt steps, the advertise-
ments of manufacturers of planetary substrate holders not withstanding,
are possible sites for reduced metal thickness. With a nonrotating sub-
strate and a point source at infinite distance and assuming the dielec-
tric contour to be a faithful replica of that of the metal, the thickness
on the step would be
\[ t' = t \cos \alpha. \]
Thus the requirements of this Section and the preceding one are incompatible.
A similar situation arises in the case of feed-through holes in the dielec-
tric and in the case of contact holes. However, in these cases the usual
designs provide conducting films on all four sides of the holes. Thus
the current density is not increased at these places and they are generally
not frequent sites for electromigration failures.

5.5.4 Closely related to the preceding is the influence of the substrate
smoothness. It has been long recognized that continuous films of a given
thickness cannot be deposited on surfaces having greater than a certain
amount of roughness. With some combination of film thickness and sub-
strate roughness the deposited films, while continuous, will have areas
of reduced thickness. These areas are potential failure sites when the
film is stressed under electromigration conditions for the reason given
in Section 5.5.2.

5.5.5 Pads for lead bonding are generally much wider than the metalliza-
tion stripes for reasons of convenience in applying the lead bonds. This,
of course, reduces the current density to very low levels yet electro-
migration failures frequently occur in these pads. We shall ignore the
trivial case of the lead being insufficiently bonded to the pad so that a
local area of high current density exists and shall deal separately with
ultrasonic and thermocompression bonds.

5.5.5.1 The principle in ultrasonic bonding is that welding of aluminum
usually takes place quite easily once the oxide has been scrubbed off
the surface. Unfortunately, if care is not exercised, there can be a
significant reduction in the metallization thickness just around the bond.
Photograph 25 shows a failure caused in such a way. The lead is still
attached but observe that the metal is gone from almost the entire
periphery of the lead.
Photograph 25. Electromigration Failure of an Aluminum Pad Caused by Improper Ultrasonic Bonding Conditions.
5.5.5.2 Thermocompression bonds with gold balls do not consume aluminum during the bonding operation because there is no scrubbing action and the pressure is applied through the ball. After the bond is made, diffusion between the couple takes place and a series of intermetallic compounds begins to form. Some of these have colorful names such as "purple plague" or "black death" and were, at one time, thought to be exceedingly undesirable. It is now known \(^5\) that failures after long term storage at elevated temperatures are due to voids formed by the Kirkendall effect. Condensation of these voids eventually results in the bond having zero strength. In the case of bonds which are carrying current, failure can occur much more rapidly due to electromigration. Once again, this is because of the reduced cross sectional area and consequent higher current density. One might assert that these considerations were implicit in Bleck and Sello's statement \(^{44}\) that: "...the gold ball bonds can act as sinks for the thin aluminum films surrounding them, and, hence, cause a certain enhancement of failures."

5.5.6 In the discussion so far we have assumed the absence of thermal gradients because the metal stripe was assumed to be well heat sunk to the substrate. This is a fairly reasonable assumption as long as there is good adhesion of the metal to the substrate. If there is not, temperature gradients will be formed and failure will occur. \(^{41}\) If there are places where the adhesion is marginal, possibly because of nonuniformly distributed dirt on the substrate prior to metal deposition, they may become sites of nonadhesion under the influence of ohmic heating. Once this happens, they become places where heat sinking is minimal and then they become hotter and sites of temperature gradients and ultimately failure sites. Howard and Ross \(^{45}\) have reported on techniques to detect the positions of thermal maxima in samples which have been subjected to electromigration conditions.

Electromigration in gold film conductors has been found by Hartman and Blair \(^{44}\) to be "correlated with thermal gradients along the conductor in contrast with observations on aluminum where the effect is dominated by structure or metallurgical properties of the film." \(^{76}\) (Emphasis added) It should be noted that it has been found \(^{47}\) that gold films are far more resistant to electromigration failure than aluminum films.
5.5.7 Some of the faults discussed in the previous sections are randomly distributed across the slice and hence along a metallization stripe. Examples of this type of fault are places of poor adhesion, substrate asperities and the semicircular irregularities distributed along the edges of the stripe such as are shown in Photograph 24.

The irregularities appear to be about 1μ in diameter and to occur with an incidence of about one per 5μ of edge. Since, according to Black, M depends on the cross sectional area of the stripe and not on the volume, the existence of many of these faults does no more harm than a single one which, as has been shown, is considerable. With a fixed incidence of faults the longer the stripe the greater the chance that the stripe will contain at least one fault. Thus, in order to insure that M is independent of stripe length, the stripe length should be minimized. Since we are dealing with the probability of a single fault occurring in any particular stripe, the influence will be statistical. That is, with a fixed incidence of faults, short stripes should give a tighter distribution of the experimentally determined values of M than should long stripes. In effect this means that it will be easier to draw conclusions from a small number of experiments if the stripes are short rather than if they were long. In this respect the test vehicle which we are using is very poor. (q.v.)

5.6 Experimental Results

5.6.1 We had three purposes in doing electromigration experiments. The first was to learn about the various factors which cause the ultimate failure when the stripes carry a high current density. Second, we wanted to know if we could confirm some of Black's results. Finally, we wanted to know if the lifetime of a conductor could be influenced by post-etching heat treatment.

In order to obtain as high a resolution in the etching step as is permitted by the other process parameters, it has long been known that it is necessary to have the aluminum, prior to etching, in a fine grained condition. This is generally done by proper choice of the deposition parameters. However, there is considerable evidence, which has been cited in Section 5.4.1 that indicates that fine-grained aluminum films have a shorter mean time to failure than do large grained films. (We also cited some work which did not support this.) Thus, it was our
intention to find a way in which aluminum crystallites can be regrown after etching so as to provide large grains. In this way the aluminum which will be etched will be fine grained and hence capable of yielding the ultimate results with respect to line definition.

5.6.2 We are aware of the fact that failure of a conductor due to electromigration is a function of gradients, most noticeably in temperature and current density. Our test vehicle, which will be described below, has, like most which have been reported in the literature, a built-in current density gradient at each end. In addition, this commonly used configuration also provides for a thermal gradient which is, more or less, congruent with the gradient in current density. Blech and Meieran \((31)\) used a test vehicle in which the effect of a temperature gradient in electromigration is nicely separated from the effect produced by a current density gradient. Their test specimen, which is illustrated in Figure 20 provides for the presence of a thermal gradient in the absence of a current gradient. In their words: "When electrical current is passed through the strip, the heat generated by the current can be removed only by the conduction along the metal strip and then through the oxide into the Si and sample holder. Hence, the major portion of the sample remains as ambient temperature \(T_a^\circ\text{C}\), while the test portion of the strip can be considered as a current carrying wire, heat sunk at both ends."

5.6.2.1 We have devised, but not built, a test vehicle which provides for the presence of a current density gradient while minimizing the thermal gradient. Data obtained from such a test vehicle would be expected, because of the influence of heat sinking in actual circuits, to yield data capable of extrapolations over a wider range of film thicknesses than data obtained otherwise.

Consider a conductor of variable thickness such as is shown in Figure 21. Although the thickness is variable note that the cross sectional area is constant. The figure has been drawn so that the contact zones at the ends are five units thick and two units wide, whereas the central zone is one unit thick and ten units wide, giving a cross sectional area of ten units squared. Similarly, each of the transition zones at each end of the central zone also has a cross sectional area of ten units squared. Now, a conductor such as this would have a lower temperature at the central zone than at the contact zones because of the
Figure 20. Test Vehicle with Temperature Gradient and No Current Density Gradient (at Center of Specimen) after Blech and Meieran
Figure 21. Test Vehicle with Current Density Gradient and no Temperature Gradient
greater heat sink area in the former zone. (Remember that the $I^2R$

dissipation per unit of length is constant). A current gradient can be

supplied by a slight change to the conductor. Imagine that the central

zone is somewhat thinner than shown in Figure 21. Now, the $I^2R$
dissipation per unit length is no longer constant. At some particular thickness the

non-constant $I^2R$ dissipation will just compensate for the increased heat

sink area of the central zone. To a first order of approximation this

will happen when the thickness is two tenths of a unit.

Typical dimensions might be as follows. In order to ensure ease

of lead bonding $w_1$ needs to be at least 100$\mu$, and in order to ensure
ease of etching $t_1$ needs to be less than 2$\mu$. Taking these two values

($w_1 = 100\mu$, $t_1 = 2\mu$) gives a cross sectional area, $a$, of $200\mu^2$. If

$w_2 = 200\mu$, then making $t_2 = 1\mu$ would give a test vehicle in which there

was no current density gradient. If $t_2$ is made only 5000$\AA$, then we will

have a test vehicle with a gradient in the current density but not in
temperature. It could be fabricated by first evaporating aluminum to
the thickness $t_2$ and then a noncontacting mask would be put in front of
the vapor stream in order to shadow the central zone. From the profile
of the penumbra, either experimentally or theoretically determined, one

could calculate the shape of the photo etching mask needed to provide a
constant current gradient in the end and transition zones.

5.6.2.2 Our test vehicle is shown in Figure 22. The test vehicle pro-
vides, on each chip, five 1 mil wide lines, four 0.5 mil lines, five 0.3
mil lines, and four 0.15 mil lines. The lines are of two lengths:

0.09 in and 0.12 in. Some of the lines are in the form of pairs separated
by 0.1 mil, 0.3 mil, or 0.5 mil. There was no particular reason for having
the lines in pairs for electromigration experiments: they resulted from
the fact that the test vehicle was prepared by modifying the artwork of a
test pattern made for another purpose. However, it turned out that having
the lines associated in pairs revealed a new potential failure mechanism
which will be discussed later.

It should be clear from the discussion in Sections 5.5.1, 5.5.2, and
5.5.7 that our test vehicle is very poor from the standpoint of studying the
primary factors which determine the intrinsic failure of conductors due
to electromigration. But being forced to use such a poor test vehicle
Figure 22. Test Vehicle for Electromigration Experiments
has alerted us to the real value of electromigration tests. This will be discussed later.

5.6.3 The duration of an experiment depends on the mean time to failure. Some investigators\(^{(34)}\), in order to save time, terminate their experiment when 50% of the samples have failed and use this period as the life time. In any event, we can expect the duration of an experiment to be longer when employing lower current densities and lower temperatures. It has been shown that samples run at lower temperatures are more sensitive to differences in grain size\(^{(27)}\) and the presence or absence of an overcoat\(^{(38)}\). Thus, if we are interested in assessing these influences we must use lower temperatures. Judging from the published work, the change produced in the mean time to failure by differences in grain size or overcoating is about the same magnitude or less than the scatter of the experimental data at temperatures greater than 200°C. Consequently, in order to be able to obtain results in a reasonable length of time we must resort to high current densities. For example, at 200°C a conductor with a cross sectional area of 10\(^{-7}\) cm\(^2\) (≈ 1µ thick, 0.4 mil wide) would have according to Black\(^{(27)}\), a mean time to failure of somewhat more than 200 hours if the current density were 5 x 10\(^{5}\) amps/cm\(^2\); and only 50 hours at a current density of 1 x 10\(^{6}\) amps/cm\(^2\). This latter period of time is an experimentally convenient one, but if the temperature is reduced as little as 50°C the expected mean time to failure rises to 200 hours; about eight days. Consequently, the determination of the activation energy becomes a procedure of long duration. If the current density is increased enough so that the expected mean time to failure is experimentally convenient at the lowest temperature to be investigated, then it becomes awkwardly short after raising the temperature the minimum amount necessary to get a temperature range sufficient to give a useful estimation of the activation energy. For example, suppose that a temperature range of 100°C is considered to be the minimum and that the maximum temperature to be examined will be 200°C (For the reasons given above). At the lowest temperature to be examined, 100°C, a current density of 5 x 10\(^{6}\) amps/cm\(^2\) will give an expected mean time to failure of 55 hours, which is a reasonable period of time. However, at the uppermost end of the range, 200°C, the expected mean time to failure will be only slightly more than two hours. The thickness must not be reduced too much or
difficulty will be experienced in making bonds which do not fail before the stripes. We have used temperatures of 150° and 200°.

It is important to emphasize that the time periods which have been mentioned are means. They are only identical to the true lifetime if all samples fail simultaneously, which they don't. If the assumption is made that the distribution of true lifetimes of all the samples is symmetrical, then the length of time required for 50% of the samples to fail is identical to the mean time to failure. It has not been established that the distribution is symmetrical. If the distribution is sufficiently narrow, the shape of the distribution is of no importance. The various factors which affect the size and shape of the distribution of lifetimes have been discussed in Section 5.5.

5.6.4 Our electromigration test apparatus consists of an oven and twelve power supplies. Each power supply can operate in the constant current mode and yield up to 500 mA with a voltage compliance of 50V. Associated with each power supply is the ancillary equipment shown schematically in Figure 23. The potentiometer is adjusted so that without the test unit in the circuit the relay just barely closes and the neon lamp lights. With the test unit in place, the relay remains open, the elapsed time indicator operates and the neon lamp does not. When the test unit fails, the relay operates, turns the neon lamp on and the elapsed time meter off. The elapsed time meter reads in units of 0.1 hour.

We attempted to make it possible to test a number of samples greater than the number of power supplies by wiring a number of the samples in series. Each sample was provided with a SPST switch which could be closed in order to bypass the defective test units. This scheme has the disadvantage that the test time ceases to accumulate for the entire string of specimens from whenever one of the test stripes burns out until the time that it is bypassed. A much greater disadvantage, and the one which caused the scheme to be abandoned, was that transients occurred when the switches were operated and frequently caused apparently instantaneous failures.

5.6.5 Very early in our work on electromigration we found that when a test stripe failed it frequently destroyed the stripe adjacent to it. Photograph 25 shows a conductor which has failed due to electromigration.
Figure 23. Schematic Diagram for Electromigration Experiments.
Photograph 26. Destruction of a Metal Stripe
Due to Electromigration-induced
Failure of an Adjacent Metal Stripe
The current density was $6.3 \times 10^5$ amps/cm² and the current was 100 mA. Next to it, and separated by 0.3 mil, is another stripe. This second stripe was not carrying current but when the first one opened up the violence of the arc evidently was sufficient to destroy the second.

Just how important this mechanism may be to the user of integrated circuits cannot be evaluated at present. Certainly it is of no consequence if the conductors are widely separated. Examination of our raw electromigration data suggests that it operates for stripes as large as 1 mil spaced as far apart as 0.5 mil. Typical stripe dimensions are less than 1 mil. As circuit complexity increases we can expect to see a greater number of situations where this mechanism could operate. It would seem that a silica overcoat would contain the violence of the vaporizing metal but we have no data on the relationship between the minimum thickness of the overcoat, the spacing of the conductors, and the current density and operating voltage. On the other hand, for many integrated circuits two electromigration-caused open circuits are no worse than one in which case the existence of this failure mechanism is of no importance. In any case the use of an overcoat is probably a good idea even if the only reason that it lengthens the lifetime is that it prevents delamination of the stripe.

5.6.6 Our initial results were very poor from the standpoint of the lifetimes observed. It was concluded that some of the secondary factors were dominating the results. Accordingly, we instituted a routine inspection of the units with a lower power (80X) microscope prior to testing. Units which had visible flaws, such as scratches or notches were rejected. The results of this inspection can be seen in Table 8 where the values of the parameter B are compared. Note that B has decreased by a factor of about three. This means, that if the current density and cross sectional area were all equal, the lifetime has also been increased by a factor of three.

**TABLE 8**

<table>
<thead>
<tr>
<th>Lot</th>
<th>Inspected?</th>
<th>$B = a^3/I^2M$ (cm², amp², hrs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>No</td>
<td>$1.7 \times 10^{-21}$</td>
</tr>
<tr>
<td>2</td>
<td>No</td>
<td>$2.0 \times 10^{-21}$</td>
</tr>
<tr>
<td>3</td>
<td>Yes</td>
<td>$5.6 \times 10^{-22}$</td>
</tr>
</tbody>
</table>
5.6.6.1 The majority of our electromigration experiments were made using a batch of oxidized silicon slices on which 3820Å of aluminum had been deposited by electron beam evaporation at a rate of 8.4Å/sec. The substrates were at ambient temperature and the pressure during the deposition was $1.4 \times 10^{-5}$ torr although just prior to the evaporation the pressure was $3.2 \times 10^{-6}$. The grain size was less than 1 μ in the largest dimension.

For each group of samples of identical material, nominal width, post-etching treatment, and test temperature the average lifetime was used to calculate the value of the parameter B. Figure 24 shows this parameter plotted as a function of the reciprocal of the absolute temperature for four groups of samples. For those groups which were identical except for test temperature the activation energy for electromigration was calculated. Values of 0.44 eV and 0.41 eV were obtained for groups of 1 mil and 1/2 mil nominal width, respectively. These values are in fair agreement with the value of 0.48 eV reported by Black (27) for small crystallite films.

From the discussion in Section 5 it should be apparent that the samples having the longest lifetime might be more relevant to intrinsic electromigration than are the average lifetimes. Accordingly, the activation energy was calculated using the maximum observed values of the lifetime. For 1 mil and 1/2 mil nominal widths values of 0.73 eV and 0.52 eV, respectively, were obtained. The values of the parameter, B, are also seen in the graph of Figure 24. Observe that although there has been a considerable decrease in B the activation energy is not drastically changed. Less change, in fact, than would be observed in going from small crystallite films to those of large crystallites. This is exactly what would be expected from the discussion in Section 5.5 and 5.6.2. One would not expect most of the secondary factors which influence extrinsic electromigration to be thermally activated. Whereas large extremes in temperature might possibly be expected to influence the adhesion to the substrate, the temperature range used, 50°, would not. In the ideal case the test vehicle would be such that secondary factors were non-existent. In such a case there would be little difference in the average and maximum values of B. Or, in other words, the distribution of lifetimes would be very narrow. Additional evidence which confirms the correctness of the analysis comes from the observation that the value of B is always
Figure 24. Arrhenius Plot of Electromigration Results for 3820Å Film
Straight Lines Drawn from Black's Data (27)

-89-
lower for the narrower specimens, even though, in the case where average values were used, the activation energies were not notably different.  

5.6.6.2 In order to be able to make a comparison with the results just mentioned some additional samples were prepared using a batch of oxidized silicon slices on which 2950Å of aluminum had been deposited by electron beam evaporation in a different laboratory and by a different operator. The rate was 4.1Å/sec and the substrate was at ambient temperature. The pressure at the start of the deposition was 4-5 x 10^{-6} torr and the pressure just prior to the evaporation was 1.3 x 10^{-6}. The Arrhenius plot of the data is shown in Figure 25. It is clear that this sample of aluminum is much worse, from the standpoint of resistance to electromigration, than was the previous sample; the values of the activation energy being 0.17 eV and 0.10 eV for 1 and 1/2 mil nominal widths, respectively. Using maximum values of the lifetime gave 0.46 eV and 0.21 eV for the corresponding values. Note that once again, when average values were used, there was a relatively small difference in the activation energy for the two widths compared to when maximum values were used. The SEM showed the same grain size. Thus the chief difference in the two samples was in the deposition rate, the second being done at a rate about half that of the first. This certainly doubles the chances of oxidation. It is probably significant that whereas, when this deposition was performed, the parameters employed were standard in that laboratory, they are no longer.  

5.6.6.3 In order to determine if the lifetime could be influenced by a post-etching heat treatment we searched for conditions under which the aluminum crystallites could be regrown without concomitant oxidation. The samples, after etching and dicing were heated for various lengths of time and temperatures. The experimental conditions and the results are shown in Table 9.  

It can be seen that both experiments using a 200° heat treatment were unsuccessful probably because oxidation of the aluminum proceeded faster than regrowth of the crystallites.  

In the case of treatments at 350° a similar result obtains if the treatment is prolonged. However, it appears that a 350° treatment for between 21.5 hours and 3 days results in a noticeable decrease in the value of B. The greatest improvement is noted when one compares the 1/2 mil nominal width samples in runs H and F. The ratio of the corre-
Figure 25. Arrhenius Plot of Electromigration Results for 2946Å Film
Straight Lines Drawn from Black's data(22)
<table>
<thead>
<tr>
<th>Run</th>
<th>Treatment</th>
<th>1 Mil Nominal</th>
<th>1/2 Mil Nominal</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>B&lt;sub&gt;AV&lt;/sub&gt;</td>
<td>B&lt;sub&gt;Max&lt;/sub&gt;</td>
</tr>
<tr>
<td>H</td>
<td>None</td>
<td>5.6 x 10&lt;sup&gt;-22&lt;/sup&gt;</td>
<td>1.2 x 10&lt;sup&gt;-22&lt;/sup&gt;</td>
</tr>
<tr>
<td>C</td>
<td>200° 12 days</td>
<td>1.5 x 10&lt;sup&gt;-21&lt;/sup&gt;</td>
<td>3.4 x 10&lt;sup&gt;-22&lt;/sup&gt;</td>
</tr>
<tr>
<td>D</td>
<td>200° 27 days</td>
<td>1.1 x 10&lt;sup&gt;-21&lt;/sup&gt;</td>
<td>3.9 x 10&lt;sup&gt;-22&lt;/sup&gt;</td>
</tr>
<tr>
<td>E</td>
<td>350° 21.5 hours</td>
<td>1.0 x 10&lt;sup&gt;-22&lt;/sup&gt;</td>
<td>6.4 x 10&lt;sup&gt;-22&lt;/sup&gt;</td>
</tr>
<tr>
<td>F</td>
<td>350° 3 days</td>
<td>4.7 x 10&lt;sup&gt;-22&lt;/sup&gt;</td>
<td>3.5 x 10&lt;sup&gt;-22&lt;/sup&gt;</td>
</tr>
<tr>
<td>G</td>
<td>350° 15 days</td>
<td>6.5-8.9 x 10&lt;sup&gt;-22&lt;/sup&gt;</td>
<td>3.3 x 10&lt;sup&gt;-22&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Note: The conditions shown under Treatment are not to be considered as optimum; they represent the results of individual experiments. It is probable that the optimum conditions will be influenced by the nature of the aluminum.
sponding values of B is $2.3/0.49 = 4.7$. This means that if one compares samples which differ only in the presence or absence of this post-etching heat treatment ($350^\circ C$, 3 days) one finds the lifetime to have increased nearly five times.

An increase of lifetime of this magnitude is not inconsiderable but is by no means to be considered as a maximum. From inspection of the data it appears that oxidation of the aluminum is responsible for the limitation on improvement that was observed. It would be of great interest to continue these experiments under conditions in which oxidation was precluded: in an ultra-high vacuum system for example. At the time this work was done we did not have such a system available, hence our choice of the conditions actually used.

One more conclusion which can be drawn from the results of this experiment is that the observations in the literature cited previously in which crystallite size influenced lifetime are confirmed whereas the contrary ones are not.

**5.7 The Real Value of Electromigration Testing**

From the discussion in Section 5.6.3 it can be seen that the only way to reduce the time required to obtain significant results from electromigration experiments at temperatures which will allow separation of the subtle influences of crystallite size or the presence or absence of a silica overcoat is, for any given power supply, to reduce the cross sectional area of the test conductor. If this reduction is made by only decreasing the thickness, the extension of the results obtained to normally designed integrated circuits becomes tenuous. If, instead of reducing the thickness, the cross sectional area is decreased by reducing the width of the conductor, then the irregularities and uncertainties in the width, produced by the etching operation, become so significant as to dominate the results obtained. In other words, if the slice is over etched, or the photoresist has partially failed, or if a similar accident has happened then this will be revealed in the diminished lifetime when subjected to electromigration conditions. In fact, if any of the secondary factors discussed in Section 5.5, which lead to extrinsic electromigration failure are of a magnitude which is greater than the norm, they will signal that fact through the reduced lifetime of the test sample. Thus, it should be clear, that electromigration testing is of
great value in ensuring that all processes were under control when the device was being manufactured.

5.7.1 There are three major advantages to this type of test.

5.7.1.1 For the purposes of extrinsic electromigration failure one fault is as bad as many. In general, the fault which is worse, that is the one which results in the minimum cross sectional area, will be the one which will be responsible for the ultimate failure of the test stripe. Of course, the possibility exists that another region of reduced cross sectional area, due to a different fault, can grow smaller fast enough so that it becomes the failure site. Such a situation might arise in the vicinity of an improperly made gold-aluminum thermocompressive bond. In the case of faulty adhesion the failure does not result from a region which initially had a reduced cross sectional area but rather from one which had a temperature gradient. In any event there will be a failure and that failure will be due to the worst fault. It will not be the result of the cumulative effect of many minor faults. Having made that statement we must now qualify it: it is possible to conceive of a stripe which has a notch in it (because, say, of a dirt particle in the photoresist) which also has poor adhesion. Certainly, in this case the failure will be due to the presence of a thermal gradient in a stripe of reduced cross sectional area. Moreover, on examining devices which had failed, we have found some which had two regions from which all the metal had disappeared. Obviously, those were cases where more than one fault influenced the ultimate failure.

5.7.1.2 In Section 5.5 we mentioned the commonly recognized statistical nature of failures due to electromigration and then discussed a number of reasons for this. When electromigration testing is to be used for surveillance of the totality of control of all the processes, as we are now considering, there is no necessity for using a large number of samples. A relatively small number would be sufficient and this is a definite asset.

5.7.1.3 Determining the time to failure of a test stripe is obviously a test which is destructive of the stripe, but need not damage any other device included on the chip. (See Section 5.6.5 for a possible exception to this.) Consequently it may be possible to devise a scheme whereby a finished and packaged integrated circuit can be tested in order to
qualify the device without actually stressing the circuit itself.

5.7.2 The principle disadvantage to electromigration testing is that it does not indicate exactly which process went out of control. Examination of the test units which have failed can sometimes give information as to the primary cause of failure. This is especially true if the cause was improper bonding. Generally the only conclusion that can be drawn from the result that a test stripe failed earlier than anticipated is that something went wrong.

5.7.3 In Section 5.7.1.3 we have alluded to the use of electromigration testing in device qualification. Imagine that each chip which carries an integrated circuit also carries a suitably designed electromigration test stripe. When the chip is packaged and lead bonded the test stripe would also be lead bonded and connected to a pair of leads. Using these leads, samples of the device could be subjected to suitable conditions and the mean time to failure determined. If this was less than expected, either from experience or Black's or similar results, the conclusion could be drawn that the associated integrated circuits had not been processed according to design specifications. One would therefore be justified in not qualifying the devices for use. If two samples, or two stripes on the same chip, were tested at different temperatures, it would be possible to determine the activation energy, a useful indicator of metal quality.

The requirements for a test vehicle to be used for device qualification is different from those discussed previously. For the present purposes the test stripe should be long and narrow and should cross the same type of steps crossed by the device metallization. Because of the considerations mentioned in Section 5.6.3 the narrowest stripe utilized in the device should be employed for the test stripe, and the current density selected so as to allow the test duration to be a reasonable length of time. The ambient temperature can be high because it is not desired to detect the subtle differences which are masked at temperatures greater than 200°C.
6. APPLICATION OF THE PROPOSED TEST METHODS

In order to prevent any ambiguity from arising we wish to define certain terms used in this section. By "run" we mean a collection of slices, all of which are processed together. By "lot" we mean a collection of slices, all of which have the same devices on them and all of which started through the processing sequence at the same time. In applying the Proposed Test Methods we used three runs which, as it happened, consisted of four lots.

6.1 Nature of the Production Runs Monitored

The Proposed Test Methods were used to monitor three production runs of MOS/LSI devices. These runs are identified in Table 10.

TABLE 10
IDENTIFICATION OF PRODUCTION RUNS.

<table>
<thead>
<tr>
<th>Run</th>
<th>Lot No.</th>
<th>Name of Device</th>
<th>Slices</th>
</tr>
</thead>
<tbody>
<tr>
<td>48</td>
<td>W-150</td>
<td>Universal Register</td>
<td>18-27</td>
</tr>
<tr>
<td>49</td>
<td>W-151</td>
<td>Universal Register</td>
<td>30-38</td>
</tr>
<tr>
<td></td>
<td>W-149</td>
<td>Combiner</td>
<td>12-16</td>
</tr>
<tr>
<td>54</td>
<td>W-158</td>
<td>Combiner</td>
<td>76-80</td>
</tr>
</tbody>
</table>

6.2 Nature of the Test Specimens

For each run three test specimens were included. One was a silicon slice from the same origin as that used for the devices and the other two were 2" x 2" x 1/8" Pyrex flats. Only one of these flats was required for each run but, because it was desired to learn something about cleaning techniques, one was cleaned with detergent and the other with dilute HF just prior to aluminum evaporation.

The silicon slices were from Ingot G013-3-2, n-type, 111, 3.5-4.4 n cm. The boron diffusion gave 87 μ on a monitor under conditions which generally resulted in a layer 2.7 μ deep. The contact pattern consisted of squares 89 μ on a side and were arranged in a line on 200 μ centers.
6.3 Tests Applied

6.3.1 The resistivity was determined from the measured sheet resistance and the measured thickness of the deposit (q.v.). The sheet resistance was measured at the center of the Pyrex flats by the four-point probe technique using a current of 4.45 mA. In addition to the resistivity, the ratio of the resistivity to the bulk resistivity was also calculated. For all six specimens, the ratio was 1.1 thus indicating (1) a high degree of consistency in the deposition parameters, and (2) that a high-quality aluminum film was being deposited. The data is shown in Table 11.

<table>
<thead>
<tr>
<th>Run</th>
<th>Sample</th>
<th>E</th>
<th>I</th>
<th>( R_s )</th>
<th>Center Thickness</th>
<th>( \rho )</th>
<th>( \rho/\rho_{\text{Bulk}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>48</td>
<td>3</td>
<td>( 23.3 \times 10^{-6} \text{V} )</td>
<td>4.45 mA</td>
<td>0.0233 ( \Omega/\square )</td>
<td>1.30 ( \mu )</td>
<td>3.02 ( \mu \text{\Omega/cm} )</td>
<td>1.1</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>( 23.0 \times 10^{-6} \text{V} )</td>
<td>4.45 mA</td>
<td>0.0230 ( \Omega/\square )</td>
<td>-</td>
<td>2.98 ( \mu \text{\Omega/cm} )</td>
<td>1.1</td>
</tr>
<tr>
<td>49</td>
<td>5</td>
<td>( 22.5 \times 10^{-6} \text{V} )</td>
<td>4.45 mA</td>
<td>0.0225 ( \Omega/\square )</td>
<td>1.32 ( \mu )</td>
<td>2.97 ( \mu \text{\Omega/cm} )</td>
<td>1.1</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>( 22.3 \times 10^{-6} \text{V} )</td>
<td>4.45 mA</td>
<td>0.0223 ( \Omega/\square )</td>
<td>-</td>
<td>2.94 ( \mu \text{\Omega/cm} )</td>
<td>1.1</td>
</tr>
<tr>
<td>54</td>
<td>1</td>
<td>( 22.5 \times 10^{-6} \text{V} )</td>
<td>4.45 mA</td>
<td>0.0225 ( \Omega/\square )</td>
<td>-</td>
<td>2.86 ( \mu \text{\Omega/cm} )</td>
<td>1.1</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>( 22.5 \times 10^{-6} \text{V} )</td>
<td>4.45 mA</td>
<td>0.0225 ( \Omega/\square )</td>
<td>1.27 ( \mu )</td>
<td>2.86 ( \mu \text{\Omega/cm} )</td>
<td>1.1</td>
</tr>
</tbody>
</table>

*Based on a bulk resistivity of 2.7 \( \mu\Omega\text{-cm} \).

6.3.2 In order to determine the thickness of the deposit, a step was produced on one of each pair of Pyrex flats by using the etching technique specified in the Proposed Test Method. After depositing a reflective layer of aluminum, we measured the thickness using a Varian A-Scope. The data is shown in Table 12.
It can be seen that the range in the first two runs was quite small, but somewhat wider for the third. On checking on the causes for this it was found that the third run was performed using a deposition rate 50% higher than that for the first two. It has been shown that in electron beam evaporation the distribution of the vapor stream is changed by increases in the applied power. (50)

6.3.3 The data obtained from testing for mettallization adherence according to the proposed test method is shown in Table 13. In the column labeled Critical Load are given two numbers. The first is the maximum load under which the film did not fail, and the second is the minimum load under which it did.

<table>
<thead>
<tr>
<th>Run</th>
<th>Sample</th>
<th>Cleaning Method</th>
<th>Critical Load</th>
</tr>
</thead>
<tbody>
<tr>
<td>48</td>
<td>3</td>
<td>Detergent</td>
<td>60 - 63g</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>Etch</td>
<td>60 - 63g</td>
</tr>
<tr>
<td>49</td>
<td>5</td>
<td>Detergent</td>
<td>68 - 70g</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>Etch</td>
<td>74 - 76g</td>
</tr>
<tr>
<td>54</td>
<td>1</td>
<td>Etch</td>
<td>65 - 68g</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>Detergent</td>
<td>62 - 64g</td>
</tr>
</tbody>
</table>

It will be observed that, except for Run 48 in which there was no difference observed, cleaning with detergent results in poorer adhesion than cleaning by etching. The difference is small, but significant. In
order to have a comparison, we also determined the adherence of a well-aged Ti-Al sample. The critical load was found to be 140 - 150g; about twice that of the production samples. It should be emphasized that the production samples were aluminum only and that they were less than two weeks old. Several investigators have noted that adherence of aluminum films generally improves on aging. (18,19,49)

6.3.4 The results of qualitative testing of ohmic contacts are pretty well summarized in Photographs Nos. 27 and 28 which happen to be taken from run 48. The first photograph shows that the traces were linear, as they were when made using the most sensitive settings of the curve tracer. The second photograph also appears to show a linear trace, but if one places a straight edge against it, one finds a very slight upward (towards higher current) curvature in both current directions. This could easily be overlooked and serves to emphasize the importance of the quantitative test of ohmic contact quality.

When we wrote our Proposed Test Method on ohmic contacts, we followed Terry and Wilson (22) and specified current levels of 10 and 100 mA. While these levels may be realistic for some devices, for others they are not. Certainly they are not for the MOS-LSI devices we were monitoring, where the maximum currents are about 1 mA. Nevertheless, testing at 10 and 100 mA is more revealing of contact quality than testing at, say, 1 and 10 mA. This is shown in Table 14.

Examine first the data for run 48 and note that the contact resistance is greatly different for 10 and 100 mA. If the contact were ideal, this resistance would not change with current. Such behavior can be seen in the data for runs 49 and 54 where the contact resistance is essentially unchanged by the current changing from 1 to 10 mA. The fact that the measured resistance is essentially independent of current direction indicates that the current carrying contacts are all behaving in a similar way.

Ordinarily, contact resistance would not be measured before sintering. We did so to illustrate a point which we emphasized in Section 4; namely, that it is not necessary to use extreme sintering conditions if the contact
Photograph No. 27

Vertical: 1 mA/div
Horizontal: 0.05V/div

Photograph No. 28

Vertical: 10 mA/div
Horizontal: 0.5V/div

Lower trace is with current in reverse direction
### TABLE 14. QUANTITATIVE TESTING OF QUALITY OF OHMIC CONTACT

<table>
<thead>
<tr>
<th>Run</th>
<th>Location on Slice</th>
<th>I</th>
<th>R(fwd)</th>
<th>R(rev)</th>
</tr>
</thead>
<tbody>
<tr>
<td>48 (after sintering)</td>
<td>Center</td>
<td>10 mA</td>
<td>12.8 Ω</td>
<td>12.4 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 mA</td>
<td>69.7 Ω</td>
<td>66.6 Ω</td>
</tr>
<tr>
<td></td>
<td>Edge</td>
<td>10 mA</td>
<td>12.5 Ω</td>
<td>12.2 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 mA</td>
<td>68.1 Ω</td>
<td>66.0 Ω</td>
</tr>
<tr>
<td>49 (before sintering)</td>
<td>Center</td>
<td>10 mA</td>
<td>16.8 Ω</td>
<td>16.4 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 mA</td>
<td>49.7 Ω</td>
<td>49.2 Ω</td>
</tr>
<tr>
<td></td>
<td>Edge</td>
<td>10 mA</td>
<td>16.6 Ω</td>
<td>15.9 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 mA</td>
<td>48.2 Ω</td>
<td>97.1 Ω</td>
</tr>
<tr>
<td>49 (after sintering)</td>
<td>Center</td>
<td>1 mA</td>
<td>13 Ω</td>
<td>14 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10 mA</td>
<td>13.5 Ω</td>
<td>14.4 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 mA</td>
<td>57.3 Ω</td>
<td>58.0 Ω</td>
</tr>
<tr>
<td></td>
<td>Edge</td>
<td>1 mA</td>
<td>13 Ω</td>
<td>13 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10 mA</td>
<td>13.7 Ω</td>
<td>13.2 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 mA</td>
<td>55.9 Ω</td>
<td>57.9 Ω</td>
</tr>
<tr>
<td>54 (before sintering)</td>
<td>Center</td>
<td>10 mA</td>
<td>15.5 Ω</td>
<td>15.5 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 mA</td>
<td>54.3 Ω</td>
<td>51.6 Ω</td>
</tr>
<tr>
<td></td>
<td>Edge</td>
<td>10 mA</td>
<td>16.5 Ω</td>
<td>16.7 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 mA</td>
<td>50.0 Ω</td>
<td>48.5 Ω</td>
</tr>
<tr>
<td>54 (after sintering)</td>
<td>Center</td>
<td>1 mA</td>
<td>14 Ω</td>
<td>14 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10 mA</td>
<td>13.2 Ω</td>
<td>12.8 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 mA</td>
<td>57.0 Ω</td>
<td>61.4 Ω</td>
</tr>
<tr>
<td></td>
<td>Edge</td>
<td>1 mA</td>
<td>14 Ω</td>
<td>14 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10 mA</td>
<td>13.6 Ω</td>
<td>14.0 Ω</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 mA</td>
<td>53.1 Ω</td>
<td>56.3 Ω</td>
</tr>
</tbody>
</table>
holes are completely clean before the deposition of the aluminum. In the cases at hand, the contacts were almost as good before sintering as after. After one hour at 450°C in argon, they were somewhat improved when measured at reasonable current levels. An exception to this can be seen in the comparison of the data for Run 49 taken near the edge using a current of 100 mA before and after sintering. Observe that there was considerable difference in $R_{(\text{fwd})}$ and $R_{(\text{rev})}$ before sintering but not after. This probably represents an area of the wafer in which the contact holes were not completely clean prior to sintering. However, the degree to which they were not clean must have been small considering that the relatively mild sintering conditions used were sufficient to make them the equal of the contacts at the center.

In conclusion we can state that the runs monitored were in an excellent state of control with the exception of the uniformity of thickness in Run 54. In this case the spread, which was by no means excessive, indicated that a correction in the deposition rate or the position of the substrates should be made. The combination of contact hole cleanliness and sintering conditions was more than adequate for the devices being manufactured.
7. CONCLUSIONS

7.1 Existing Test Procedures

(1) Test Methods 6070-5 of "Test Standards for Microcircuits" contain certain errors and deficiencies.

(2) Procedures related to thickness and uniformity of metallization, Nos. 6070A, 6070B, and 6070C, should be deleted and replaced by two new Test Methods. These new Test Methods consist of one for the determination of thickness and one for the determination of resistivity.

(3) "Scotch tape" tests have only limited usefulness and the Test Methods employing them, Nos. 6071A and 6071B should be deleted.

(4) Test Methods concerned with the quality of ohmic contact should be combined.

7.2 New Test Methods

(1) Since the resistivity is very sensitive to small amounts of impurities or changes in the deposition parameters, it should be used as a prime indicator of metallization quality.

(2) The proposed Test Method on the determination of the resistivity of an aluminum film, which uses an accurate determination of the thickness, together with the sheet resistance of the film as determined by a four point probe measurement should be adopted.

(3) The new method for producing a step for the Tolansky multiple beam interference method of thickness determination should be used.

(4) The entirely new Test Procedure for determining metallization adherence, which involves moving the film under a stylus having a variable load should be adopted.

(5) Current level, polarity, and instrument sensitivity are of great importance when doing a qualitative determination of contact quality.

(6) Both qualitative and quantitative determinations of contact quality should be performed as process monitors.

7.3 The Microcrack Problem

(1) As long as the metal is deposited by the usual evaporation methods, the potential for microcrack formation will be present, especially since no Test Method has been devised for monitoring the thickness of the metal on the walls of a contact hole.
(2) For a variety of types of silica masked with KTFR and etched with buffered oxide etch, the angle between the wall and the original surface of the silica is not 45° as would be expected for an isotropic, amorphous substance but rather 34 ±3°.

(3) The profile of a hole etched into a film of one or more types of silica can be predicted.

(4) Microcrack formation proceeds by surface diffusion driven by crystallite regrowth.

(5) Failure occurs where the metallization is thinnest and this in turn is determined by edge profile and evaporation technique.

(6) For any combination of edge profile and evaporation technique, failure is more likely to occur if the sintering conditions are severe.

(7) The processing sequence should be performed in such a way as to permit moderate sintering conditions to be used.

(8) There is a great need for a non-destructive technique for monitoring the contact holes.

(9) If the windows are clean one hour at 450°C in argon is sufficient to provide good contacts.

7.4 Electromigration

(1) There is a definite distinction between intrinsic and extrinsic failure under electromigration conditions.

(2) Extrinsic failures are influenced by the stripe width, stripe cross section, presence of abrupt steps, substrate asperities, lead bonding, adhesion to substrate, and frequency of faults.

(3) The error produced by one micron irregularities in the width of the stripe, such as might be caused by pinholes in the mask or by dirt on the mask or slice, or in the photoresist, is more than about 20% for stripes of one-half mil width or less.

(4) The error produced by assuming that the stripe has a rectangular cross section instead of a trapezoidal one is a function of stripe thickness. For 1 μ thick films the error is about the same as that contributed by stripe width.

(5) It is possible to design test vehicles which can either minimize or maximize the probability of extrinsic failure.
The results of our electromigration experiments are in agreement with Black's model. Activation energies of 0.44 eV and 0.41 eV for stripes of 1 mil and 0.5 mil nominal width respectively were found in agreement with Black.

The absence of thermal activation for factors influencing extrinsic failure is strongly suggested.

A new failure mechanism exists in which failure of one conductor due to electromigration results in the destruction of a conductor running parallel to it.

Electromigration testing is of greatest value as a monitor of the totality of influences of all the processes and may be of use in qualifying integrated circuits.

It is possible to give aluminum conductors a post-etching treatment which increases the lifetime.

Heating in nitrogen for three days at 350°C increased the expected lifetime by a factor of 4.7.

The work of previous investigators who claimed that large-grained aluminum conductors are more resistant to failure than small-grained ones has been confirmed.

7.5 Application of the Proposed Test Methods

The Proposed Test Methods are useful in monitoring the production of MOS/LSI devices.
8. SUGGESTIONS FOR FUTURE WORK

During the course of this work there were certain questions raised, answers to which we were unable to seek. Some of these are assembled here. In the final paragraph we list those which we think are of greatest importance.

8.1 Reliability

The purpose in our entire study has been to increase the reliability of devices to which the results of our study might be applied. Although the Test Methods which we, and others, have proposed should result in devices of higher reliability the question remains: assume that a certain set of Test Methods has been applied during the manufacture of a lot of devices. Is there really any difference in the reliability of that lot of devices and another lot manufactured using a different set of Test Methods? This question certainly should be answered. At the moment all that we can say is that the reliability probably won't be any worse. This is not a certainty, for as we have shown in Section 4.2.3 it is possible to apply a Test Method, discover a deficiency in one step of the process sequence, apply a "fix", and end with a device of greatly decreased reliability.

8.2 Device Qualification

We feel very sanguine about our proposed use of electromigration in order to qualify a device prior to use as mentioned in Section 5.7.3. In order for this method to be used a complete specification concerning the design of the test stripe, the method of testing, and the criteria to be used in assessing the results must be written.

8.3 Improvements In Techniques

In the course of developing new Test Methods or modifying the old ones, the need for certain improvements in the usual way of performing them became apparent.

In Section 2.2.2 we mentioned our use of sputtered copper as a reflective layer for use in the multiple beam interferometric method of thickness measurement. Someone ought to build a very simple dc sputtering apparatus consisting only of a chamber, copper cathode, mechanical pump, leak valve, and an unregulated dc power supply. The purpose would be to show how simple the apparatus can be in order to induce some apparatus manufacturer to put it on the market.
A similar goal would be the purpose in developing a microellipsometer as mentioned in Section 4.2.3.3. However, in this case, the need is far greater - and the probability of success somewhat less. In the Section cited we enumerated several places in the process sequence where such an instrument could be used to remove a considerable amount of guesswork.

We readily admit that the weakest part of our Proposed Test Method on metallization adherence lies in the determination of the end point. It should be possible to improve this by plotting the optical transmission of the track left by the stylus as a function of applied load. Possibly resistance measurements could be used instead of the optical transmission. If no discontinuity made itself apparent as a sign of adhesive failure perhaps the scanning electron microscope could be used to correlate the optical transmission or resistance change with adhesive failure.

8.4 Contact Hole Profile

In Section 4.2.2.2 we reported that a variety of types of silica, masked with KFTR and then etched, all gave essentially the same value for the angle between the wall and the original surface of the silica. We consider this finding of great importance to the device designer. However, for this finding to be exploited more fully it will be necessary to know if other resist materials give the same angle and if other resist materials give the same angle with all types of oxide. In addition, the types of oxide examined should be extended at least to include boron doped silane-silica. This material is unusual in that its etch rate in buffered oxide etch is less than undoped silane-silica. In addition, certain other etches such as P-etch and dilute HF should be examined to see what sort of wall angle they produce with various resists and oxide types.

8.5 Electromigration

A considerable number of questions concerning electromigration remain unanswered. In addition some new ones can now be asked. These are related to our results on the post-etching regrowth of aluminum films, to our discovery of a new failure mechanism, and to our experience with bond failures during the course of the electromigration experiments.

8.5.1 The post-etching regrowth results, while permitting an increase in the anticipated time to failure due to electromigration from, say, two to nearly ten years, are mainly indicative of what might be accomplished
by means of this stratagem. In particular, we anticipate a considerable improvement merely from changing the ambient. Instead of nitrogen one should use ultra-high vacuum. Possibly hydrogen or forming gas might be of value although hydrogen sometimes produces certain deleterious effects.

Epitaxy on a micro-scale might be used to decrease the time and/or temperature required. A typical experiment might involve first depositing a very thin layer of aluminum at a low rate using a high substrate temperature. Another way might be to apply a thin layer by sputtering. In either case the idea is to supply sufficient energy so that the initial deposit consists of a very thin layer of large crystallites. The remainder of the deposit would be put on at a high rate so that it would be fine grained and hence capable of high resolution etching. The initial layer of large crystallites would be so thin as not to interfere. After the film was etched it could then be subjected to regrowth conditions where-upon the small crystallites could grow epitaxially on the polycrystalline layer of large crystallites.

85.2 The existence of the new failure mechanism reported in Section 5.6.5 might cause difficulties in using a built-in electromigration test stripe for a qualification test. Care would have to be taken so that when the test stripe failed the violence of the arc produced did not damage the integrated circuit. Is it possible that this arc could be contained by the presence of a dielectric overcoat?

Dielectric overcoats are generally conceded to increase the mean time to failure due to electromigration. Thus it would be interesting to know why r.f. sputtered silica layers do not.\(^{(39)}\)

Overcoats are, of necessity, of a dielectric material but what would be the effect on life time of a heavy copper overcoat, suitably isolated? Chabra and Ainslie\(^{(30)}\) emphasize the importance of thermal gradients in determining the lifetime. Since the mounting pads are generally wider than the test stripe there is a thermal gradient at the end of the stripe and this, they say, is one of the reasons that the failures generally occur at that point. A copper overcoat, possibly tapered as it came over the beginning of the mounting pad, might serve to reduce that gradient and hence lengthen the lifetime. It would be of great interest to know the mean time to failure of a conductor which had no thermal gradients. The device described in Section 5.6.2 should be built and tested.
8.5.3 We have pointed out in Section 2.2.7.2 that the presence of intermetallics at a gold-aluminum thermocompression bond is not, in itself, harmful, and in fact is necessary. But the growth of voids, due to the Kirkendall effect, even if it does not lead to zero strength bonds can result in premature failure when the device is subjected to electromigration conditions. It may be possible to avoid, or at least delay for an exceedingly long time, this growth of voids by merely depositing a layer of the intermetallic compound AuAl₂ on the aluminum at any stage prior to forming the gold wire bond. A convenient way would be to sputter the AuAl₂ after depositing the aluminum. The excess AuAl₂ could then be removed using the same photoresist operation used to delineate the aluminum pattern.

In order to understand how this simple stratagem can provide higher reliability, it is necessary to review some experimental work recently reported by B. Selikson. (48) This investigator evaporated gold on one-half of a microscope slide and then evaporated aluminum so that the remaining half of the slide was covered, as well as a portion of the gold nearest the center of the slide. This gave him slides divided approximately in half into a gold portion and an aluminum portion with an intervening band of aluminum over the gold. The intervening bands had various widths. After aging in air at 200°C for a number of days, they were examined and it was found that the gold film disappeared from the far end of the gold band and that the disappearance occurred much more rapidly in those samples where the intervening band was narrower than in those in which it was wide. This suggested that it was the flaw through the intermetallic band which controlled the interaction rate of aluminum and gold and that this was the rate governing process for the formation of voids. (It is necessary to understand that the gold and the aluminum in the intervening band would react to form an intermetallic compound in 24 to 36 hours at the temperature used to age the samples.) From his experiments, the author concluded that, in the case of semiconductor devices: "...diffusion of the two reacting metals will proceed, but at rates which are limited, not only by the anneal temperature, but also by the diffusion of each metal through the formed layer of intermetallic compound(s). Since these rates may be expected to be different, diffusion rate discrepancy will cause a vacancy buildup which will condense to
produce voids. This will result in bond weakness and eventual opening." Microscopic examination of gold wire bonds to aluminum has shown that the thickness of the intermetallic zone is seldom more than 5µ. If a 5µ-thick layer of AuAl₂ is deposited on the aluminum and then the gold wire bond is made, one is assured of having a larger than usual intermetallic zone because the AuAl₂ will react with the gold to produce some, if not all, of the four other possible intermetallic compounds. And, from the experimental work cited, the thicker the intermetallic zone, the slower the flow of gold from the ball (or the aluminum from the stripe), and the longer the lifetime of the bond.

8.6 Priorities

Probably not all of these suggestions can be followed, hence it is not amiss for us to direct the reader’s attention to those questions which we feel are most important. To a certain extent this assignment of priorities is peculiar to our requirements and to our interests.

8.6.1 Is there really any difference in the reliability of one lot of devices manufactured under one set of Test Methods and another lot manufactured under a different set?

8.6.2 Can a microellipsometer capable of monitoring contact holes be devised?

8.6.3 Do all combinations of oxide type, photoresist type, and etch give the same wall angle?

8.6.4 What is the maximum increase in lifetime which can be realized by a post-etching heat treatment?

8.6.5 Can the lifetime of gold thermocompression bonds be increased by the intentional use of a layer of purple plague?
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(12) Signatron Corporation, Sunnyvale, California


(15) R.W. Berry, P.M. Hall, and M.T. Harris, loc cit, p 176.


(17) O.S. Heavens, J. Phys. Radium 11, 335 (1950)


(37) Floor discussion at the meeting just cited.


(39) Sello's reference reads: "W.E. Mutter, J. Electrochem Soc., 114, 59C (1967)." However, the brief abstract printed in the location cited does not mention the use of r.f. sputtered silica.


(43) T.E. Hartman and J.C. Blair, ibid.


(49) C. Weaver and R.M. Hill, Phil. Mag. 4, 1107(1959).

APPENDIX A

EXISTING TEST METHODS
1. DISCUSSION

The evaporation of the aluminum metalization on monolithic microcircuit wafers is performed under vacuum conditions which must be the best obtainable consistent with productivity. In no case should the pressure exceed $10^{-5}$ mm Hg. In a given system the amount of the aluminum charge, heater current, and time are the variables affecting the average thickness of the aluminum on those areas normal to the source. The optimum aluminum thickness must be selected and verified on the basis of design, process, and reliability considerations. Once established the design thickness must be maintained (with tolerances) to assure an adequate thickness on those areas of the circuit which are not normal to the source (i.e. the pronounced topographical variations of the circuit resulting from the series of photolithographic operations).

The evaporation cycle is subject to two types of control:

1. Control of evaporation cycle
2. Control of uniformity of deposit.

The control of the evaporation cycle is typically performed by commercially available thickness monitoring equipment. The measurement principle is based on the calculable detuning of a crystal oscillator proportional to the mass of the build up of the evaporant on the exposed face of the crystal. (Figure 1)

The control of uniformity of deposit is performed by the interference measurement on judiciously placed monitor wafers in the evaporation chamber. The interference method is also a control on the performance of the thickness monitor. The method described is basically the Nomarski method.
1. DISCUSSION (Continued)

A control tolerance of ±20% of the design thickness is adequate unless otherwise specified.

2. APPARATUS

Thickness Monitor  - Sloan Thickness Monitor
Sloan Instruments Corporation, Model DTM3

Sensor  - Model DTM-3 (water cooled)

Microscope  - Reichert Meta test "C"

Interferometer  - Watson Interference Objective 8mm

Camera  - Polaroid Land

Light Source  - Sodium

3. ACCURACY

The accuracy of the monitoring system instrumentation in relating frequency drift to the build up of evaporant on the sensor crystal surface is ±2% of full scale reading.

The precision with which the monitor cut off defines the point at which the design thickness of aluminum is obtained and the uniformity thereof, is determined by the displacement of the interference fringe pattern to ±1/2 fringe of sodium light.

4. PROCEDURE

Sample  - Unoxidized monitor wafers or portions thereof will be placed in the evaporator chamber in a pattern judiciously selected among the in-process wafers.

Preparation  - Each monitor wafer will be appropriately masked so that a step down to the silicon surface will remain after evaporation is complete. The demarcation line between evaporated aluminum and adjacent silicon will be as straight as possible.

Figure 2 demonstrates one method of masking which results in a sloped edge. The sloped edge makes it possible to evaluate thickness directly from a single black and white photograph.

-2-
Evaporation cycle will be completed in accordance with detail specification.

**Measurement**
- The monitor wafer will be positioned under the interference objective such that the edge formed on the silicon by the evaporated metal will be centered in the field.

The adjustment of the interference objective will be manipulated until the fringe pattern is clearly established.

A Polaroid photograph will be taken of the image obtained.

**Evaluation**
- Figure 3 shows the type of interference pattern characteristic of the film thickness measurement. The thickness of the film is determined from the quantities defined in Figure 4.

### 5. METHOD DOCUMENTATION

The following must be supplied in support of thickness control:

1. Specification of Design thickness and tolerance
2. Procedural instructions.
FIG 1 Thickness Monitor Position in Relation to Process Wafers and Block Diagram of Associated Electronics (Sloan System)
FIG 2  Masking Scheme to Assure
Sloped Edge Between Metallization
Surface and Silicon Surface
FIG. 3 Characteristic Interference Pattern for Metalization Thickness Measurement
\[ \frac{D}{d} \lambda = T \]

- \( D \) = units of fringe displacement
- \( d \) = distance between fringes
- \( \lambda \) = wavelength of sodium light (2946 Å)
- \( T \) = thickness of Al film in Å

**FIG 4** Fringe Configuration and Evaluating Relationship
Method 6070 B

Thickness of Metallization
(Tolansky Interferometer)

1. Purpose

This test procedure is applicable to the measurement of any thin film. It utilizes the interference of monochromatic light introduced into the optical system through a 95% mirror and reflected between that and the metallized specimen. The two mirrors are inclined to one another, causing the interference fringes to be normal to the edge of the measured layer. The horizontal displacement of any fringe enables one to calculate the layer thickness by the following relationship:

\[ \frac{x_o}{D} = \frac{\lambda}{2w} \]

Where \( x_o \) = Film thickness
\( \lambda \) = Wavelength of incident light
\( w \) = Distance between fringes
\( D \) = Distance any fringe is displaced.

See Figures 1 and 2.

2. Apparatus

2.1 Metallurgical microscope
Metalstar trinocular body AO No. 2200T-4
American Optical Co.
(or equivalent)

2.2 Polaroid camera AO No. 1052A
American Optical Co.
(or equivalent)
2.3 Interferometer jig
   Fairchild design
   See Figure 3.

2.4 Monochromatic light source
2.4.1 Mercury, 5461 Å
   LaPine Scientific Co. Model 216-01
   (or equivalent)

2.4.2 Sodium, 5893 Å (optional)
   LaPine Scientific Co. Model 215-96
   (or equivalent)

2.5 Hot plate, 6" x 6", electric
temco, type 1900
Corning, Pyroceram PC-35
(or equivalent)

2.6 Polyethylene beaker
   (any source)

3. Materials
3.1 Hydrofluoric acid, 49%, reagent grade

3.2 Acetic acid, glacial, reagent grade

3.3 Trichloroethylene, electronic grade

3.4 Acetone, electronic grade

3.5 Apiezon wax

3.6 Slides, glass microscope, 1" x 3"

4. Procedure
4.1 Preparation
4.1.1 Process a polished test chip through the metalli-
   zation operation to be evaluated.

   NOTE: Form a step in the film either by etching or
   "shadowing" with a glass slide during evaporation.
4.1.2 Evaporate a 1000Å silver or aluminum film over the film to be examined.

NOTE: While silver has a higher index of reflectivity, it must not be allowed to tarnish prior to measurement.

4.2 Measurement

4.2.1 Place the specimen on the platform, mirrored side up.

4.2.2 Rotate the specimen on the platform until the step to be measured is normal to the front edge of the Fizeau plate.

4.2.3 Raise the platform until the specimen is in contact with the Fizeau plate by turning the knurled brass elevation nut. This will give maximum fringe spacing.

4.2.4 If a smaller fringe spacing is desired, the platform may be raised slightly, reducing the wedge angle, \( \theta \), and, as a result, fringe spacing.

4.2.5 Make a photomicrograph.

4.2.6 Measure distance between fringes on the photomicrograph, with a precision rule. (w)

4.2.7 Measure distance which any fringe is displaced as it passes across the film step. (D)

4.2.8 Calculate film thickness, \( x_0 \), using the relationship

\[
\frac{x}{D} = \frac{\lambda}{w}
\]

5. Calibration

Not applicable.
6. **Accuracy**

Accuracy obtainable increases with increasing film thickness.  
6.1 For films greater than 0.2 microns, ±10%.  
6.2 For films less than 0.2 microns, ±20%.

7. **Test Frequency**

This test should be performed on a periodic basis.
Figure 1. Tolansky Interferometer Setup
Figure 2. Film Measurement

Sodium, $\lambda = 5890 \text{Å}$

$W = 1.12 \text{ inches}$

$D = 0.14 \text{ inches}$

$X_0 = D \frac{\lambda}{2} \frac{1}{W}$

$= 0.14 \frac{5890}{2} \frac{1}{1.12}$

$= \frac{1.4 \times 2.945 \times 10^2}{1.12}$

$= 3.4 \times 10^2$

$= 340 \text{Å}$

NOTE: A fixed error of 30 - 50 Å becomes significant at this thickness range.
Figure 3. Interferometer Jig
1. **Purpose**

This procedure may be used for the indirect determination of a single metal evaporated film. It utilizes the measurement of a sheet resistance from which the film thickness is calculated using the following equation:

\[ w = \rho \left( \frac{1}{\pi} \frac{1n^2}{V} \right) \text{ (For } w \rightarrow \text{ Zero)} \]

Where:

- \( w \) = Film thickness in cm.
- \( \rho \) = Bulk resistivity of metal in \( \Omega \text{ cm} \).
- \( \frac{V}{I} \) = Sheet resistance.

2. **Apparatus**

2.1 Four-point probe, 0.050-inch spacing.

(any source)

2.2 Power supply, constant current, 0.1, 1.0, 10mA ranges.

(any source)

2.3 Voltmeter, Digital

Fairchild 7100

(or equivalent)
3. **Materials**

3.1 Glass slides, 1" x 3"

4. **Procedure**

4.1 Coat glass slides according to the process under evaluation.

4.2 Place the slide to be measured upon the probe stage. See Figure 1.

4.3 Lower the probe until it touches the sample lightly.

4.4 Record the voltmeter reading.

4.5 If the current source is 100\(\mu\)A, 1mA, 10mA, etc., the voltmeter will read directly in sheet resistance with application of the proper decimal point.

4.6 Using the formula from Para. 1, calculate the film thickness.

Example: \[
\frac{V}{I} = 0.006\Omega
\]

Film - Aluminum

\[
\rho = 2.72 \times 10^{-6} \Omega \cdot \text{cm}, 25^\circ \text{C}, \text{Aluminum}
\]

\[
w = \rho \frac{\ln 2}{\pi \frac{V}{I}}
\]

\[
= \frac{2.72 \times 10^{-6} \times .693}{\pi \times .006}
\]

\[
= \frac{2.72 \times 10^{-6} \times 6.93 \times 10^{-1}}{\pi \times 6 \times 10^{-3}}
\]

\[
= \frac{2.72 \times 6.93 \times 10^{-4}}{\pi \times 6}
\]

\[
= 1 \times 10^{-4} \text{ cm}
\]

\[
= 1 \text{ micron}
\]

\[
w = 10^4 \text{ Å}
\]
5. **Calibration**

5.1 Probe spacing

5.1.1 Cover a 1" x 3" glass microscope slide with aluminum foil.

5.1.2 Obtain an impression of the four probe points.

5.1.3 Measure spacing with any optical comparator.

5.1.4 Adjust spacing per probe manufacturer's instructions.

6. **Accuracy**

   Accuracy obtainable is approximately ±10%.

7. **Test Frequency**

   This test should be performed on a sample slide from every evaporation.
Figure 1.
1. DISCUSSION

The adhesion of the aluminum metallization to the surface dioxide of monolithic integrated circuit wafers is a direct function of the cleanliness of the oxide surface at the time the film is evaporated. The atomic intimacy of the evaporated film and the oxide surface promotes the characteristic tendency for aluminum to form a bond to the silicon dioxide in the absence of contamination. The adherence between the aluminum and silicon is enhanced by heat treatment during subsequent processes and is one major reason that aluminum is widely used for metallization of monolithic microcircuits.

The method for establishing the adequacy and uniformity of adherence is a qualitative one, involving the gross comparison of the metallization adherent force to the oxide with the adherent force of cellophane tape to the metallization film. This is commonly referred to as the "scotch-tape" test.

2. APPARATUS

Tape - Scotch Brand Cellophane Tape 1/2" wide
Microscope - Bausch and Lomb stereo Magnification 10X

3. PROCEDURE

3.1 Sample

The sample shall consist of an oxidized wafer.

3.2 Preparation

The sample wafer shall be subjected to pre-evaporation cleaning procedures concurrent with or as specified for in-process material.

3.3 Measurement

3.3.1 Apply tape to metallized surface observing precaution to avoid contamination of either adhesive side of tape or metallized surface wafer should be restrained on a flat surface and smoothed using a suitable non-metallic edge to squeeze out any entrapped air.
PROCEDURE (Continued - Measurement)

3.3.2 Allow taped sample to remain undisturbed for 3 minutes.

3.3.3 Free end on tape shall be held at an angle of 60 to 90° from plane of wafer.

3.3.4 Tape shall be rapidly "stripped" from the metalized surface.

4. EVALUATION

Examine metallized surface under 10X magnification. There shall be no evidence of flaking or peeling of the metallization. Failure of metal to adhere infers contamination of wafer surface prior to or during evaporation. NOTE: A somewhat more rigorous test is performed by using a degreased new razor blade or sharp metal probe to inscribe a cross-hatch pattern in metalization and then proceeding as in para. 3.3.

5. METHOD DOCUMENTATION

The following shall be supplied in the detail specification:

1. Procedural instruction

2. Sample constants
   (a) cleaning procedure performed
   (b) aluminum thickness
   (c) chamber pressure
Method 6071B

Metallization Adhesion

(Pressure-Sensitive Tape)

1. **Purpose** This method may be used to evaluate adhesion of the metallization film to the substrate. It utilizes a pressure-sensitive tape to remove metal with poor adhesion followed by a microscopic examination.

2. **Apparatus**
   2.1 Metallurgical microscope
       Bausch and Lomb dynoptic No. 31-20-68-38
       (or equivalent)
   2.2 Hot plate, 6" x 6", electric
       Corning, Pyroceram PC-35
       (or equivalent)

3. **Materials**
   3.1 Apiezon wax
   3.2 Slides, glass microscope, 1" x 3"
   3.3 Pressure-sensitive tape, Scotch, 3/4", No. 810

4. **Procedure**
   4.1 **Preparation**
       4.1.1 Process a polished, passivated substrate or a clean glass slide through the metallization operation to be evaluated.
       
       **Note:** For a slide, omit 4.1.2 through 4.1.5

       4.1.2 Place a glass slide on a hot plate set a temperature slightly higher than the melting point of apiezon wax (150-160°C).

       4.1.3 Add a small quantity of wax to the slide.
4.1.4 Place the sample upon the slide, metallization up, and seat it in the wax.

4.1.5 Remove the slide from the hot plate and allow it to cool.

4.1.6 Apply one end of a four (4) inch length of pressure-sensitive tape to the metallized surface. Insure contact across the maximum sample dimension with no voids or bubbles.

4.1.7 Hold the slide firmly against a solid surface and grasp the free end of the tape.

4.1.8 Apply a force normal to the substrate surface and smoothly strip the tape from the metallization.

4.1.9 The tape may be applied to a clean slide for preservation.

4.2 Inspection

4.2.1 Place the sample on the stage of a metallurgical microscope.

4.2.2 At 100x, scan the metallized surface for any voids or missing metal.

5. Calibration

Not applicable.

6. Accuracy

This method should detect areas where metal is removed which are approximately 0.0005 square inches.

7. Test Frequency

7.1 Evaluation of glass slides should be done for each evaporation.

7.2 Substrate testing should be on a periodic basis.
1.0 DISCUSSION

Aluminum is generally used to form the metalization patterns in monolithic microcircuit technology. The selection of this element is based on:

1. Adequate electrical conductivity;
2. Relatively low eutectic temperature (576°C) with silicon at which stable ohmic contacts are readily formed on p or highly doped N type Silicon.
3. Reacts with SiO₂ sufficiently to provide an adherent bond. Such reaction is also sufficient at alloying temperature to achieve penetration of the angstroms of oxide which form over the exposed silicon contact areas prior to evaporation. This is necessary if low ohmic contact is to be achieved.

The extensive use of aluminum in thin films of increasingly small dimension however, has emphasized several conditional limitations to its stability in the microcircuit application. These are:

2. Electromigration

The methods for defining the conditions which must be maintained to prevent device destruction through the phenomena are treated separately as method A and B.
1. DISCUSSION

Excess silicon atoms, which exist in the Aluminum during the alloying step, must recrystallize upon reduction of temperature. Characteristically, the atoms are precipitated preferentially by the aluminum film at the grain boundaries. Since the typical monolithic device has aluminum paths on the order of 1/4 to 1 mil width and perhaps 10,000 Å thick, it is obvious that there is a relatively infinite source of silicon which in the limit could result in the total consumption of aluminum near the contact cut areas. The supply of silicon is further enhanced by the interaction between the metal film and the silicon dioxide. Sustained exposure, then, of the metalized device to temperatures which allow migration of silicon combine to enrich the silicon concentration in the grain boundaries to the detriment of the conductivity of metalization paths. Particularly sensitive regions of the monolithic device are the oxide step-downs, which are typically close to open contact cuts and hence to the silicon source.

The formation of grain boundaries is inherent to the metalization system (Al-SiO₂-Si). The method applied to control this form of metalization instability must define the extent, rather than the existence, of the phenomenon in a given process. The method described is a straightforward microscopic examination of the metallization after all thermal processes are completed. In addition to this initial evaluation, the long-term stability of the metalization must be verified by operation and storage life testing at maximum rated temperature and dissipation. And at selected levels beyond these ratings, Figure 2 shows effects of grain boundary formation on a device after...
9000 hours operating life at 25°C and 1.8 times rated dissipation.

2. APPARATUS

Metallurgical Microscope – Bausch and Lomb or equivalent.

3. PROCEDURE

3.1 Sample – The sample shall be selected from completed devices which have completed all thermal process and screens. Process history must be available and include aluminum metalization thickness, alloy temp. and time, bonding temp. and time (when applicable), sealing temp. and time and details of any other thermal process or screens which are normally applied.

3.2 Preparation – The sample packages will be opened in the prescribed manner for the package type.

3.3 Measurement – The sample devices will be examined at magnification levels which allow detailed observation of metalization surface texture and detail.

3.4 Evaluation – Visible evidence of grain boundary development to the extent shown in Figure 1 infers questionable basic process or loss of control of process.

4. METHOD DOCUMENTATION

The following information must be supplied in the detail specification:

1. Inspection Procedure

2. Process Constants – aluminum thickness
   alloying temperature
   bonding conditions

3. Acceptance Criteria
FIG. 1 Marginal Grain Boundary Development

FIG. 2 Catastrophic Condition Due to Extreme Grain Boundary Development
II. ELECTROMIGRATION

1.0 DISCUSSION

The phenomenon of electromigration is due to the mass transport of aluminum ions from one point in the conductor to another. The result is the creation of voids toward the cathode end of the conductor and accumulation toward the anode side. Most investigators attribute this transport of aluminum atoms (ions) to a momentum exchange between the electrons and aluminum atoms under condition of high current density and attendant high temperature. The critical current density is generally reported to be of the order $10^6$ amperes/cm$^2$ and above. Time to failure thereafter is a function of temperature. The voids in the conductor metal which occur due to electromigration appear to propagate along grain boundaries on the increasing temperature gradient. In the limit the voids interconnect along grain boundaries to the point of severance of the conductor. The electromigration effect is self enhancing since each incremental reduction of conductor cross section results in increased current density with attendant temperature increase.

The maximum current density and temperature which would be compatible with a very long life ($\gg 1000$ hours) has not been precisely determined. From the curves presented by Blech and Sello (Physics of Failure in Electronics Vol. 5 pp 496-505) it might be estimated that a current density of $5 \times 10^5$ could be sustained at a maximum conductor temperature of 150°C although these authors specifically decline the generality of these curves for extrapolation.

Determination of the threshold of electromigration will depend largely on the geometry of a given device. The method described is a basic procedure which generally outlines an experimental approach.
Figure 1 relates conductor thickness at various conductor widths to the current levels at which the critical current density of $10^6$ amperes/cm$^2$ is reached. Figure 2 shows the effects of electromigration.

2.0 APPARATUS

The following apparatus is typically suitable to electromigration studies. This method assumes suitable and adequate facilities for test device fabrication.


Digital Voltmeter - Hewlett-Packard Model 3439A

Milliammeter (0-500 mw) - Weston

3.0 PROCEDURE

The following procedure may be followed to observe the electromigration effect. The test pattern may be prepared in a manner which allows microscopic observation during test (short term) or suitably encapsulated. The encapsulated condition more nearly approximates the device environment and is more conveniently implemented for long term testing. Actual devices may be substituted for test patterns.

Sample - A test pattern which represents the worst case metalization cross section for a specific device geometry shall be fabricated. Pattern shall be deposited on an oxidized substrate which is identical in thickness and chemistry to that of the specific device. Length, width and thickness of test metal shall be determined to be within 10% of specified values.

Preparation - Pattern shall be deposited on an oxidized silicon substrate which is identical to that used for device fabrication. Length width and thickness of metal shall
be within 10% of specified values and shall be recorded. Measurement - Electrical connection to the test pattern shall be of the 4 probe Kelvin arrangement. Test patterns shall be grouped and stressed at various levels of current density and temperature. A minimum evaluation would typically involve:

Test Group 1 rated current max. rated temp. (ambient)
Test Group 2 $10^6$ amp/cm$^2$ max. rated temp (ambient)
Test Group 3 $10^6$ amp/cm$^2$ 25 to 50% beyond max rated temperatures (ambient)

Duration of test - 1000 hour minimum unless otherwise specified.

Simple observation of electromigration effect may be accomplished by subjecting exposed test samples to the necessary current density on a microscope stage. Electrical connection is provided either by four probe arrangement. Electromigration at a given current density may be accelerated by increasing the sample temperature.

Evaluation - Failure to observe electromigration under rated condition infers adequacy of metal geometry. Such information shall be supplemented by extended life on the product for periods commensurate with application.

4.0 METHOD DOCUMENTATION

The following documentation is applicable to the investigation of electromigration effects:

1. Procedural Instructions
2. Test specification defining
   a. Number devices to be tested
   b. Test parameters - current temperature
c. Duration of Test

d. Equipment required

3. Recording format.
CURRENT LEVELS REQUIRED THROUGH ALUMINUM METALIZATION OF VARIOUS WIDTHS AND THICKNESSES TO OBTAIN A CURRENT DENSITY OF $10^6$ AMPS/CM$^2$.

**Figure 1**

- 2 MIL WIDE METAL
- 0.5 MIL WIDE METAL
- 0.25 MIL WIDE METAL
- 0.20 MIL WIDE METAL
- 0.1 MIL WIDE METAL

CURRENT THROUGH METALIZATION

ALUMINUM METALIZATION THICKNESS ANGSTROMS

0 2000 4000 6000 8000 10,000
FIG 2 - The effect of electromigration on a monolithic device. Note voids formed in conductor indicated by arrow #1 and accumulation at emitter fingers. Same conditions observable at collector metal and Vcc conductor. Current density calculated slightly greater than $10^6$ amperes/cm$^2$. Device temperature approximately 130°.
Method 6072 B

Stability of Metallization

1. **Purpose**
   This method may be used to evaluate the stability of circuit metallization. It utilizes a high temperature storage test to detect formation of intermetallic compounds and a high temperature D-C bias test to observe "Electro-Migration". The test device is manufactured as process control devices are fabricated on a standard integrated circuit wafer.

2. **Apparatus**
   2.1 High Temperature Storage Oven
       300°C air ambient
       (any source)
   
   2.2 High Temperature D-C Bias Chamber
       200°C, \( I_F = 100\text{mA} \) per device
       (any source)
   
   2.3 Curve Tracer
       Fairchild 6200
       (or equivalent)

3. **Materials**
   None
4. **Procedure**

4.1 **Test Device Manufacture**

4.1.1 Fabricate one or more test devices on each standard production wafer. See Figure 1.

4.1.2 After dicing, separate test devices from production.

4.1.3 Process through standard device assembly.

4.1.4 Electrically screen test devices for continuity.

4.2 **Intermetallic Formation (Gold-Aluminum)**

4.2.1 Store this group of test devices at 300°C for approximately 200 hours.

4.2.2 Cool to room temperature.

4.2.3 Monitor conductance at 10 - 20mA. Full scale. Any slope greater than 20Ω will indicate intermetallic formation.

4.3 **Electro-Migration**

4.3.1 Place devices in chamber at 200°C under D-C bias which forces a current density of about $10^6$ amperes/cm$^2$ through each sample.

4.3.2 Store for approximately 200 hours.

4.3.3 Test for continuity at 10mA. Opens will be failures.

5. **Accuracy**

Not applicable. Current density for 4.3 should be set to deliver 10 - 20% open circuits.

6. **Calibration**

Not applicable.
7. **Test Frequency**

This test should be performed periodically.
Figure 1. Metallization Test Device

For Current Density ~ $10^6$ Amperes/$\text{cm}^2$

Metal Thickness = 10,000 Å

Test Section Width = 0.0004 inch

Current (D.C.) = 100mA
1.0 DISCUSSION

The ohmic contact between the metalization and the active silicon areas is accomplished at or near the eutectic temperature (576°C) of the Al-Si system. Characteristically the contact thus formed is stable and ohmic on P type or heavily doped N type silicon. (The N type material must be heavily doped at the contact interface to prevent formation of a rectifying junction since aluminum is a P type dopant).

The factors which affect ohmic contact quality are process oriented.

1. Cleanliness of Contact Cuts at the time of metalization
2. Cleanliness of evaporation system
3. Uniformity of oxide removal from contact cuts
4. Alloying temperature
5. Metalization thickness

The method described for the determination of the quality of the ohmic contact requires the inclusion of a suitable test pattern. Specific test patterns must be based on the geometrical and diffused layer characteristics of the specific monolithic device. Figure 1 demonstrates one approach to the general problem of in process determination of contact resistance quality.

2.0 APPARATUS

The following apparatus is used to evaluate the quality of ohmic contact between the metalization and silicon:

Four Probe Assembly - Philco
Microscope - Bausch and Lomb
        Stereo Zoomar (Mag. ≤ 10X-60X)
Digital Voltmeter - Hewlett-Packard
        Model 3439A
Milliammeter

Microscope - Bausch and Lomb
   Metallographic Type
   Vertical Illuminator
   Movable Stage
   Magnification Range
   Objection 10X, 20X, 40X
   Eyepieces 10X and 20X

3.0 PROCEDURE

The following procedure is applicable to the evaluation of ohmic contacts. The test pattern of Figure 1 demonstrates the principle used for contact resistance determinations.

Sample - Metalized and alloyed wafers containing the specified number of test patterns. Test pattern principle shall be similar to that of Figure 1.

Preparation - Test pattern diffusion sequence:
1. P test areas and P background shall be deposited and diffused with isolation.
2. P test areas and P background shall be reopened and enriched at base deposition and diffusion.
3. N+ test areas shall be opened in the P background at the time of emitter cut. Deposition and diffusion shall concurrent with emitter.
4. Contact cuts shall and metalization shall be performed on the entire wafer including test patterns.

Measurement - The resistance of the diffused test areas is essentially constant and very low so that the major resistance component will be a summation of the individual contact resistances. Measurement is accomplished.

-2-
by contacting the metal lands of either the P test strip or the N+ test strip. The four probe Kelvin contact system will be used. A specified test current shall be established through the series of contacts and the voltage drop recorded.

Evaluation - Resistance of the series connected contacts shall be calculated from the voltage and current readings. Resistance values obtained will be compared with the norms for the particular geometry.

4.0 METHOD DOCUMENTATION

The following documentation is applicable to the performance of contact resistance measurements.

1. Procedural instruction
2. Test limits applicable to specific geometry
3. Recording format
Contact Resistance
Test Pattern Dice
Locations on Wafer

N Type Background
P Test areas (Isol. diff and base diff.)

Metal
Test Cuts

Diffused P Background

N+ Test areas (normal emitter diff.)

Figure 1
Test Pattern Configuration for
Determination of Contact Resistance
Between Al to N+ type Silicon and
Al to P type Silicon
Quality of Ohmic Contact

1. **Purpose**
The purpose of this test is to determine that an ohmic contact exists between aluminum interconnections and the diffused structure. It employs evaluation of a series of diffused resistors.

2. **Apparatus**
   2.1 Transistor Curve Tracer
       Fairchild 6200
       Tektronix 575
       (or equivalent)
   2.2 Two-point Probe with micro-manipulator
       (any source)

3. **Materials**
   3.1 Silicon wafer with diffused and metallized test pattern.
       Figure 1.

4. **Procedure**
   4.1 Obtain wafer with test pattern after metallization alloy.
   4.2 Place wafer upon the probe stage. Figure 2.
   4.3 Probe one pad with the emitter probe and one with the collector probe.
   4.4 The oscilloscope trace should be a straight line through the origin with a finite slope. Figure 3.
   4.5 A vertical line (short) indicates excessive alloying and a horizontal line indicates either a rectifying contact or an open.
5. **Calibration**
   
   5.1 Calibrate the curve tracer per manufacturer's instructions.
   
   5.2 A typical trace should be obtained by placing a 1-10 KΩ resistor across the probes.

6. **Accuracy**
   
   Adjustment of current and voltage scales to deliver a 45° slope will provide maximum accuracy.

7. **Test Frequency**
   
   Each wafer should be sampled at final wafer test.
Figure 1. Test Pattern
Figure 2. Resistor Check

F.S.C. 2 POINT PROBE

F.S.C. 6200

D.U.T.
Figure 3. Oscilloscope Trace
1.0 DISCUSSION

The cross sectioned uniformity of monolithic microcircuit metalization is primarily affected by the surface topography of the chip. The sequence of photolithographic steps and oxidation steps through which the monolithic structures are formed result in differences of elevation between adjacent areas of 3000 to 6000 Å. These oxide step downs are most abrupt in the area of the contact cuts. Since the surfaces of these step downs is not normal to the aluminum sources during evaporation it follows that less aluminum will be deposited thereon. The measurable thickness on these surfaces normal to the source must be adjusted to insure adequate thickness on the step downs. Cross section studies have shown that contact cut edges have a slope due to lateral etching that approaches 45° and that the thickness of the evaporated deposit thereon is about 70% of thickness of the deposit on the areas of the wafer normal to the source. Further assurance of adequate conductivity at contact cuts is obtained by layout of the metalization pattern such that the entire periphery of a contact cut including the step down is covered by metal.

Voids in the metalization are generally the result of photolithographic defects and are subject to specific inspection criteria. In general voids or scratches which significantly reduce the cross section of the metal conductor are not acceptable. The criteria for inspection of scratches and voids must take into account the amount by which the reduced conductor cross section will increase current density in order that the threshold of electromigration (Method 29B) will not be approached.

2.0 APPARATUS AND PROCEDURES

The cross sectional uniformity of metalization is not directly
measurable. Control in this area is based on specific inspection criteria under conditions of appropriate magnification and lighting.

3.0 METHOD DOCUMENTATION

The following documents and aids are required for inspection of microcircuit metalization:

1. Inspection criteria defining acceptance limits for:
   1. Metalization coverage of contact cuts
   2. Metalization voids
   3. Scratches or other forms of damage to metalization

2. Photographic examples of rejectable conditions.
1. **Purpose**
The purpose of this test method is to evaluate the uniformity of cross section of the metallized interconnections and bonding pads.

2. **Apparatus**
2.1 Metallurgical Microscope
Bausch and Lomb 31-20-68-38 (DMETR)
(or equivalent)

3. **Materials**
3.1 Glass microscope slides, 1" x 3".

4. **Procedure**
4.1 Place the alloyed, metallized wafer upon a clean glass slide on the microscope stage.

4.2 Adjust the magnification to 150x and scan the wafer for the following defects:

4.2.1 **Geometry** - Adherence to design dimensions and shape.

4.2.2 **Scratches** - Scratches which reduce the designed cross-sectional area.

NOTE: A scratch with oxide, substrate, etc., showing through will usually be deeper and more severe than one with metal covering the bottom of the damaged area. Figure 1.

4.2.3 **Alignment** - Registration of metallized interconnections with the passivating layer cut-outs. Figure 2.
4.2.4 **Undercutting** - Failure of the metallization-photoresist interface resulting in the width at the interconnection top being significantly narrower than the width in contact with the passivated substrate. Figure 3.

4.2.5 **Voids** - Holes in the metallization film which significantly reduce the cross-sectional area either by their individual size or density.

4.2.6 **Cut-Out Continuity** - Cracking, narrowing or discernible thinning of interconnections at the passivating layer cut-out. Figure 4.

5. **Calibration**
   
   Not applicable.

6. **Accuracy**
   
   6.1 Scratches and cracks greater than 1 micron.

   6.2 Voids greater than 2 microns.

   6.3 Geometry alignment and undercutting within designed tolerance.

7. **Test Frequency**
   
   Every wafer should be sampled prior to the dicing operation and all circuits should be inspected.
Figure 1. Interconnection Scratches

Figure 2. Alignment
Figure 3. Interconnection Undercutting

NORMAL

UNDERCUT
Figure 4. Cut Out Continuity