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QUANTITATIVE ANALYSIS OF DEFECTS IN
SIL.7CON

Silicon Sheet Growth Development for the Large
Area Silicon Sheet. Task of the Low-Cost Solar Array Project

FINAL REPORT
by
R. Natesh
J.M. Smith
T. Bruce
H. A. Qidwai

7
April 1920

JPL Contract No. 954977

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## QUANTITATIVE ANALYSIS OF DEFECTS IN SILICON

Silicon Sheet Growth Development for the Large Area Silicon Sheet Task of the Low-Cost Solar Array Project

FINAL REPORT
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April 1980

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The JPL Low-Cost Silicon Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Fhotovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, by agreement between NASA and DOE.

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## CONTENTS

;
SECTION ..... Page
" LIST OF FIGURES ..... 4
LIST OF TABLES ..... 7
I SUMMARY ..... 8
II INTRODUCTION ..... 10
III TECHNICAL DISCUSSION ..... 12
IV RESULTS ..... 28
v CONCLUSIONS ..... 34
a VI REFERENCES ..... 35-

## LIST OF FIGURES

|  | Figure No. |
| :---: | :---: |
|  | 1 |
| $\cdots$ |  |
|  | 2 |
|  | 3 |
| 1 |  |
|  | 4 |
| 6 | 5 |
| \% | 6 |
|  | 7 |
| 4 | 8 |
|  | 9 |
|  | 10 |
| k | 11 |
|  | 12 |
| 1 |  |

Figure Title Page
IBM \#1 - Section 1 - Area 1, micrograph ..... 36of silicon ribbon surface showing inter-section of three grains after cleaningorganic materials from surface. 200X
IBM \#1 - Section 1 - Area 2, micrograph ..... 36 of ribbon surface showing grain boundaries after cleaning organic materials from surface of ribbon. 200X
IBM \#1 - Section 1 - Area 1, micrograpli ..... 37of ribbon surface after oxide removal. 200X
IBM \#1 - Section 1 - Area 2, micrograph ..... 37
of ribbon surface, after oxide removal. 200k
IBM \#1 - Section 1 - Area 1, micrograph ..... 38of ribbon surface, after chemicalpolishing. 200X

IBM \#1 - Section 1 - Area 2, micrograph 38 of ribbon surface after chemical polishing. 200X

IBM \#6 - Section 1 - Side 1, micrograph 39 of ribbon surface after chemical polishing. 200X
IBM ${ }^{\#} 6$ - Section 1 - Side 1 , micrograph
of ribbon surface after a 15 second
etch by Etching Solution I. 200 X
IBM \#6 - Section 1 - Side 1, micrograph ..... 40 of ribbon surface after a 15 second etch by Etching Solution I. 200xIBM \#6 - Section 1 - Side 1 , micrograph 40of ribbon surface, after a 30 secondetch by Etching Solution I. 200xIBM \#6 - Section 1 - Side 2, micrograph41of ribbon surface after chemicalpolishing. 200XIBM \#6 - Section 1 - Side 2, micrograph41of ribbon surface after a 30 second etchby Etching Solution II. 200X

## LIST OF FIGURES (Continued)

Figure No. Figure Title Page
13 IBM \#6 - Section 1 - Side 2, micrograph ..... 42 of ribbon surface after a 60 secondetron by Etching Solution II. 200X
14 IBM \#6 - Section 1 - Side 2, micrograph ..... 42of ribbon surface after a 90 secondetch by Etching Solution II. 200X
15 IBM \#6 - Section 3-Side 2 - Area 1, ..... 43micrograph of ribbon surface afterchemical polishing. 200X
16 IBM \#6 - Section 3 - Side 2 ..... $43 \& 44$micrograph of ribbon surface after a60 second etch by Etching Solution III. 200X
17 Flow Chart of BASIC Program for QTM ..... 45Operation and Data Reduction.
$18 \mathrm{~A}, \mathrm{~B}, \mathrm{C}$ Photographs illustrating manual Image ..... $46 \& 47$ Editing in the "ACCEPT" mode on the QTM 720. 800X
19 A,B,C Photographs illustrating manual Image Editing in the "PROJECT" mode on the47 \& 48QTM 720. 800X
$20 \mathrm{~A}, \mathrm{~B}$ Photographs illustrating manual Image ..... 49Editing in "CUT" mode on the QTM 720. 800x
21 Graphical plot showing systematic ..... 50 variation in twin density with respectto specimen position in IBM RibbonNo. 4-457.
22 Histogram of grain boundary length ..... 51 of Mobil Tyco samples
Histogram of twin boundary density of ..... 5223Mobil Tyco samples.
24 Histogram of dislocation pit density ..... 53 of Mobil Tyco samples.
25 Histogram of grain boundary length ..... 54 of Motorola samples.

## LIST OF FIGURES (Continued)

Figure No. Figure Title Page
26 Histogram of twin boundary density of ..... 55 Motorola samples.
27
Histogram of dislocation pit density ..... 56 of Motorola samples.
28
Diagram showing the position of Mobil Tyco ..... 57samples MRI \#19-30 as cut from ribbon 5-685.
29
Diagram showing the position of Mobil Tyco ..... 58 samples MRI \#31-46 as cut from ribbons5-742 and 5-744.Diagram showing the position of Mobil Tyco59samples MRI \#47-77 as cut from ribbons$5-745,5-743,5-640$ \#1 and 5-640 \#2.
Diagram showing the position of Mobil Tyco ..... 60 samples MRI \#78-90 as cut from ribbon 5-867.Diagram showing the position of Mobil Tycosamples MRI \#91-98 as cut from ribbon 5-640.Diagram showing the position of Mobil Tyco62samples MRI \#99-105 as cut from ribbon 5-990.
34
Diagram showing the position of Mobil Tyco ..... 63 samples MRI \#106-134 a.s cut from ribbons184-88, 184-175, 5-1094-33, and 5-1094-69.3536Diagram showing the position of Motorola64samples MRI \#1-32 as cut from ribbons6-792, 6-837, 6-656, and 6-840.
Diagram showing the position of IBM samples ..... 65MRI \#1-7 as cut from ribbon 4-457.

## LIST OF TABLES

Table No. Title Page
1 Chemical polishing times of Wacker samples. ..... 66
2 Chemical polishing times of IBM samples. ..... 67
3 Chemical polishing times of Motorola samples. ..... 68
4 Chemical polishing times of Mobil Tyco samples. ..... 69
5-20 Data from QTM analysis of Mobil Tyco samples ..... 70-85MRI \#1-134.
21 Data from QTM analysis of IBM samples ..... 86 MRI \#1-7.
22-25 Data from QTM analysis of Motorola samples ..... 87-90MRI \#1-32.
26 Listing of BASIC program "Defects in ..... 91 Silicon $3^{\prime \prime}$.
QTM data on Wacker sample. ..... 94
QTM printout of data on Mobil Tyco sample ..... 95
28 MRI \#100.

The analyses of one hundred and seventy four (174) silicon sheet samples, about 1200 square centimeters, for twin boundary density, dislocation pit density, and grain boundary length has been accomplished. One hundred and thirty three (133) of these samples were manufactured by Mobil Tyco, thirty two (32) by Motorola, seven (7) by IBM, one (1) by Honeywell, and one (1) by Wacker.

Procedures have been developed for the quantitative analysis of the twin boundary and dislocation pit densities using a QTM-720 Quantitative Image Analyzing System. The QTM-720 system has been upgraded with the addition of a PDP 11/03 mini-computer with dual floppy disc drive, a Digital Equipment Writer (III) high speed printer, and a Field-Image Feature Interface Module (F.I.F.I.). These changes have greatly enhanced the speed and reliability of the QTM-720 System as well as improving the data storage and printout capability.

Three versions of a computer program that controls the data acquisition and analysis on the QTM-720 have been written.

Procedures for the chemical polishing and etching of Mobil Tyco, Motorola, IBM, \&Hacker samples have been developed.

This report describes the complete procedures for the defect analysis of silicon samples using a QTM-720 Image Analyzing System, and includes chemical polishing, etching, and QTM operation. The data from one hundred and seventyfour (174) samples, and a discussion of the data is also included herein.

In addition to the above work, comparisons of the capabilities of a variety of powerful analytical techniques in analyzing impurities from four different silicon matrix was performed. The silicon matrix analyzed were Mobil Tyco (EFG-RH and EFG-RF). Honeywell (SOC), and Motorola (RTR). The techniques used were: Neutron Activation Analysis, Spark Source Mass speetrometry, Ion Scanning Spectrometry, Secondary Ion Mass Spectrometry, Scanning Auger Microanalysis, Electron Spectroscopy for Chemical Analysis, Ion Microprobe Mass Spectroscopy,
and Optical Microscopy. The results showed significant differences in the capability of the various analytical techniques fur analyzing silicon impurities and, in addition, provided important information regarding the type and distribution of impurities present in the various silicon matrix. The details of this work is presented in a separate report (MRI-267) to JPL.

## SECTION II

## IMTRODUCTION

The main objective of this program was to develop maging techniques to subsequently allow rapid, reproducible, and accurate evaluation of silicon sheet defect structure. Secondly, defect data accumulated for many samples would allow for potential cross correlation between structires revealed and specific sheet fabrication technique and/or efficiency. Structural defects that were quantified included grain and twin boundarles, precipitates, and dislocations, Quantitative characterization of these structural defects, which have been revealed by etching the surface of silicon samples, can then be performed using a Quantimet 720 Imaşe Aneilyzer.

The silicon sheet samples wert orlginally obtained by JPL from different manufacturers. Each of these manufactureers use their own crysta? growth and fabrication techniques and, therefore, the various types of silicon produced contain a variety of trace impurity elements and structural defects. The most important criteria in evaluating the various silicon types for terrestrial solar cell applications are: (i) cost, and (ii) conversion efficiency. At present, the solar cells with highest conversion efficiency are made of high purity silicon single crystals, which are free from structural defects such as dislocations, twin boundaries, precipitate particles, etc. But these crystals and subsequent processing are very expensive and may not meet the DOE goal of 50 cent/watt by 1986. On the other hand, silicon crystals such as Edge-defined Film-fed Growth (EFG) ribbons, Silicon on Ceramic (SOC), Wacker, etc, are NOT single crystals; but made of highly ordered crystals which contain large and differing numbers of dislocations, twin boundaries, grain boundaries, and precipitates compared to the premium grade or Czochralski grown silicon.

The following important questions must be answered to evaluate low and high cost silicon sheet: (i) What effect do these defects have on conversion efficiency? (ii) Df the various types of defects, which defect/defects severely affects conversion efficiency? (iii) At what concentrations does this effect become significant? (iv) Is there a rapid, accurate, quantitative method that can be used routinely as a Quality Assurance tool?

Quantitative analysis of surface defects was developed and is being performed by using a Quantimet 720 Quantitative Image Analyzer. This system can differentiate and count 67 shades of grey levels between black and white contrasts. In addition, it can characterize structural defects by measuring their lenyth, perimeter, area, density, spatial distribution, frequency distribution (in any preselected direction), and is programmable in these measurements. However, the Quantitative Image Analyzer is extremely sensitive to optical contrasts of various defects. Therefore, to obtain reproducible results, the contrasts produced by various defects must be similar and uniform for each defect types along the entire surface area of samples to be analyzed. To achieve this, a chenical cleaning and polishing technique has now been perfected for silicon samples from Mobil Tyco, Wacker, Motorola, and IBM. The cleaning and polishing preparation technique produces a very clean and even surface for silicon crystals suitable for analyses by the QTM 720 Image Analyzer. We have now obtained quantitative information from a variety of silicon crystals.

## SECTION III

## TECHNICAL DISCUSSION

As mentioned in the introduction, it has been found necessary to chemically polish silicon samples before analyzing them with QTM. The chemical polishing procedures are discussed below:

CHEMICAL POLISHING
The first step in the chemical polishing process is to clean the surfaces of the silicon crystals. This is achieved by rubbing the surfaces with swabs soaked in trichloroethylene. This process removes most of the organics from silicon surfaces. However, to remove remaining residues and water spots, an acetone rinse followed by ethyl alcohol rinse are required. Silicon surfaces are then dried by blowing nitrogen or freon gas over them. Figures 1 and 2 show the silicon surfaces after cleaning. All optical micrographs were taken in a Baush \& Lomb metallograph.

An acid resistant protective coating is applied to one surface of the silicon sheet sample in order to prevent it from being polished. This allows MRI to complete the etching and defect analyses \& then send the silicon samples back to JPL. JPL may then remove the protective coating from the unpolished surface, and process the sample into a solar cell and measure its conversion efficiency. This will allow JPL to determine the effects, if any, of the density and type of structural defects to conversion efficiency. Since both these data are obtained on the same silicon sample, the results obtained will be of signif. icant value in determining the effects, if any, of structural defects on the performance of solar cells.

Of the various coating materials studied, Aplezon Wax (W) gave best results, This is resistant to many acids at $80^{\circ} \mathrm{C}$ for at least 120 seconds. A solution is prepared by dissolving a very small amount of Apiezon Wax (W) in trichloroethylene. This solution is sprayed by air brush or applied by a fine paint brush to one of
the silicon crystal surfaces. The surface is then baked for $10 \pm 1.0$ mirutes at $125^{\circ} \mathrm{C} \pm 10^{\circ} \mathrm{C}$. Baking is necessary to evaporate the trichloroethylene and allow the wax to flow uniformly on the surface.

In order to start with a uniform surface for acid polishing, any $\mathrm{SiO}_{2}$ coating on the silicon sample surface nust be removed. This is done by immersing the sample in concentrated HF for 2 minutes at room temperature. The sample is rinsed in deionized water, and ethyl alcohol respectively. Freon gas is used to dry the sample surface. Figures ? and 4 show the silicon surfaces fter removal of the $\mathrm{SiO}_{2}$ layer. Only a few angstroms thick layer of $\mathrm{SiO}_{2}$ is covering the surface of silicon samples, therefore, the removal of this $\mathrm{SiO}_{2}$ layer does not significantly alter the microstructure as mas be seen by comparing figs. 1 and 3; and Figs. 2 and 4.

The most suitable polishing solution for silicon surfaces is a mixture of $70 \% \mathrm{NNO}_{3}: 49 \% \mathrm{HF}: 99.9 \% \mathrm{CH}_{3} \mathrm{COOH}$ in 1:2:3 ratio by volume. All acids used were Electronic Grade, Low Sodium MOS qualiry. The polishing solution is heated to $50^{\circ} \mathrm{C} \pm 3^{\circ} \mathrm{C}$ in a teflon beaker on a hot plate. The silicon sample is then immersed in this solution. It has been found that silicon samples from different manufacturers require varying polishing times. The polishing times required for Mobil Tyco, Motorola, IBM, and Wacker samples are summarized in Tables 1 to 4.

The polished samples is then rinsed in deionized distilled water for 5 minutes, followed by rinsing in ethyl alcohol. It is then dried by blowing freon gas on the surface.

It may be noted that samples which are slightly underpolished as well as samples which are well-polished, exhibit bright and shiny surfaces when observed by the naked eye. Therefore, visual observation can not be used to determine the quality of polishing. However, when the samples are observed at high magnifications ( $800 \times$ or greater) in a high quality optical metallograph, the underpolished samples show growth lines and overpolished samples show faceting and sub-grain type structure, whereas the well-polished samples show clearly
defined grain boundaries and some of the twin boundaries in sharp contrast. Therefore, an optical metallograph must be used to determine the quality of polishing.

Figures 5 and 6 show the polished surfaces of silicon samples.
After the silicon samples are chemically polished, they are etched to reveal structural defects.

## CHEMICAL ETCHING:

The etching solution that has been developed is a dilute variation of the Sirtl etch. Composition of the Sirtl etch is as follows:

Solution $A$
$50 \mathrm{~g} \mathrm{CrO}_{3}: 100 \mathrm{ml}$ deionized water

Solution B
49\% HF, electronic grade Solution $B$ equal in volume to Solution A

Three dilute variations were prepared from the Sirtl etch. The results obtained by using each of these three etchants are discussed below:

## ETCHING SOLUTION I:

The first variation from the Sirtl etch was prepared by dissolving 20 grams of $\mathrm{CrO}_{3}$ in 60 ml of deionized distilled water, and then adding an equal volume of concentrated HF. A 15 second etch by this first etching solution revealed dislocations, twin boundaries, and grain boundaries. The resolution of the defects are limited only by the optical equipment used.

Figure 7 shows the structure of an IBM silicon ribbon after chemical polishing. Figures 8 and 9 are photomicrographs after a 15 second etch.

The variation in contrast between different boundaries may be indicative of different energies associated with different types of boundaries. Grain boundaries and twin boundaries have different energies, which may affect their etching rates.

An additional 15 seconds etch by the Etching Solution 1 revealed a higher number of defects and less contrast variation between different twin boundaries (Figure 10).

## ETCHING SOLUTION II

The second variation from the Sirtl etch was prepared by dissolving 10 grams of $\mathrm{CrO}_{3}$ in 40 ml of deionized water, and adding an equal volume of concentrated HF .

Figure 11 is a phitomicrograph of the chemically polished surface. Figure 12 is a photomicrograph of the same surface after 30 seconds etch by Etching Solution II. Figure 12 shows all dislocations, twin boundaries, and grain boundaries present in the sample. Variations in contrast of dislocations is, however, due to focusing on a slightly curved surface.

The silicon surface in Figure 12 was etched for an additional 30 seconds. This resulted in deeper etching of dislocations and overlapping of twin boundaries (Figure 13). An additional 30 seconds etch (i.e., a total of 90 seconds) on the same surface resulted in significant overlapping of dislocations and twin boundaries (Figure 14).

## ETCHING SOLUTION III

The third variation from the Sirtl etch comprises 10 grams of $\mathrm{CrO}_{3}$ in 60 ml of deionized distilled water; and an equal volume of concentrated HF.

Figure 15 is a photomicrograph of a chemically polished silicon surface. Figure 16 is a photomicrograph of the same area after 60 seconds etch by Etching Solution III.

The etching treatment by Etching solution III resulted in an optical resolution of $10^{-4} \mathrm{~cm}$ for twin boundaries and an optical density resolution of $10^{7}$ dislocations per $\mathrm{cm}^{2}$ at magnifications of $800 x$ and above. A higher resolution, however, can be achieved if a higher magnification is used for observation.

It has been observed on many : son surfaces that an optimum etching time of approximately 50 seconds by Etching Solution III is sufficient to distinctly reveal grain boundaries, twin boundaries, and dislocations. Etching Solution III has been used to etch Mobil Tyco, Motorola, IBM, Wacker, and Honeywell samples.

High quality defect structures without overlapping and without wide variations in contrast of each defect type were always obtained. USE OF THE QTM 720-PDP 11/03 SYSTEM FOR IMAGE ANALYSIS:

During the months of March and April, 1979, changes were made to the QTM system to allow for more efficient daca storage and analysis capabilities.

Before these changes, the QTM 720 was ru, in a semi-automated fashion ${ }^{1}$, making use of a Hewlett-Packard Model 9810 programmable calculator interfaced to the system by means of a special QTM module, the Field Data Interface. In addition, the data output was printed on a conventional teletype. In the present configuration, a PDP $11 / 03$ with a Digital Equipment Corporation Writer (III) and a RXO1 dual floppy disc drive is interfaced to the QTM-720. Two special OTM modules are used for the interfacing: a Field-Image-Feature Interface (FIFI) and a Control Interface (CI).

The FIFI links the QTM 720 to the PDP 11/03 computer allowing high speed data transfer from the QTM directly into the memory of the PDP $11 / 03$. The Control Interface permits QTM module switching instructions to be transferred from the PDP $11 / 03$ directly to the QTM. Both FIFI and CI are under the control of BASIC language, and programs may be written on the PDP $11 / 03$ to perform module switching, as well as data acquisition and analysis.

The following section gives specific instructions for the system operator so that, given a silicon wafer which has been properly polished and etched, the wafer is viewed with the microscope interfaced to the QTM 720 Image Analyzer. The following section gives detailed instructions to the operator for the actual sample run.

The following QTM 720 modules are used in the present system configuration: ID Auto Detector, MS-3 Standard Computer, two Function Computers, Classifier/ Collector, Variable Frame, Control Interface, Image Editor, Auto Focus, X-Y Stage Control, and the Field-Image-Feature Interface.

## PREPARATION FOR SAMPLE RUN

1. Select proper objective on the microscope for desired magnification (a total optical magnification of $\times 800$ is normally used).
2. Adjust optics for "Kohler illumination," following steps in the microscope manual ${ }^{2}$, if necessary. It is important that the field of view be uniformly illuminated so that features of interest will te detented uniformly.
3. Adjust the light intensity (with filters and/or lamp voltage) to obtain a reading of 1 on the white level meter with light sensitivity switch in MANUAL. The sensitivity is then set to AUTO.
4. Place the sample on a blank field of view and perform shade correction, setting the RANGE at about 10-11 o'clock. If a suitable blank field cannot be found, one may de-focus the field of view so that no distinct features may be identified, and a relatively uniform, featureless field is observed. For best results, the entire standard frame should be detected as uniformiy as possible. (Light sensitivity switch should be in AUTO to perform shade correction.)
5. Place sample at the origin of the scan, which will be the lowest left-hand corner of the sample. Make certain that the sample is firmly held to the stage. Select the size of the $X-Y$ step on the automatic stage control. Generally, the $X$ and $Y$ steps will be of the same size (units are in mm). Determine the number of steps in a single row (X-direction). (The number of fields in a row is one greater than the number of $X$ steps). After setting the number of steps on the automatic stage control, place control in AUTO and push ORIGIN. Whenever manual control of the stage is desired, switch from

AUTO to MANUAL. When returning to AUTO mode, stage must be at ORIGIN. Alsays set ORIGIN after pushing AUTC. At this time, set the Automatic Focusing module to AUTO and SKIP FIELDS to zero.
6. Determine the size of the Variable Frame to be used for scanning and position it. The product of the horizontal and vertical divisions (in picture points) will be the frame area called for at the beginning of the program.
7. There are two twisted-pair leads in the back of the FIFI module which feed into BIG FRAME OUIT and VARIABLE FRAME OUT. It is necessary to interchange these leads if it is desired to perform measurements on dislocations and twin boundaries. For the analysis of twin boundaries, the full frame ( 500,000 picture points) of the T.V. screen is used. This is because the twin boundaries remain in focus over the entire screen area. But for the dislocation pits, half the frame ( $250,000 \mathrm{pp}$ ) is used. This is because the dislocations tend to go out of focus near the edges of the full frame. It will be necessary to determine manually the average feature area (in pp) by sampling several fields throughout the sample. This value is called for in the program. (Note: The automatic stage will have to be placed in the MANUAL mode during this operation, followed by step 5 above).
8. Set proper detection of the features in the field using the "flicker method" and the Detector Module.
9. The Standard Computer, both Function Computers, and the ClassifierCollector should be set to AUTO.

## PREPARING THE PDP $11 / 03$ FOR OPERATION OF THE QTM-720

1. Place the System floppy disc into the left-hand drive of the RXO1 dual disc drive and the data file storage disc into the right hand drive. Turn on power to the PDP 11/03 and to the DECWRITER. "Boot" the system
in the sequence ENABLE-DC-LTC. The symbol \$will appear on the DECURITER
2. Type $D X<C R>$ and the message "RT-11SJ VO2C-02H" will be returned.
3. Type the current date in the format DATE 06-Jun-79 <CR>.
4. Type $R$ QBS203 <CR>, and the symbol * will be returned. Input a carriage return, <CR>, and the message "READY" will be typed out.
5. The current program for defect characterization of silicon is program DS2. Therefore, type OLD "DS2" <CR> and upon obtaining the "READY" response, again type RUN <CR>.
6. The following steps describe where necessary the information called for as input data for the program:

HEADING - Any one line description of the current run. PRINT FILE NAME . . . - This is the name of data file on the appropriate floppy disc where this run will be stored. OPERATOR - Name of onerator. MAGNIFICATION

UNITS
CALIBRATION FACTOR (UNITS/PP)
FRAME AREA (PP) - The Standard Frame area is $500,000 \mathrm{pp}$. QTM OUTPUT DATA DIVIDED BY - It may be necessary to use the classifier-collector module to divide the QTM output data by a power of ten if the OVERFLOW light comes on during sample analysis.

AVERAGE FEATURE AREA (PP) - This must be determined manually before the sample run.
7. The heading for the data output is now printed. The raw data in units of picture points will be typed out in parentheses for each field. These are the actual QTM measurements of the detected features within the frame area in the order : area, perimeter, vertical projection, and horizontal projection.

After the parameters are printed out for each field, a question mark is printed, If a carriage return, 〈CR>, is typed, the next field will be measured and printed out. However, if a $D$ is typed, then the data acquired in the last field of measurement is deleted and the message "LAST FIELD DELETED" is printed.

If an $A$ is typed in response to the question mark, the average of each parameter, along with its standard deviation and standard error of the mean, is printed. The average is taken for all measurements previous to this time, except for fields deleted. Following the average, the field numbers continue consecutively. The average values for Mean Free Path are determined by dividing the cumulative sum of the frame areas by the cumulative sum of the projection. In this case, standard deviation and standard error are not defined.

## COMPUTER PROGRAMMING FOR THE PDP 11/03

The PDP 11/03 minicomputer controls many of the functions of the QTM-720 Image Analyzing System. Programming for this minicomputer determines how the raw data from the QTM is analyzed. Three versions of a computer program designed to analyze the data from silicon samples have been written. The current program being used is "Defects in Silicon $3^{\prime \prime}$, which analyses the raw data faster and allows for a more convenient printout format than in the previous two versions. A Flow Chart of this program is shown (Fig. 17) along with a listing of the BASIC program "Defects in Silicon 3" (Table 26).

In many situations when anaiyzing silicon samples with the quantimet 720, it is necessary to manually edit the image that is being detected. These include situations where extraneous features are present on the surface of the sample such as dust particles or stain marks. Also, due to the uneveness of the sample surface in some locations the entire area in a field cannot be focussed, causing detection problems in the unfocussed areas. In many cases clusters of dislocation pits are joined to the twin boundaries causing the QTM to detect a larger twin area than is really present. In such cases, manual image editing can be used to overcome these problems.

Image editing on the QTM 720 is performed by the use of a light pen coupled with the Image Editing Module. The light pen is used to indicate on the QTM screen the areas or features that are to be edited or manually manipulated. The Image Editor is capable of specifying particular regions or features for measurement and rejecting others. The Image Editor is also capable of filling in imperfectly detected features or separating features that are touching.

The use of the Image Editor as it pertains to the analysis of silicon samples is illustrated by the photographs shown in Figures 18A through 20B.

The first three photographs, Figures 18A through 18C, show the operation of the image editor in the ACCEPT mode. The photograph in Figure 18A shows the QTM screen with the image of a polished and etched silicon sample* displayed. A large field of dislocations can be seen on the left side of the picture with a heavy band of twins running down the center. On the right side of the screen, clusters of dislocation pits are present. The top of the OTM display screen indicates that the image editor is in the "ON" position, and in the ACCEPT mode, and also indicates the count in picture points of the features detected.

[^0]In Figure 18A, the number 13 refers to the counts from the previous field and should be ignored. In Figure 18A, the light pen is shown being used to circle a region that is to be accepted for detection. When the DETECT switch is pushed on the QTM, the area that has been accepted is displayed on the screen while all other areas are not displayed. This is shown in Figure 18B. Only the features in this region will be counted by the QTM and all other features will be ignored. The photograph shown in Figure 18C shows the same specimen area with only the dislocation pits being accepted, and all the twins rejected.

The REJECT mode of the Image Editor operates in much the same way as the ACCEPT mode. This operation is illustrated in the photographs shown in Figures 19A through 19C. In Figures 19A, 19B, and 19C the same specimen area is shown as in the previous photographs.

On the right side of the photograph in Figure 19A, the operator's hand can be seen with the light pen circling an area to be rejected. In Figure 19B, the light pen is pointing towards the region that has been rejected. The features in this region are no longer displayed on the screen when the DETECT switch is pushed on, and these features are no longer counted. Figure 19C shows the same specimen area with most of the dislocation pits rejected leaving only the twins displajed. In these three Figures 19A, 198 and 19C, the count of features detected in picture points is indicated as 87,79 and 13 respectively. The detected feature count was being divided by 100 when these samples were analyzed. The actual number of counts in picture points are 8700,7900 , and 1300. The 1300 counts in Figure 19C are from the residual dislocation pits that have not been rejected. In order to determine the number of dislocations being counted, these numbers must be divided by the average feature area for dislocations, which range between 5 and 10 picture points depending on the sample.

The Image Editor can also be used to separate features which are touching one another. To do this, the Image Editor is put into the CUT mode. This is illustrated in the photographs in Figures 20A and 20B. Figure 20A shows a region containing dislocation pits with a single twin boundary running down the center. Some of the dislocations are touching the twin boundary and, therefore, are being included in the total twin area count. The twin area is indicated as 3183 picture points. In figure 20B the light pen has been traced around the twin with the Image Editor in the CUT mode. This separates the twin from the adjoining dislocation pits. The feature area count is the 2870 picture points, which is the true area of this twin.

The Image Editor need not be used in the analysis of silicon samples if the sample surface is flat and well-polished. However, in samples that are uneven, or in samples where large fields of dislocations are connected with twins, image editing must be used to obtain accurate results.

MEASUREMENT OF TWINS AND DISLOCATION PITS:
In all of the samples analyzed, except the Wacker samples, most of the twins are oriented parallel to one another and run from one edge of the wafer to the opposite edge (parallel to the longitudinal axis of the silicon ribbon, the growth direction). Therefore, in order to measure twin density, 50 fields were chosen along the central transverse axis of the sample perpendicular to the growth direction. In other words, the central transverse axis is perpendicular to the twins. The distance between each of these 50 fields where measurements for twins were made was 0.31 mm . The long dimension of each field was 0.30 mm . Thus, each of these fields were adjacent to one another by a distance of 0.01 mm and, therefore, did not overlap one another. It is important that the fields do not overlap, since the same twin should not be counted twice. At the same time, the fields must be close to one another so that almost all the
twins are counted by the QTM. On the other hand, counting may also be done using a square raster of 50 fields distributed evenly over the entire sample surface. In this case, the horizontal distance separating each field will be 2.5 mm , which is much larger than the long dimension of the frame i.e., 0.30 mm . Therefore, under the method of square raster, there is a possibility that areas in the sample where the twin or dislocation density is very high may not be counted. This will result in large errors. Therefore, all the 50 fields were counted along the central transverse axis of the samples.

It has also been found that the density of dislocation pits in the samples have longitudinal symmetry similar to the twins. Therefore, for dislocation pit density measurements, all the fifty fields were chosen along the central transverse axis of the silicon samples.

## MEASUREMENT OF AVERAGE AREA OF TWINS AND DISLOCATION ITT5:

Before measurements were made for twins, each sample was scanned to determine manually the average area of one twin. The method of determining the averug twin area is as follows: First, the sample surface was randomly scanned, and those fields were selected where the twins were not touching each other. Each field, generally containing more the 5 distinct twins, were then displayed on the display module of the QTM. The total area of all the twins in each field was determined and divided by the number of twins in that field to get the average twin area for that field. The average twin area was then determined in an additional 4 fields. The arithmetic average was then calculated from the average twin area in these five fields. Generally, 30 to 40 twins were used in 5 fields to get the averaga twin area. The same procedure was used to obtain the average dislocation pit area. The average twin area in in each sample was then fed into the QTM software. Thin is an important step to get the actual number of twins and dislocation pits, expucially in areas where
the densities of these defects are high and they touch one another, In order to verify that the average area of a twin so obtained was accurate, an additional six fields were selected at random where the twin density was high, and the twins were touching one another. The twin density in each of these six fields were counted manually, and also counted by the QTM using the average area of a twin. The entire procedure was repeated until close agreement was reached between manual counting and QTM counting. After this procedure, measurements were then made on all the fields using the automatic QTM mode.

## EXPLANATION OF COMPUTER PRIN:OUTS:

In the computer printouts, the first paragraph shows the name of the computer program and date.

The second paragraph shiws the MRI and JPL sample numbers.
The third paragraph 1ists; 1) the name/names of the operator; 2) magniffcation being used ( 800 X ); 3) units used f.e., mm for twins, and microns for dislocation pits; 4) calibrated equivalent value of one picture point in the units being used; 5) frame area used; 6) QTM output data was divided by 100 and corrected in the case of twin measurements to avoid frequent overflow problems in the Classifier-Collector. In the case of dislocation pits, the data was divided by 1 as indicated in the computer printouts; 7) average feature area (pp), for twins and dislocation pits.

All the information listied in the third paragraph of the computer printouts were fed into the computer on its command before collecting the data using the automatic mode.

The frame area of a standard frame in the QTM is 500,000 picture points (pp). In case of twins, the standard frame was used. However, during dislocation density measurements the uneven sample surfaces caused problems in focusing dislocation pits over the entire standard frame. Therefore, during dislocation density measurements half the standard frame ( $250,000 \mathrm{pp}$ ) wis used. This is listed
as "Frame Area" in the QTM data sheets. The unit of measurement was millimeter for twins, and microns for dislocation pits.

The fourth paragraph of the computer printout lists the titles for the different measurements, which are explained below:

FLD: ( $A, P, V P, H P$ ) indicates the sequence number of the field in which measurements were made. The raw data in terms of picture points are also shown in parentheses. The raw data listed is area, perimeter, vertical projection, and horizontal projection of the detected features in each field.

No. denotes the total number of features detected in any field. This is obtained by dividing the total area of a feature by the average area of that feature.

No./AREA: denotes the computed number of features $/ \mathrm{mm}^{2}$ or features/ microns ${ }^{2}$ in each field.

MFPV: denotes the mean free path in the vertical direction. This quantity is the frame area divided by the vertical projection of all detected features in the field (frame).

MFPH: denotes mean free path in the horizontal direction. This is the horizontal analogue of MFPV.

L/A: This quantity is length of detected features per unit area. The unit area is $\mathrm{mm}^{2}$ in the case of twins, and microns ${ }^{2}$ in the case of dislocation pits.

The quantity $L / A$ is subject to large errors when twin bands are present. The QTM computes L/A by dividing the perimeter by 2. A twin band usually contains 20 to 100 individual twins, many of them touching one another. The QTM will compute L/A by dividing the perimeter of the twin band by 2 . In other words, the QTM may count the entire twin band as one large area rather than consisting of several individual twins. Thus, L/A is subject to large errors and is underestimated by QTM.

The attached computer printouts show, after 25 and 50 fields, the computed values of average, standard deviation, and standard error for all data from field No. 1 onwards. This averaging can be done at any time during the course of the measurement (Table 28).

The grain boundaries in each sample were counted under the binocular microscope using $7 X$ magnification. Most of the grain boundaries were parallel or approximately parallel to the twins.

Due to the large volume of computer printouts, all of these printouts will not be included in this report but are available in Quarterly Progress Reports (MRI-255, MRI-260, MRI-264, MRI-269, MRI-273). The data on twin boundary density, dislocation pit density, and grain boundary length have been summarized in Tables 5 to 25.

A complete computer printout for Mobil Tyco sample MRI \#100 is shown in Table 28 to illustrate the data printout format. The data for all of the Motorola samples, Mobil Tyco samples MRI 78-134, and Honeywell sample are recorded on floppy discs. The data from the other samples are recorded on paper tape.

## SECTION IV

## RESULTS

A total of one hundred and seventyfour (174) silicon samples, approximately 1200 square centimeter, have been analyzed to date. One hundred and thirtythree (133) of these samples were manufactured by Mobil Tyco, thirtytwo (32) by Motorola, seven (7) by IBM, one (1) by Honeywell, and one (1) by Wacker. These samples were analyzed for twin boundaries, grain boundaries, and dislocation pits. Twin boundary and dislocation pit measurements were made using the QTM-720 as described in this report, and grain boundary measurements were made using a binocular microscope at 7 X magnification. Data from these measurements are summarized in Tables 5 to 25. Histograms showing the distribution of twin boundary density, dislocation density, and grain boundary length in the Mobil Tyco and Motorola samples are shown in Figures 22 to 27.

Due to the large number of computer printouts containing the data on the 174 samples analyzed, these printouts are not included in this report. The information is available on floppy discs for later analysis, however. The data on vertical mean free path (VMFP) and horizontal mean free path (HMFP) have not been summarized and included in this report. It is unclear at present whether this data will be pertinent to the correlation of defect density with conversion efficiency. If it is found to be useful, this data will be included in later reports.

Diagrams showing the sample position as cut from the ribbons for the Motorola samples, the IBM samples, and Mobil Tyco samples 19-134 are shown in Figures 28 to 36. Also, on these diagrams are listed the dislocation pit and twin densities as found by QTM analysis.

## MOBIL TYCO SAMPLES

Two types of Mobil Tyco EFG Silicon samples have been analyzed. Mobil Tyco EFG -RH (Resistance Heating) and Mobil Tyco EFG -RF (Radio Frequency Heating) samples.

Mobil Tyco samples MRI \#1-18 are EFG -RH samples. These samples have fairly low dislocation and twin boundary densities as compared with later analyzed Mobil Tyco samples. The average dislocation density for these samples is 0.0107 dislocations $/ \mu \mathrm{m}^{2}$ and the average twin boundary density is 308.7 twins $/ \mathrm{mm}^{2}$ (as calculated from Table 5).

Mobil Tyco samples MRI $119-30$ are EFG-RH samples. These were some of the first Mobil Tyco EFG-RH samples to be manufactured and contain a large number of SiC particles. The number of SiC particles in these samples are listed in Table $\dot{b}$. These samples contain very large dislocation densities. The average dislocation density for these samples is 0.0748 dislocations $/ \mu \mathrm{m}^{2}$. The average twin density for these samples is 261.79 twins/mm ${ }^{2}$, and the average grain boundary length/em ${ }^{2}$ is 1.14 . The high dislocation density of sample MRI \#19-30 seems to indicate that dislocations tend to nucleate around SiC particles. This high dislocation density around precipitate particles has also been observed by other researchers in EFG ribbons ${ }^{3}$. The highest local dislocation density found in samples $19-30$ was .407 dislocations $/ \mu m^{2}$ which corresponds to a density of $4.07 \times 10^{7}$ dislocations $/ \mathrm{cm}^{2}$. This local dislocation density was found in sample MRI \# 30. The average dislocation density in this sample is . 084 dislocations $/ \mathrm{mm}^{2}$ or $8.4 \times 10^{6}$ dislocations $/ \mathrm{cm}^{2}$. These samples have slightly lower grain boundary length/ $/ \mathrm{cm}^{2}$ than the other Mobil Tyco samples.

In the later Mobil Tyco samples, few SiC particles were found and lower dislocation densities were observed.

Mobil Tyco samples 31-77 are of the type EFG-RF., The twin boundary density, dislocation pit density, and grain boundary length are listed in
tables 7 to 12. The average dislocation pit density for samples 31-72 is .0408 dislocations $/ \mu \mathrm{m}^{2}$, the average twin density was found to be 556.93 twins $/ \mathrm{mm}^{2}$, and the average grain boundary length $/ \mathrm{cm}^{2}$ is 1.86 .

Mobil Tyco samples 78-134 are of the EFG -RH type. The average dislocation density for these samples is . 0292 dislocations $/ \mu^{2}$, the average twin density is 750.49 twins $/ \mathrm{mm}^{2}$, and the average grain boundary length $/ \mathrm{cm}^{2}$ is 2.95. The mean defect densities for all the 133 Mobil Tyco are 0.037 dislocations $/ \mu \mathrm{m}^{2}$ (Fig. 24), 540.4 twins $/ \mathrm{mm}^{2}$ (Fig. 23), and $2.35 \mathrm{~cm} / \mathrm{cm}^{2}$ grain boundary length.

As mentioned previously most of the twins in the Mobil Tyco samples run longitudinally through the ribbons, therefore samples cut from the same ribbon, or from the same side of a ribbon tend to have similar twin densities. Detailed discussions of the twinning process for EFG ribbons are presented in references 4 and 5. Dislocation pit density also has some longitudinal symmetry, but the dislocation pit density is more variable from sample to sample in the same ribbon. The highest dislocation density in the Mobil Tyco samples is found in areas where few twins are present, and in heavy twin bands few dislocations pits are found. The highest local dislocation pit density was found in sample MRI No. 101 and is . 528 dislocations $/ \mu \mathrm{m}^{2}$, i.e., $5.28 \times 10^{7} / \mathrm{cm}^{2}$.

The surfaces of all of the Mobil Tyco samples are very uneven with surface ripples. These surface ripples have been observed by other researchers and are described in more detail by De Angelis ${ }^{6}$.

Figures 28 to 34 are diagrams showing the position of the Mobil Tyco samples as cut from the ribbons. The twin density and the dislocation pit density are shown on these diagrams.

MOTOROLA SAMPLES:
Data on twin boundary density, dislocation pit density, and grain boundary length for thirty two Motorola samples are summarized in Tables 22 to 25.

Figure 35 indicates the sample position as cut from the Motorola ribbons. The figure also indicates the twin boundary and dislocation pit densities. Figures 25,26 , and 27 are histograms relating twin boundary densities, dislocation pit densities, and grain boundary length to the number of samples analyzed.

There is no clear cut relationship between twins, grain boundaries, and dislocation pits among these samples whether cut from the same ribbon or when samples from different ribbons are compared.

Specimens from the ribbon 6-840 contains the lowest twin and dislocation densities (especially, sample 6-840 G). This ribbon, however, has very high grain boundary length/ $\mathrm{cm}^{2}$. In general, the twin, dislocation pit, and grain boundary measurements for the other specimens taken from the ribbons 6-792, 6-837, 6-656, and 6-791 are comparable in magnitude.

There are large variations in the twin boundary, dislocation pit, and grain boundary measurements for individual samples from the same ribbon. For example, for the ribbon $6-340$ the highest twin density is $1272.02 \mathrm{twins} / \mathrm{mm}^{2}$ and lowest twin density is 157.91 twins $/ \mathrm{mm}^{2}$. The highest dislocation density from this ribbon is .0129 dislocations $/ \mu^{2}$ and the lowest is .0014 dislocations $/ \mu m^{2}$.

There seems to be no relationship between twin boundaries, dislocation pits, and grain boundaries with respect to the specimen position on the ribbon.

The average dislocation pit density for all of the Motorola samples is .0136 dislocation pits $/ \mu \mathrm{m}^{2}$, the average twin density is $1032.21 \mathrm{twins} / \mathrm{mm}^{2}$, and the average grain boundary length $/ \mathrm{cm}^{2}$ is 3.27 (Figs. 25, 26, and 27).

As compared with the Mobil Tyco samples, the Motorola samples have a higher average grain boundary length and a higher twin density, but have a lower average dislocation density. It can be seen however, that the Motorola samples have a larger variation in twin density, dislocation pit density, and grain boundary length than in the Mobil Tyco samples. In the Motorola samples, the twin boundaries -31-
and dislocation pits have the same longitudinal symmetry as in the Mobil Tyco samples, but the twin bands and dislocation pit areas seem to be more intermittent, and do not run throughout the whole length of the ribbons. This explains why samples cut from the same ribbon have such a large variation in defect densities.

IBM SAMPLES
Data on twin boundary density, dislocation pit density, and grain boundary length for seven (7) IBM samples are listed in Table 21. The average dislocation density for the IBM samples is . 010 dislocation pits $/ \mu \mathrm{m}^{2}$, the average twin density is 499.64 twins $/ \mathrm{mm}^{2}$, and the average grain boundary length $/ \mathrm{cm}^{2}$ is 1.11 .

The IBM samples were the only samples analyzed that seemed to have a systematic variation of defect density with respect to specimen position as cut from the ribbon. This variation is shown graphically in Figure 21. This figure indicates that twin boundary density decreased as the ribbon was grown. No such variation was found in these samples for dislocation pit density or for grain boundary length.

## HONEYWELL SAMPLE

The Honeywell sample consisted of a ceramic substrate coated with a film of silicon. The densities of dislocations, grain boundaries, and twin boundaries are listed in Table l. The dislocations tended to be more evenly distributed throughout the Honeywell sample than in the Mobil Tyco samples and the dislocation density is slightly less. The twin density in this sample is also lower than that found in the Mobil iyco samples.

The twin boundaries and dislocation pits tended to have longitudinal symmetry as in the Mobil Tyco and Motorola samples.

The surface of the Honeywell sample shows ripples that are approximately 2 mm apart and run perpendicular to the twin boundaries.

## HACKER SAMPLE:

One Hacker sample was analyzed for twin boundaries on the QTM; the printout of data on this sample is listed in Table 2.7. Unlike the other samples analyzed, the twin boundaries in the Wacker samples do not run parallel to one another. The twins within different grains are oriented in different directions. To further complicate the counting of these defects, all of the twin boundaries intersect the grain boundaries, and there are a large number of such intersections in each field of view.

Wacker sample No. 7 was the first sample to be analyzed on the QTM. This sample had a surface area of $40.32 \mathrm{~mm}^{2}$. As shown in Table 17, a total of 50 fields (or frames) were analyzed on the QTM. These 50 fields were uniformly distributed in a square raster covering the entire sample surface.

The average twin density was found to be 15.8 twins $/ \mathrm{mm}^{2}$, which is much lower than that found in the other samples analyzed. The grain boundary length in these samples, however, is much higher than in the samples from other manufacturers, although grain boundary length $/ \mathrm{cm}^{2}$ was not quantitatively determined for the Wacker sample.

## SECTION V

## CONCLUSIONS

Procedures have been developed for the analysis of defects in silicon sheet using a QTM-720 Image Analysis system. The analysis technique proved to be rapid, accurate, and reproducible.

Chemical polishing and etching techniques have been developed that can effectively reveal structural defects and prepare the silicon surface for automatic QTM analysis. These procedures have been developed for Mobil Tyco, Motorola, IBM, Honeywell, and Wacker samples.

One hundred and seventy four (174) silicon samples, approximately 1200 square centimeter surface area, have been analyzed for twin boundary density, dislocation pit density, and grain boundary length. The data from these samples being included herein.

The samples analyzed under this contract have been returned to JPL and may be manufactured into solar cells with the electrical conversion efficiency measured. The conversion efficiency can then be correlated to the defect density and quantitative relationships obtained between twin boundary density, dislocation density, grain boundary length, and conversion efficiency.

## SECTION VI

## REFERENCES

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3. M. Leipold, R. De Angelis, "Structure Development ir Silicon Sheet By Shaped Crystalization", Proceedings from the International Photovoltaic Solar Energy Conference, D. Reidel Publishing Company, 1978.
4. M. Leipold, R. Stirn, J. Zoutendyk and R, De Angelis, "Evaluation of Silicon Ribbon Material for Solar Cell Fabrication", Proceedings of the Eleventh IEEE Photovoltaic Conference, May, 1975.
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6. R. J. De Angelis, "Structural Characterization of Edge-Defined Film Growth (EFG) Silicon Ribbon", unpublished report.


Figure 1. IBM \#l-section l-area 1, micrograph of silicon ribbon surface showing intersection of three grains after cleaning organic materials from surface of ribbon. Mag 200X


Figure 2. IBM \#1-section l-area 2, micrograph of ribbon surface showing grain boundaries after cleaning organic materials from surface of ribbon. Mag 200X


Figure 3. IBM \#1-section 1-area 1, micrograph of ribbon surface, shown earlier in Fig. 1, after oxide removal. Mag 200X


Figure 4. IBM \#l-section 1-area 2, micrograph of ribbon surface, shown earlier in Fig. 2, after oxide removal. Mag 200X


Figure 5. IBM \#1-section 1- area 1 , micrograph of ribbon surface, shown earlier in Fig. 1, after chemical polishing. Growth lines are removed and grain boundaries are revealed.

Mag 200X
ORIGINAL PAGE IS
OF POOR QUALITY


Figure 6. IBM \#1-section 1- area 2, micrograph of ribbon surface, shown earlier in Fig. 2, after chemical polishing. Growth lines are removed and grain boundaries are revealed.

Mag 200X


Figure 7. IBM 46 - Section 1 - Side 1, aicrograph of ribbon surface after chemical polishing. Mag 200X



$$
\begin{aligned}
& \text { Figure9. } \begin{array}{l}
\text { IBM } \neq 6 \text { - Section } 1 \text { - Side } 1 \text {, micrograph of } \\
\text { ribbon surface after a } 15 \text { second etch by } \\
\text { Etching Solution } \mathrm{I} \text {. } \\
\end{array} \quad \begin{array}{l}
\text { Mag } 500 \mathrm{X}
\end{array} .
\end{aligned}
$$


,Figure 10. IBM $\$ 6$ - Section 1 - Side 1, micrograph of ribbon surface, shown earlier in Fig.7, E and 9, after a 30 second etch by Etching Solution I


Figure 11. IBM $\# 6$ - Section 1 - Side 2 , micrograph of ribbon surface after chemical polishing.

Mag 200X


[^1]

Eigure 13. IBM $\# 6$ - Section 1 - Side 2, micrograph of ribbon surface, shown earlier in Figs. 11 and 12 after a 60 second etch by Etching Solution II.

Mag 200X


[^2]ribbon surface, shown earler in Figs.11, 12,
and 13 after a 90 second etch by Etching Solution II.


Figure 15.
IBM ${ }^{2} 6$ - Section 3 - Side 2 - Area 1,
micrograph of ribbon surface after chemical polishing.

Mag 200X


Eigure16. IBM \$6 - Section 3-Side 2-Area 1, micrograph
of ribbon surface, as shown in Fig. 15, after a
60 second etch by Etching Solution III
Mag 200X


Figure 16 A.
CBM $\# 6$ - Section 3 - Side 2 - Area 2 , micro
of ribbon surface after a 60 second etch by
Etching Solution III.
Mag 200X




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Figure 18A - Mobil Tyco # 33 - Field # 1
    Photograph from 2TM display screen
    Mag. 800X
```



```
Figure 18B-Mobil Tyco # 53 - Field # 1
    Photograph from GTM display screen showing only the
    area of the sample that has been accepted.
        Mag. 800x
```


Figure 18 C - Mobil Tyco \#53-Field \#
Photograph from grM display screen showing dislocation
pits only.
Mag. 800X


[^3]

[^4]

[^5]

Figure 20A. Mobil Tyco \#53-Field \#2
Photograph from grM display screen showing an area of dislocation pits with one twin boundry.

Mag. 800X


```
Figure 20B- Mobil Tyco # 53 - Field # 2
    Photograph from CTM display screen showing the same
    area as in Fig.20A. The twin has been saparated from
    the dislocation pits by use of the image editor.


Fig. 21. Graphical plot showing systematic variation in twin density with respect to specimen location in IBM Ribbon No. 4-45\%.
MOBIL TYCO SAMPLES

GRAIN BOUNDARY LENGTH/cm \({ }^{2}\)
FIGURE 22
llistogram of grain boundary length of Mobil Tycc samples
-51-


FIGURE 23
llistogram of twin houndary density of Mobil Tyco samples
MOBIL TYCO SAMPLES

FIGURE 24
llistogran of dislocation pit density of Mobil Tyco samples
MOTOROLA SAMPLES

FIGURE 25
llistogram of grain boundary length of Motorola samples
MOTOROLA SAMPLES


\footnotetext{
FIGURE 26
lisstogran of twin boundary density of Motorola samples
}

FIGURE 27
saldues eloajoz fo kitsuəp fid uotzeoolstp fo dealbozsth

MOBIL TYCO
RIBBON *5-685 (18-63-1)

* MOBIL TYCO 5-685 (18-63-1) SPEC. L LOST IN SCRIBING.
\(\pm\) THIS PORTION OF SPECIMEN D LOST IN SCRIBING.
T SPEC. \(D\) MEASURED APPROX. 0.018 in thick.

Figure 28. Diagram showing the position of Mobil Tyco samples MRI \# \(19-30\) as cut from ribbon \(5-685\). Twin density (per \(\mathrm{mm}^{2}\) ) is printed at the top of each sample box, the dislocation density (per \(\mu \mathrm{m} 2\) ) is printed at the botton on each sample square.

MOBIL TYCO
RIBBON 144-36,*5-742


MOBIL TYCO
RIBBON 145-76,*5-744


Figure 29. Diagram showing the position of Mobil Tyco-samples MRI \# 31-46 as cut from ribbons 5-742 and 5-744.

\(7.43 \mathrm{in}^{2}\)
Useable ared \(3.89 \times 1.91 \mathrm{in}\).

MOBIL TYCO
144-36
\#5-743

\(7.35 \ln ^{2}\)

MOBIL TYCO
Piece No. 1 of \#5-640
MULTI RIBBON RUN

\(7.60 \mathrm{ln}^{2}{ }^{2}\)
Useable area \(3.9 \times 1.95\)

MOEIL TYCO
Piece No. 2 of
\#5-640 MULTI RIBBON RUN

\(7.70 \mathrm{in}^{2}\)

Fig.30. Diagram showing the position of Mobil Tyco samples MRI \(\frac{27-77}{}\) as cut from ribbons \(5-745,5-743,5-640 \% 1\) and 5-640 \(\%\).

MOBIL TYCO
SAMPLE *16-163-2 JPL \#5-867

\(C=1.00\) approx.
\(D=0.94\) approx.

Figure 31. Diagram showing the position of Mobil Tyco samples MRI \#78-90 as cut from ribbon 5-867

MOBIL TYCO JPL \#5-640


Figure 32. Diagram showing the position of Mobil Tyco samples MRI \#91-98 as cut from ribbon 5-640.

MOBIL TYCO
SAMLE 16-166-1-14 JPL 5-990


Figure 33. Diagram showing the position of Mobil Tyco samples IRI \#90-105 as cut from ribbon 5-990.

MOBIL TYCO
JPL \#- 5 -1092 EFG RUN 16-187. STATION I SAMPLE 69

MOBIL TYCO
JPL"5-1094 EFG
RUN 16-187, STATION 3
SAMPLE 33

MOBIL TYCO
JPL \# 5-1063 EFG
RUN 16-184
SAMLE 184-88 (Marked on packaga)
"I84-225" MARKED IN INK ON SPECIMEN
MOBIL TYCO
JPL \#5-1063 EFG
RUN 16-184
SAMPLE 184-175 (Marked on package)
"184-366" MARKED IN INK ON SPECIMEN


Figure 34. Diagram showing the nosition of Mobil Tyco samples MRI \(4100-134\) as cut from ribbons 184-88, 134-175, 5-1094-33, and 5-1094-69


MOTOROLA 918-A NO. 6-837


MOTOROLA S889-C NO. 6-791
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline \multicolumn{7}{|l|}{-1 1 yp.-m} \\
\hline 802.77 & 430.62 & 417.11 & 1272.02 & 582.97 & 917.21 & 157.91 \\
\hline \[
\begin{gathered}
A \\
.0129^{\circ}
\end{gathered}
\] & \begin{tabular}{l}
\[
B
\] \\
0067
\end{tabular} & \[
\begin{gathered}
C \\
.0069
\end{gathered}
\] & D .0014 &  & \[
{ }_{.}^{F}
\] & \({ }_{\text {G }}^{\text {G }}\). \({ }^{\circ}\) \\
\hline 7.5 & 4.5 & 3.8 & 4.4 & 5.8 & 6.6 & 5.6 \\
\hline
\end{tabular}

MOTOROLA 829-A NO. 6-840

Figure 35. Diagram showing the position of Motorola samples MRI \({ }^{\mathbf{\pi}} 1-32\) as cut from ribbons 6-792, 6-837, 6-656, and 6-840.

Ribbon Identified as 18M \#4-457


Figure 36. Diagram showing the position of IBM samples MRI \(\# 1-7\) as cut from ribbon 4-457.

\section*{TABLE1}

\section*{CUEMYCAL ZOLISHING OF HACKER SAMPLES}
\begin{tabular}{|c|c|c|c|c|}
\hline Tempe: & zature & ( \({ }^{\circ} \mathrm{C}\) ) & Time (sec.) & Sursace Conditsons \\
\hline & 50 & & 30 & slighe smoghaniag of susface; but no polishing \\
\hline & 50 & & 45 & underpolishing of surface, growth lines rematn. \\
\hline & 50 & & 60 co 75 & slight undarpolishing. Subgrain sype structure (due to iacets) becomes larger, and, in some places, becomes Eaine and stares disappearing. GeE staining and pite tomation inside subgrain type structure. \\
\hline & 50 & & 80-85 & Good even polishing. Subgrain type structure, and pies within subgrains completely disappear. \\
\hline & 70 & & 45 & slight underpolishing \\
\hline & 80 & & 55 & reasonably good polish \\
\hline \multicolumn{2}{|l|}{\multirow[t]{2}{*}{Noce: (1)}} & \multicolumn{3}{|l|}{THe of polishing is to be increased or decreased depending on how soon and how fast bubbles evolve from sample suriace.} \\
\hline & & \multicolumn{3}{|l|}{For each polishing operation, a inash solution must be used since the strength of solucion decreases drascleally after just one use.} \\
\hline
\end{tabular}

\section*{TABLE2}

\section*{CHEMICAL POLISHING OF IEM SAMPTES}
\begin{tabular}{|c|c|c|}
\hline Temperature ( \({ }^{\circ} \mathrm{C}\) ) & Itme (sec.) & Surtace Condition \\
\hline 30 & 30 & growth Lines persist. Faceting persists. \\
\hline 50 & 45 & growth lines disappeaz, bue facees join together to form subgriin type structure. \\
\hline 50 & 60 & surface appears very aven and bright, however, Eaint remnants of उubgrasa bype structure still persises. \\
\hline 50 & 85 to 90 & Good even polishing \\
\hline
\end{tabular}

Note: (1) Trme of polishing is to be increased or decreased depending on how soon and how iast bubties evolve Erom sample surface.
(2) For each polishing operation, a Eresh golution must be used since the strangeh of solutiod decreases drastically aEser just one use.

\section*{TABLE 3}

\section*{CHEIICAL POLISHIMG OF MOTOROLA SAAPLES}

Polishing solution: mixture of \(\mathrm{FNO}_{3}: 4 \mathrm{H}: \mathrm{CH}_{3} \mathrm{COOH}=1: 2: 3\) by volume
\begin{tabular}{|c|c|l|}
\hline Temperature ( \({ }^{\circ} \mathrm{C}\) ) & Time (Sec.) & Surface Condition \\
\hline 50 & 30 & \begin{tabular}{l} 
Growth 1ines persist. \\
Sub-grain type structures \\
present \\
Good even polishing
\end{tabular} \\
50 & \(35-45\) & \begin{tabular}{l} 
Faceting develops
\end{tabular} \\
\hline
\end{tabular}

\section*{TABLE 4 \\ CHEMICAL POLISHING OF MOBIL TYCO SAMPLES}

Polishing solution: mixture of \(\mathrm{HNO}_{3}: \mathrm{HF}: \mathrm{CH}_{3} \mathrm{COOH}=1: 2: 3\) by volume
\begin{tabular}{|c|c|l|}
\hline Temperature \(\left({ }^{\circ} \mathrm{C}\right)\) & Time (Sec.) & Surface Condition \\
\hline 50 & 30 & \begin{tabular}{l} 
Growth lines persist. \\
Sub-grain type structures \\
present \\
Good even polishing
\end{tabular} \\
50 & 40 & Faceting develops \\
50 & 50 & \\
\hline
\end{tabular}

TABLE 5

ANALYSIS OF MOBIL TYCO SAMPLESF゙ M-5-738
\begin{tabular}{|c|c|c|c|c|c|}
\hline Sample No . MRI & Average No. of Twins/ field & Average No. of Twins/mm \({ }^{2}\) & No. of grain Boundaries & Average No.of dislocations/ field & Average No.of dislocations/2 \\
\hline 1 & 18.0 & 261.2 & 5 & 362.6 & 0.010 \\
\hline 2 & 26.0 & 368.5 & 2 & 688.6 & 0.020 \\
\hline 3 & 31.8 & 451.9 & 2 & 411.4 & 0.012 \\
\hline 4 & 13.1 & 186.8 & 5 & 256.3 & 0.007 \\
\hline 5 & 14.6 & 207.9 & 6 & 387.8 & 0.017 \\
\hline 6 & 1.3 & 18.4 & 6 & 485.3 & 0.014 \\
\hline 7 & 9.7 & 137.7 & 5 & 505.8 & 0.014 \\
\hline 8 & 16.7 & 238.1 & 3 & 495.4 & 0.014 \\
\hline 9 & 24.6 & 350.3 & 0 & 401.0 & 0.011 \\
\hline 10 & 15.8 & 224.7 & 7 & 368.0 & 0.010 \\
\hline 11 & 32.8 & 4561 & 4 & 250.4 & 0.007 \\
\hline 12 & 13.2 & 188.0 & 4 & 578.1 & 0.016 \\
\hline 13 & 27.2 & 387.0 & 1 & 353.6 & 0.010 \\
\hline 14 & 39.6 & 563.3 & 0 & 143.2 & 0.004 \\
\hline 15 & 27.0 & 384.0 & 7 & 227.6 & 0.006 \\
\hline 16 & 33.0 & 470.0 & 4 & 197.1 & 0.006 \\
\hline 17 & 34.5
11.4 & 490.5
162.2 & 1 & 214.1 & 0.006 \\
\hline 18 & 11.4 & 162.2 & 4 & 503.7 & 0.014 \\
\hline
\end{tabular}
TABLEG
ANALYSIS OF MOBIL TYCO SAMFLES
\begin{tabular}{|c|c|c|c|c|c|c|c|}
\hline MRI Sample (Mobil) Tyco) & JPL No. & Ave. No.of Twins/field & No.of Si Carbide Particles & Ave. No. 0 \{ Twins/mm & Grain boundary length/cm & Ave.No.of Disl./field & \begin{tabular}{l}
Ave. No. of \\
Dis1./fin
\end{tabular} \\
\hline 19 & 5-685 A & 24.40 & 37 & 347.085 & 1.22 & 2759.08 & 0.078 \\
\hline 20 & 5-685 В & 14.76 & 55 & 209.916 & 0.89 & 3702.18 & 0.105 \\
\hline 21 & 5-685 C & 21.25 & 21 & 302.189 & 1.39 & 2480.51 & 0.071 \\
\hline 22 & 5-685 E & 14.52 & 50 & 206.449 & 0.54 & 3595.42 & 0.102 \\
\hline 23 & 5-685 F & 20.76 & 27 & 295.247 & 1.08 & 2219.70 & 0.063 \\
\hline 24 & 5-685 G & 23.26 & 28 & 330.782 & 1.91 & 1191.12 & 0.034 \\
\hline 25 & 5-685 II & 19.10 & 48 & 271.659 & 0.88 & 1796.13 & 0.051 \\
\hline 26 & 5-685 J & 17.78 & 23 & 252.923 & 0.80 & 1818.59 & 0.052 \\
\hline 27 & 5-685 K & 12.87 & 30 & 182.978 & 1.82 & 2914.59 & 0.083 \\
\hline 28 & 5-685 M & 10.86 & 21 & 268.240 & 0.93 & 3536.89 & 0.101 \\
\hline 29 & 5-685 N & 15.45 & 28 & 219.769 & 1.15 & 2606.93 & 0.074 \\
\hline 30 & 5-685 P & 17.87 & 18 & 254.185 & 1.02 & 2960.46 & 0.084 \\
\hline
\end{tabular}

AMALYSIS OF MOBIL TYCO SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline \begin{tabular}{l}
MRI \\
Sample \\
f \\
(Mobil \\
Tyeo)
\end{tabular} & JPL No. & Avg. No. of Twins/field & Avg. No. 2 Of Twins/mm & Grain boundary 2 length/cm \({ }^{2}\) & Avg. No. of Dislocation Pits/field & Avg. No. of Dislocation
Pits/um \\
\hline 31 & 5-744 E & 28.10 & 399.595 & 1.12 & 792.45 & 0.023 \\
\hline 32 & 5-744 F & 10.60 & 278.780 & 1.76 & 814.16 & 0.023 \\
\hline 33. & 5-744 H & 25.96 & 369.253 & 2.32 & 1161.55 & 0.033 \\
\hline 34 & 5-744 G & 22.05 & 313.650 & 2.51 & 1512.83 & 0.043 \\
\hline 35 & 5-744 A & 16.89 & 240.180 & 2.01 & 2704.46 & 0.077 \\
\hline 36 & 5-744 B & 29.35 & 417.464 & 1.20 & 1861.84 & 0.053 \\
\hline 37 & 5-744 C & 51.78 & 736.382 & 1.74 & 1189.71 & 0.034 \\
\hline 38 & 5-744 D & 59.35 & 844.084 & 0.74 & - 1256.99 & 0.036 \\
\hline
\end{tabular}

ANALYSIS OF MOBIL TYCO SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|c|}
\hline Sample No. & \multicolumn{2}{|l|}{\[
\mathrm{JPL}
\]
No.} & Avg. No. of Twins/field & \[
\begin{aligned}
& \text { Avg. No. }{ }^{\text {Twins } / m m i} \\
& \mathrm{~T}^{2}
\end{aligned}
\] & Grain boundary length/cm & Avg. No. of Dislocation Pits/field & Avg. No. of Dislocation Pits/ \(/ \mathrm{m}^{2}\) \\
\hline 39 & 5-742 & A & 38.83 & 552-220 & 1.76 & 2740.94 & 0.078 \\
\hline 40 & 5-742 & B & 28.15 & 400.298 & 1.6 & 1798.02 & 0.051 \\
\hline 41 & 5-742 & \(C\) & 53.75 & 764.476 & 1.50 & 949.78 & 0.027 \\
\hline 42 & 5-742 & 0 & 46.79 & 665.410 & 1.57 & 846.78 & 0.024 \\
\hline 43 & 5-742 & E & 45.57 & 648.059 & 0.80 & 1904.34 & 0.054 \\
\hline 44 & 5-74i & F & 29.32 & 416.969 & 1.76 & 2705.71 & 0.077 \\
\hline 45 & 5-742 & \(\underline{G}\) & 49.34 & 701.677 & 2.11 & 1780.49 & 0.051 \\
\hline 46 & 5-742 & H & 48.64 & 697.801 & 0.64 & 1979.37 & 0.056 \\
\hline
\end{tabular}

TABLE 9

\section*{ANALYSIS OF MOBIL TYCO SAMPLES}
\begin{tabular}{|c|c|c|c|c|c|c|c|c|}
\hline \(:\) & \[
\stackrel{\text { MRI }}{\text { Sample }}
\] & \[
\begin{gathered}
\text { JPL } \\
\text { Sample }
\end{gathered}
\] & & No. of Twins/field & \[
\begin{gathered}
\mathrm{No} . \quad \text { of } \\
\text { Twins } / \mathrm{mm}_{2}^{2}
\end{gathered}
\] & Grain boundary, length/ \(\mathrm{cm}^{2}\) & No. of Dislocation Pits/field & No. of Dislocagion Pits/um \\
\hline & 47 & 5-745 & A & 21.78 & 309.738 & 0.72 & 1949.83 & 0.055 \\
\hline - & 48 & 5-745 & B & 53.10 & 755.151 & 1.93 & 1166.29 & 0.033 \\
\hline & 49 & 5-745 & c & 30.53 & 434.213 & 1.82 & 2428.95 & 0.069 \\
\hline & 50 & 5-745 & D & 60.51 & 860.580 & 3.44 & 1122.38 & 0.032 \\
\hline \(\therefore\) & 51 & 5-745 & E & 32.26 & 501.439 & 3.31 & 2583.40 & 0.073 \\
\hline & 52 & 5-745 & F & 43.53 & 620.473 & 2.30 & 1493.01 & 0.042 \\
\hline & 53 & 5-745 & G & 48.25 & 686.249 & 2.00 & 1556.36 & 0.044 \\
\hline \(\because\) & 54 & 5-745 & H & 59.19 & 841.787 & 1.84 & 1434.39 & 0.042 \\
\hline
\end{tabular}

\section*{TABLE 10}

\section*{ANALYSIS OF MOBIL TYCO SAMPLES}
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline \[
\begin{gathered}
\text { MRI } \\
\text { Sample }
\end{gathered}
\] &  & No. of Twins/field & \[
\begin{gathered}
\text { No. of } \\
\text { Twins/men }
\end{gathered}
\] & Grain boundary length/ \(\mathrm{cm}^{2}\) & No. of Dislocation Pits/field & \[
\begin{aligned}
& \text { No. of } \\
& \text { Dislocazion } \\
& \text { Pits/ } / \mathrm{m}^{2}
\end{aligned}
\] \\
\hline 55 & 5-743 A & 55.18 & 784.721 & 2.27 & 1423.18 & 0.040 \\
\hline 56 & 5-743 B & 59.27 & 843.002 & 0.94 & 1465.18 & 0.042 \\
\hline 57 & 5-743 C & 40.06 & 569.736 & 1.96 & 1376.75 & 0.039 \\
\hline 58 & 5-743 D & 61.62 & 831.500 & 2.00 & 1189.66 & 0.034 \\
\hline 59 & 5-743 E & 56.23 & 799.754 & 2.04 & 1532.03 & 0.044 \\
\hline 60 & 5-743 F & 43.17 & 613.955 & 2.72 & 1885.17 & 0.054 \\
\hline 61 & 5-743 G & 59.86 & 851.352 & 2.48 & 1458.14 & 0.041 \\
\hline 62 & 5-743 H & 43.67 & 621.122 & 1.76 & 2190.90 & 0.062 \\
\hline
\end{tabular}

\section*{TABLE 11}

ANALYSIS OF MOBIL TYCO SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline  &  & No. of Twins/field & \[
\begin{gathered}
\mathrm{No} . \text { of } \\
\text { Twins } / \mathrm{mm}^{2}
\end{gathered}
\] & Grain boundary length/ \(\mathrm{cm}^{2}\) & No. of Dislocation Pits/field & \[
\begin{aligned}
& \text { No. of } \\
& \text { Dislocation } \\
& \text { Pits/wm }
\end{aligned}
\] \\
\hline & (MRR \#1) & & & & & \\
\hline 63 & 5-640 B & 28.22 & 401.415 & 1.60 & 860.36 & 0. 024 \\
\hline 64 & 5-640 C & 24.98 & 355.260 & 1.40 & 1136.35 & 0.032 \\
\hline 65 & 5-640 D & 24.57 & 349.427 & 2.52 & 1072.13 & 0.030 \\
\hline 66 & 5-640 E & 23.74 & 337.584 & 2.39 & 2427.54 & 0.069 \\
\hline 67 & 5-640 F & 48.55 & 690.422 & 2.19 & 860.34 & 0.025 \\
\hline 68 & 5-640 G & 18.67 & 265.506 & 1.08 & 1434.89 & 0.041 \\
\hline 69 & \(5-640 \mathrm{H}\) & 45.92 & 653.085 & 0.59 & 1245.87 & 0.035 \\
\hline
\end{tabular}

ANALYSIS OF MOBIL TYCO SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|c|}
\hline  & \multicolumn{2}{|l|}{\[
\begin{gathered}
\text { JPL } \\
\text { Sample }
\end{gathered}
\]} & No. of Twins/field & \[
\text { No. of } 2
\]
Twins/mm & Grain boundary 2 length/ \(\mathrm{cm}^{2}\) & No. of Dislocation Pits/field & No. of Dislocation Pits/um \\
\hline & \multicolumn{2}{|l|}{(MRR \#2)} & & & & & \\
\hline 70 & 5-640 & & 20.21 & 287.431 & 1.92 & 976.38 & 0.028 \\
\hline 71 & 5-640 & B & 39.75 & 565.332 & 2.76 & 576.89 & 0.016 \\
\hline 72 & 5-640 & C & 24.08 & 342.460 & 2.60 & 685.26 & 0.019 \\
\hline 73 & 5-640 & & 22.14 & 314.947 & 2.19 & 850.34 & 0.024 \\
\hline 74 & 5-640 & E & 50.11 & 712.701 & 2.00 & 621.45 & 0.018 \\
\hline 75 & 5-640 & F & 30.21 & 429.695 & 1.55 & 842.35 & 0.024 \\
\hline 76 & 5-640 & G & 42.22 & 600.411 & 1.82 & 998.84 & 0.028 \\
\hline 77 & 5-640 & & 35.90 & 510.569 & 2.35 & 650.94 & 0.019 \\
\hline
\end{tabular}

\section*{IABLE 13}

ANALYSES OF MOBIL TYCO SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline \begin{tabular}{c} 
MRI \\
Sample \\
I
\end{tabular} & \begin{tabular}{c} 
JPL \\
Sample \\
I
\end{tabular} & Twins/Field & Twins/mm & \begin{tabular}{c} 
Grain \\
Boundary \\
Length
\end{tabular} & \begin{tabular}{c} 
Oislocation \\
Pits/field
\end{tabular} & \begin{tabular}{l} 
Dislocation \\
Pits/ \(\mu \mathrm{m}^{2}\)
\end{tabular} \\
\hline 78 & \(5-867 \mathrm{~A}\) & 32.7 & 834.18 & 4.85 & 1069.31 & .0545 \\
79 & \(5-867 \mathrm{~B}\) & 42.09 & 1073.76 & 0.76 & 494.86 & .0252 \\
80 & \(5-867 \mathrm{C}\) & 27.91 & 712.02 & 2.78 & 994.35 & .0507 \\
81 & \(5-867 \mathrm{D}\) & 50.58 & 1290.42 & 2.38 & 540.10 & .0275 \\
82 & \(5-867 \mathrm{E}\) & 26.19 & 668.19 & 3.43 & 542.68 & .0277 \\
83 & \(5-867 \mathrm{~F}\) & 41.77 & 1065.63 & 2.06 & 373.79 & .0197 \\
84 & \(5-867 \mathrm{G}\) & 24.27 & 619.21 & 3.37 & 635.78 & .0324 \\
85 & \(5-867 \mathrm{H}\) & 36.65 & 934.94 & 2.07 & 440.21 & .0224 \\
\hline
\end{tabular}

Note: Samples 78-93 were examined by the Vidicon camera with a calibration factor of \(.00028 \mathrm{~mm} / \mathrm{pp}\) and samples \(94-105\), and TYLAN \#1 were examined by the Plumbicon camera using a calibration factor of . \(000366 \mathrm{~mm} / \mathrm{pp}\).

\section*{IABLE 14}

ANALYSIS OF MOBIL TYCO SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline \begin{tabular}{c} 
MRI \\
Sample \\
\#
\end{tabular} & \begin{tabular}{c} 
JPL \\
Sample \\
H
\end{tabular} & Twins/Field & Twins/mm & \begin{tabular}{c} 
Grain \\
Boundary \\
Length
\end{tabular} & \begin{tabular}{c} 
Dislocation \\
Pits/field
\end{tabular} & \begin{tabular}{c} 
Dislocation \\
Pits/um
\end{tabular} \\
\hline 86 & \(5-867 \mathrm{I}\) & - & - & - & - & - \\
87 & \(5-867 \mathrm{~J}\) & 21.29 & 543.18 & 4.71 & 1221.77 & .0623 \\
88 & \(5-867 \mathrm{~K}\) & 32.92 & 839.89 & 2.03 & 615.54 & .0314 \\
89 & \(5-867 \mathrm{~L}\) & 19.38 & 494.43 & 4.34 & 735.97 & .0375 \\
90 & \(5-867 \mathrm{M}\) & 32.71 & 834.415 & 3.04 & 501.36 & .0255 \\
91 & \(5-640 \mathrm{~A}\) & 54.82 & 1398.61 & 2.76 & 362.87 & .0185 \\
92 & \(5-640 \mathrm{~B}\) & 22.81 & 581.86 & 3.68 & 537.89 & .0274 \\
93 & \(5-640 \mathrm{C}\) & 50.02 & 1276.11 & 1.97 & 163.81 & .0083 \\
94 & \(5-640 \mathrm{D}\) & 25.03 & 373.67 & 3.19 & 513.09 & .0153 \\
\hline
\end{tabular}

\section*{IABLE \(\quad 15\)}

ANALYSIS OF MOBIL TYCO SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline  & JPL Sample * & Twins/Field & Twins/mm \({ }^{2}\) & Grain Boundary Length & Dislocation Pits/Field & Dislocation Pits/ \(/ \mathrm{m}\) \\
\hline 95 & 5-640 E & 62.49 & 933.04 & 3.1 & 1182.93 & . 0340 \\
\hline 96 & 5-640 F & 28.92 & 446.3 & 5.15 & 999.09 & . 0298 \\
\hline 97 & 5-640 G & 51.70 & 771.92 & 3.4 & 641.73 & . 0192 \\
\hline 98 & 5-640 H & 23.39 & 360.98 & 4.38 & 886.58 & . 0264 \\
\hline 99 & 5-990 A & 36.02 & 537.85 & 2.54 & 867.54 & . 0259 \\
\hline 100 & 5-990 B & 30.39 & 453.74 & 3.52 & 1069,96 & . 0319 \\
\hline 101 & 5-990 C & 40.28 & 601.39 & 3.20 & 1396.13 & . 0417 \\
\hline & & & & & & \\
\hline
\end{tabular}

\section*{IABLE 16}

ANALYSIS OF MOBIL TYCD AND HONEYWELL SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline \begin{tabular}{c} 
MRI \\
Sample
\end{tabular} & \begin{tabular}{c} 
JPL \\
Sample \\
\#
\end{tabular} & Twins/Field & Twins/mm & \begin{tabular}{c} 
Grain \\
Boundary \\
Length
\end{tabular} & \begin{tabular}{c} 
Dislocation \\
Pits/Field
\end{tabular} & \begin{tabular}{l} 
Dislocation \\
Pits/um
\end{tabular} \\
\hline 102 & \(5-990 \mathrm{E}\) & 37.96 & 566.76 & 1.52 & 1219.67 & .0364 \\
103 & \(5-990 \mathrm{~F}\) & 35.62 & 531.78 & 4.21 & 765.96 & .0228 \\
104 & \(5-990 \mathrm{C}\) & 35.53 & 530.46 & 1.55 & 545.65 & .0162 \\
105 & \(5-990 \mathrm{H}\) & 40.01 & 597.29 & 4.36 & 745.66 & .0222 \\
1 \begin{tabular}{l} 
Honey- \\
Well
\end{tabular} & \(3=910\) & 25.55 & 387.53 & 4.21 & 425.61 & .0127 \\
\hline
\end{tabular}

\section*{IABLE 17}

\section*{ANALYSIS OF MOBIL TYCO SAMPLES}
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline MRI Sample: \(\#\) & \begin{tabular}{l}
JPL Sample \\
\#
\end{tabular} & Twins/Field & Twins/mm \({ }^{2}\) & Grain Boundary 2 Length \(/ \mathrm{cm}^{2}\) & Dislocation Pits/Field & \[
\begin{aligned}
& \text { Dislocation } \\
& \text { Pits } / \mu \mathrm{m}^{2}
\end{aligned}
\] \\
\hline 106 & 184-88 A & 53.30 & 795.85 & 2.06 & 1245.8 & . 0372 \\
\hline 107 & 184-88 в & 45.72 & 682.58 & 2,11 & 794.08 & . 0237 \\
\hline 108 & 184-88 C & 42.76 & 638.38 & 2.00 & 496.78 & . 0148 \\
\hline 109 & 184-88 & 47.06 & 702.67 & 1.82 & 1242.08 & . 0371 \\
\hline 110 & 184-88 E & 38.79 & 579.21 & 3.05 & 830.12 & . 0248 \\
\hline 111 & 184-88 F & 50.95 & 760.64 & 2.59 & 1175.68 & . 0.351 \\
\hline 112 & 184-88 G & 38.66.. & 577.21 & 2.99 & 1223.92 & . 0365 \\
\hline 113 & 184-88 H & 53.47 & 798.39 & 4.00 & 1206.46 & . 0360 \\
\hline
\end{tabular}

\section*{IABLE 18}

ANALYSIS OF MOBIL TYCO SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline MRI Sample \# & \begin{tabular}{l}
JPL \\
Sample \\
\#
\end{tabular} & Twins/Field & Twins/mm \({ }^{2}\) & Grain Boundary Length/cm & Dislocation Pits/Field & \[
\begin{aligned}
& \text { Dislocation } \\
& \text { Pits/ } / m^{2}
\end{aligned}
\] \\
\hline 114 & 184-175A & 70.18 & 1047.83 & 2.91 & 672.33 & . 0200 \\
\hline 115 & 184-175B & 58.13 & 867.96 & 2.0 & 890.19 & . 0266 \\
\hline 116 & 184-1750 & 53.97 & 805.84 & 2.09 & 1001.76 & . 0349 \\
\hline 117 & 184-175E & 53.46 & 798.25 & 1.57 & 1051.01 & . 0304 \\
\hline 118 & 184-175F & 54.79 & 818.05 & 2.03 & 1264.04 & . 0377 \\
\hline 119 & 184-175H & 57.00 & 851.09 & 3.05 & 681.88 & . 0263 \\
\hline
\end{tabular}
\(+\)

\section*{IABㅡ틍 19}

\section*{ANALYSIS OF MOBIL TYCO SAMPLES}
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline MRI Sample \# \(\qquad\) & \begin{tabular}{l}
JPL \\
Sample \\
\#
\end{tabular} & .Twins/Field & Twins/mm \({ }^{2}\) & Grain Boundary 2 Length/ \(\mathrm{cm}^{2}\) & Dislocation Pits/Field & Dislocation Pits/ \(/ \mathrm{mm}^{2}\) \\
\hline 120 & 5-1094-33A & 18.99 & 283.63 & 4.06 & 1863.86 & . 0556 \\
\hline 121 & 5-1094-338 & 29.13 & 435.05 & 3.61 & 703.59 & . 0210 \\
\hline 122 & 5-1094-33C & 8.49 & 126.89 & 3.54 & 1637.14 & . 0488 \\
\hline 123 & 5-1094-33D & 16.47 & 245.88 & 2.64 & 814.78 & . 0243 \\
\hline 124 & 5-1094-33E & 19.66 & 293.49 & 3.31 & 1228.40 & . 0367 \\
\hline 125 & 5-1094-33G & 31.19 & 465.81 & 2.8 & 762.40 & . 0227 \\
\hline 126 & 5-1094-33H & 21.42 & 319.86 & 3.33 & 927.35 & . 0277 \\
\hline
\end{tabular}

IABLE 20

ANALYSIS OF MOBIL TYCO SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline \begin{tabular}{l}
MRI \\
Sample \\
\#
\end{tabular} & JPL Sample & Twins/Field & Twins/mm \({ }^{2}\) & Grain Boundary Length/cm & Dislocation Pits/Field & \[
\begin{aligned}
& \text { Dislocation } \\
& \text { Pits } / \mu m^{2}
\end{aligned}
\] \\
\hline 127 & 5-1092-69A & 51.80 & 773.53 & 4.11 & 777.55 & . 0232 \\
\hline 128 & 5-1092-698 & 26.11 & 389.82 & 3.10 & 890.47 & . 0266 \\
\hline 129 & 5-1092-69C & 47.08 & 702.97 & 3.57 & 622.08 & . 0186 \\
\hline 130 & 5-1092-690 & 29.25 & 436.79 & 2.74 & 579.24 & . 0173 \\
\hline 131 & 5-1092-69E & 33.59 & 501.52 & 2.72 & 936.01 & . 0279 \\
\hline 132 & 5-1092-69F & 30.05 & 448.60 & 2.93 & 982.41 & . 0293 \\
\hline 133 & 5-1092-696 & 44.96 & 671.28 & 1.92 & 381.26 & . 0263 \\
\hline 134 & 5-1092-69H & 37.63 & 561.89 & 2.96 & 834.82 & . 0249 \\
\hline
\end{tabular}

\section*{TABLE 21}

ANALYSIS OF IBM SAMPLES
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline MRI Sample No. & JPL No. & Avg. No. of Twins/ field & Avg. No.gf Twins/mm & Grain boundary length/ \(\mathrm{cm}^{2}\) & Avg. No. of Dislocation Pits/field & Avg. No. of Dislocation Pits/ \(/ \mathrm{mm}^{\prime}\) \\
\hline 1 & 4-457 A & 43.70 & 621.581 & 1.3 & 460.56 & 0.013 \\
\hline 2 & 4-457 8 & 41.30 & 587.374 & 1.5 & 205.37 & 0.006 \\
\hline 3 & 4-457 C & 39.96 & 568.254 & 1.12 & 373.20 & 0.011 \\
\hline 4 & 4-457 D & 37.25 & 529.826 & 0.51 & 302.98 & 0.009 \\
\hline 5 & 4-457 E & 29.82 & 424.114 & 0.52 & 328.91 & 0.010 \\
\hline 6 & 4-457 F & 38.09 & 541.730 & 1.33 & 405.75 & 0.012 \\
\hline 7 & 4-457 G & 15.79 & 224.585 & 1.5 & 342.75 & 0.010 \\
\hline & & & & & & - \\
\hline
\end{tabular}

\section*{AVALYSIS OE MOTOROLA SAMLES}


Table 23
ANALYSIS OF MOTOROLA SAMPLES


\section*{ANALYSIS OE MOROROLA SAMPLES}
("


TABEE 25
\(\cdots\)
ANALYSIS OF MOTOROLA SRMFEES

-90-
```

IABLEE 26

```

```

G REM*******I.L NATA IS DUTFUT FOK STIRAOL UN F{IES[D(1:)************
7 REM
8 DIM T(1000)
9 FRINT 'DEFECTS JN SILICON(UERSION シ"\&/I/79)'
10 PRINT 'HEADING*\FRINT
11 RNFUIT H\$
15 PRINT "FRINT FIL.E MAME FOR STONACE OF DATA(IXI:NANKE)'
1 6 ~ P R I N T ~ T
17 INPUT A\$
18 OPEN AS FGR OUTFUT AS FILE \#1
22 PRINT "OFERATOR.
23 PRINT
24 INPUT O\$
30 FRINT •MAGNIFICATION'
31 PRINT
32 JNFUT M\$
40 PRINT "UNITG"
4 1 PRINT
42 INFUJT U\$
50 PRINT 'CALIBRATIINN FACTOR(UNITS/FF')'
PRINT
INPUT
PRINT "FFAKE AR'EA(F'P)'
PRINT
INFUT R
FRINT -gTM gutfut mata mividej my.
PRINT
INPUT X
PRINT 'AVERAGE FEATURE AREA(F'F')'
PRINT
INFUT E
PRINT \#1:'DEFECTS IN SILICON(UERSION 3-B/1/75)'\FRINT \&1:
PRINT \#1:H$\FRIN'T #1:
        PRINT #1:"IPERATOR IS ":0$;" MAGNIFICAIION=";M\$
PRINT \#1:"UNITS= ";U\#;" CALIBRATION FACTOR (UNITB/FF')=';C
89 PRINT \#1:'FRAME AREA=';R''' QTM OUTFUT WAS RKUINEI R:';X;'ANG CORRECTED'
90 PRINT \#1:'AUEFAGE FEATURE AR'FA (FF)="!E
9I FRINT \#1:
G5 PRINT \#1:'FL.D NO. NO./AREA MFFU MFF'H L/A'
96 PRINT \#1:'(A,P,UF,HP)"
100 FRRNT 'FLI NO, NO,/AREA MFFU MFFH L/A.
LOL PRINT '(a,P,UP,HP)'
10O KEM
IOT REIG GTM MEASUREMENT ROUTINE
108 REM
109 CALLL 'CIFI"
110 CALL 'STRT'(%,4,'FTFI/CIF/FCI/FC2')
*112 CAlLL 'CIFW'('ACO,')
114 CALL CCIFW'('AE4,')
120 CALL 'STEP'(1,'FIFT=FLD/FCI=A/FC2=A')
130 CALL 'STEF'(2,'FC2=F'4)
140 CALL 'STEP'(3,'FC.2=UF')
150 CALL 'STEF'(4,'FC2=HF')
150 INFUT Es
161 IF Fis'0' THEN S:0 \IF F=0 THEN 104

153 FEINT \#1:('iA;FiviH:')'
(154 IF $\mathrm{BS}=\mathrm{A}^{\prime} \mathrm{A}^{\prime}$ THEN 700
16E IF Es='END' THEN Oq9

170 CALL ${ }^{\prime \prime}$ FLD'(A,F,V,H)
130 CAILL 'CIFM'('Ald,')
$190 \mathrm{FaF+1}$
200 AuX*A $\backslash F=F: W \backslash \backslash U=U * K \backslash \mid H=H * X$
209 REM
210 REM Calcul.ation routine
211 REM
$220 \mathrm{NaA} / \mathrm{E}$
230 GN/R/C/C
235 TF $V=0$ THEN 250
236 MI=R:KEN
240 IF $H=0$ THEN 2SS
$242 \mathrm{MRIFHC/H}$
243 10 TO 2.50
250 LET M1=0\G0 TO 240
25E I.ETT M2=0\00 TO
$260 \mathrm{LaF} / 2 / \mathrm{R} / \mathrm{C}$
270 N1 $=N+N 1 \backslash G 1=\{j+6 \backslash \backslash L 1=L+L 1$
$275 \mathrm{~N}_{2}=\mathrm{N} * N+\mathrm{N} 2 \backslash \mathrm{G} 2=G * G+$ 「2 $2 \backslash \mathrm{~L} 2=1 . * L+L 2$
$280 \mathrm{H}=\mathrm{H} 1+\mathrm{H} \backslash \mathrm{V}=\mathrm{V}=\mathrm{V}$
499 REM
500 REM FRINT QUT REEGULTS
501 REM
S30 FMINT FIN;G,M1,M2,L
SE1 FRINT (';AFBU:H;')"
550 10 TO 1610
599 REM
600 RIIM IELETE LAST FIELD
601 REM
610 N. $=N_{1}-N \backslash N_{2}=N 2-N * N$
615 G1=G1-G\G2=G2-G*G
$620 \quad 1.1=L 1-L \backslash L 2=L 2^{\prime}-L$. $* L$
$625 \quad F=F-1 \backslash H t=H 1-H \backslash U t=U 1-U$
GJO FRINT 'LAST FJELI DELETEII'
635 INFIJT $\mathrm{B} \$$
640 60 TO 154
699 REM
700 fiLi ******AUERAGE,SIISE*********
701 REM
710 L.ET $2.1=N 1 / F$
720 LET Z2=G1/F
730 IF U1=0 TH-N 750
735 LET ZJ=F****C/U1
740 IF H1=0 THEN 755
$745 \quad 24=F * R * C / H 1$
746 150 TO 760
750 LET Z3=0\60 TO 740
755 I.ET $24=0$
750 LET ZS=L1/F
770 I.Er $\quad I=N 2 / F-7.1 * Z 1$
780 LET SI=SUR(LI)

```
    781 LET EI=S1/(SUR(F))
    790 LET D=G3/F-22KZO\IF (1) THEN BO1
```



```
    801 LE'r S2=0\E2=0
    810 I.ET D=L2/F-25*Z5
    811 [F D<OO THEN 321
    G20 LET SE=SQR(GI)\E5=SS/(SUR(F))\GO 10 BSO
821 LEFT S5=0\E5=0
```



```
851 FRINT NO. NO./AREA NFFU NFFHH LA.
852 PRINT *1:" ********AUERAGEE&*********
85J PRINT #1:0 NO. NIJ,/AREA MFFU
860 FRINT ( 'i%1,z2,Z3,7.4,Z5
861 PRINT #1:' 1;71,22,23,Z4,Z5
870 PRTNT 'SR';S:1,S2,,!S5
871 PRINT #L:'S[1;S!,S2,.,S5
880 PRINT 4SE'fEL,Ez,,DES
381 PRINT #1:'SE';E1,ER,,,EE
998 INFITT Es
900 60 50 164
999 ENB
```

* 



OFERATOK IS $\ddagger$ IM MAGNIFICATION=800
UNITS: MY CALIFRATION FA(YTITK (UNITS/FF) $=3.6 .5000 E-04$ FRAME AREA= 500000 RTM JUTFIUT WAS IIUIIEI EY TOO ANII CORRECTEI AUERAGE FEATURE AREA (FF)=2BO1


Table 28 (contd,)


Table 28 (contd.)

*TT: = DX1:MT100LI.DAT
DEFECTS IN SILICON(UEFSION 3-8/1/77)
MFI 100 JFL $5-990$ SPEC $B$ AFEA. 98 MMOEI TYOU UISLDCATIONS ONLY
OFERATOR IS TIM MAGNIFICATIUN=800
UNITS= MISRONS CALTEFATTON FACTOR (UNTTG/FF)=.366 FFAME AFEA = 2500NO QTM OUTFUT WAS IITUTDEI EY 1 ANL COFRECTEI AUEFAISE FEATURE AREA $\langle F F\rangle=10.6$

FLI NO.
NO. /AFEA
MFFU
(A,F,VF,HF)
$1970.5668 .07925 E-03$
$110.909 \quad 90.2367 \quad .0170929$
(2868 3128 825 1014)
254.9811 1.70149E-03
455.224
448.594

 ( 12041361407403 ) 4 237.83 7.10174E-03

224,916
227.047
7.43716E-03
(2521 2455 707 763)
129:42
$119+931$
.0134 .153
51950.75 . 0582506
18.3957
MFFH
90.2367
448.594
227.047
119.921
17.6641

L/A

Table 28 (contd.)


Table 28 （contd．）

| 321 | 1.13208 | 3．38044E．О泞 | 45750 | 13071．4 | 1．58470E－04 |
| :---: | :---: | :---: | :---: | :---: | :---: |
| （12 | 292 | 7 ） |  |  |  |
| 336 | 689，245 | ． 0205812 | 48.2341 | 54.2062 | ． 0317978 |
| （ 7306 | 65819 | 1897 1688） |  |  |  |
| $34 \%$ | \％06．792 | ．0151351 | 60.4362 | 60．1578 | ． 02338385 j 2 |
| （ 5372 | 25286 | 1509 1521） |  |  |  |
| 353 | 344．528 | ． 0102878 | 78.8793 | 82． 61553 | ．0211038 |
| （ 3652 | 23852 | 1160 1107） |  |  |  |
| 365 | 5095，19 | ．152145 | 15.154 | 14.8106 | ． 109721 |
| （5400 | 092007 | 796038 6178 | ） |  |  |
| 375 | 5326.98 | ． 159067 | 13.6853 | 12.3548 | .127393 |
| （ 5346 | 66 2331 | 3 6636 7406 | ） |  |  |
| 38 9 | 980．566 | ． 0292802 | 31．1205 | 28．5759 | ． 0564262 |
| $(1039$ | 941032 | 6 29403202 | ） |  |  |
| 393 | 2612．45 | ． 0780093 | $14+1612$ | 13.1787 | ． 11776 |
| 2769 | 92 2155 | 5 641．5 5943 | ） |  |  |
| 402 | 2614.91 | ． 0790035 | 17.2968 | 16.1205 | ． 0937596 |
| （ 2771 | 191715 | 585290567.5 | ） |  |  |
| 412 | 2856．51 | ． 085397 | 16．5551 | 15.8799 | ． 102432 |
| ＜ 3027 | 791874 | 555275762 | ） |  |  |
| 423 | 3101.23 | ． 0926043 | 13.5135 | 13.3382 | ． 122038 |
| －3237 | 73 2233 | 3367716860 | ） |  |  |
| 432 | 2443.87 | ． 0729752 | 17．6335 | 15.3652 | ． 10000.1 |
| 2590 | 051830 | 0251895955 | ） |  |  |
| 44. | 1．198．77 | ． 035796 | 27．8624 | 27.071 | ．0583\％76 |
| 1270 | 071073 | 3832843380 | ） |  |  |
| 456 | 607．453 | ． 0181389 | 61.900 | 59.6869 | ． 0275956 |
| （ 6437 | 75050 | 1478 1533） |  |  |  |
| 462 | 25，9434 | 7．74684E－04 | 1039.77 | 582．836 | 1．86339E－03 |
| ＜ 275 | 3418 | 8 134） |  |  |  |
| 475 | 58.1132 | 1．73529E－03 | 933．673 | 183゙393 | 7．97814E－（14 |
| （ 616 | 1459 | 8 56） |  |  |  |
| 48. | ．8ヶ9057 | 2．53533E－05 | 13300 | 7625 | 4．6．4481E－04 |
| （9）8 | 8551 | 2） |  |  |  |
| 490 | 00 | 0 | 0 | 0 |  |
| （ 00 | 000 | ）． |  |  |  |
| 50 | 581．415 | ． 0173614 | 75．5579 | 64.5275 | ．0238251 |
| （ 6163 | 34350 | 1211 1418） |  |  |  |
|  |  | ＊＊＊＊＊＊＊＊AUERAO | E\％＊＊＊＊＊＊＊ |  |  |
|  | NO． | NO．／AKEA | MFFV | MFFH | L／A |
|  | 69．96 | ． 0319497 | 38.743 | 37.659 | ． 043001 |
| S［135 | 53.36 | .040412 |  |  | ．0453395 |
| SE 191 | 1.393 | 5．715．2E－03 |  |  | 6．41197E－03 |
| ＊TT：$=$ | DX1：KT10 | $1 T . D A T$ |  |  |  |
| IEFECTS IN SILICON（UEFSION 3－8／1／79） |  |  |  |  |  |

MRI 101 JFI 5－990 GFEC C MDEJI．TYCO AREA ， 90 TMINS ONLY
OFERATOR IS TIM MAGNIFICATION＝800
UNITS＝MM CALIEFATION FACTOF $(U N T T S / F F)=3.66000 E-04$
FFAME AREA $=500000$ RTM SUTFUT WAS IIIUTDEI FY 100 ANH EORGECTEO AVEFAGE FEATURE AFEA（FF）＝2453

| $\begin{aligned} & F L[1 \quad N O \\ & \left(A, F^{\prime}, \cup F, H F^{\prime}\right) \end{aligned}$ |  | NO，$/$ AREA |  |  | MFF＇V | MFFH | L／A |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | $(A, F, \cup F, H F)$ |  |  |  |  |  |
| 1 | 18，7525 | 279．781 |  |  | ． 0703846 | ． 0244 | 49．7368 |
| $(4$ | 00018 | 02500 | 7500 | ） |  |  |  |
| 2 | 56.8284 | 848.463 |  |  | .0631034 | 9.891 | 1098，197 |


[^0]:    *Mobil Tyco \# 53, JPL 145-7E, 5-745, SPEC. G.

[^1]:    Figure 12. IBM \#6 - Section 1 - Side 2, micrograph of ribbon surface, shown earlier in Fig. 11, after a 30 second etch by Etching Solution II. Mag 200X

[^2]:    Figure 14. IBM 46 - Section 1 - Side 2, micrograph of

[^3]:    Figure 19A - Mobil Tyco \# 53 - Field \# 1 Photograph from QTM screen with an area being circled by the light pen.

    Mag. 800X

[^4]:    Figure 19B-Mobil Tyco \# 53 - Field \# 1
    Photograph from GTM display screen showing a small reqion that has been rejected.

    Mag. 800X

[^5]:    Figure 19C-Mobil Tyco \# 33 - Field \# 1
    Photograph from gmm dizplay screen showing only the twins. The dislocation pits have been rejected.

    Mag. 800X

