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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

QUARTERLY PROGRESS REPORT NO. 2

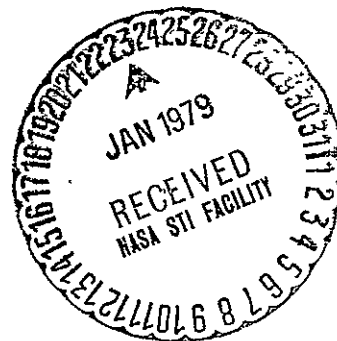
by

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Covering the period 1 June 1978 to 30 September 1978

JPL Contract No. 954977

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This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, under NASA Contract NAS7-100 for the U. S. Department of Energy, Division of Solar Energy.

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SECTION 1

SUMMARY

This report describes the various steps involved in obtaining quantitative information of structural defects in crystalline silicon samples. The report discusses the following procedures: 1) chemical polishing; 2) chemical etching; 3) automated image analysis of samples on the QTM 720 System; and 4) preliminary discussion of data.

SECTION II

INTRODUCTION

The objective of this program is to evaluate and, if possible, predict the conversion efficiency for a variety of silicon samples with differences in structural defects, such as grain boundaries, twin boundaries, precipitate particles, dislocations, etc. It is important to know the type of defect/defects, and the concentration at which such defect/defects severely cause deterioration of conversion efficiency. Quantitative data of this nature may then enable us to predict the performance of silicon samples before they are fabricated into solar cells. Also, such a technique may, in addition, be used routinely as a Nondestructive Quality Assurance Tool to rapidly and routinely evaluate and survey a variety of silicon samples with differences in growth, fabrication, and processing variables.

Quantitative analysis of surface defects will be performed by using a Quantimet 720 Quantitative Microscopy System. This system can differentiate and count 67 shades of gray levels between black and white contrasts. In addition, it can characterize defects (such as dislocations, twin boundaries, precipitates, grain boundaries, etc.) by measuring their length, perimeter, area, density, spatial distribution, frequency distribution (in any preselected direction), and so on. However, the Quantitative Microscopy System 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 chemical cleaning and polishing technique has now been perfected, and this technique is now routinely used. The cleaning and polishing preparation technique produces a very clean and even surface for silicon crystals suitable for analyses by the QTM 720 System. We have just started obtaining quantitative information from a variety of silicon crystals, and these will be reported in due course.

SECTION III

CHEMICAL POLISHING

In our previous Quarterly Report¹, the chemical polishing technique was briefly explained. Since the above work was reported, the silicon surfaces were subsequently examined in a QTM 720 Quantitative Microscopy System. It was found that further refinements in polishing will have to be done in order to obtain surfaces suitable for QTM 720 analysis. This work has now been completed and will be described herein.

For the present study, two types of samples e. g., those manufactured by Wacker and IBM were used. It was observed that the time required to polish the two types of samples varied by a small amount; however, this variation is important for QTM 720 analysis and, therefore, should be understood and used carefully in practice. The variation in polishing time is due to certain inherent manufacturing/fabrication/processing variables, which affect the reaction rates for the two types of samples under identical polishing conditions. Similar refinements in polishing and etching steps will have to be perfected and used, when silicon crystals manufactured by other manufacturers are used in future. These will require additional development time and effort.

Polishing of Wacker and IBM groups of samples were performed at different combinations of temperatures and times. The results are summarized in Tables 1 and 2 respectively for Wacker and IBM samples. It may be observed from these tables that for Wacker samples, polishing temperature of $50^{\circ} \pm 3^{\circ} \text{C}$ and polishing time of 80 to 85 sec. is acceptable. For IBM samples, polishing temperature of $50^{\circ} \pm 3^{\circ} \text{C}$ and polishing time of 85 to 90 sec. is acceptable.

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 cannot be used to determine the quality of polishing. However, when the same samples are observed in a high quality optical metallograph, the underpolished samples show faceting and subgrain-type structure; whereas the well-polished samples show clearly defined grain boundaries and some of the twin boundaries in sharp contrast. Therefore, optical metallograph must be used to determine the quality of polishing. Because of the presence of subgrain-type structure in underpolished samples, it is better to slightly overpolish a sample by a few seconds. This will insure good quality surface and reproducibility from sample to sample.

SECTION IV

CHEMICAL ETCHING

COMPOSITIONS OF ETCHING SOLUTION:

The main objectives of etching a polished silicon sample are:

- 1) to reveal grain boundaries in sharp contrast;
- 2) to reveal clearly defined twin boundaries which do not touch or overlap one another,
- 3) to reveal dislocation etch pits which do not touch or overlap one another.

A variety of etching solutions, which are commonly used in the semiconductor industry were tested on JPL-supplied silicon samples. After considerable testing and evaluations, it was observed that Sirtl etch produced good quality surfaces when observed under QTM 720 System. Therefore, Sirtl etch has been selected to etch the silicon ribbon samples. The etching solution that has been developed is a diluted variation of the Sirtl etch. Composition of the Sirtl etch is as follows:

Solution A

50g CrO₃:100 ml deionized water

Solution B

HF
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 and also shown in Table 3.

ETCHING SOLUTION I:

The first variation from the Sirtl etch was prepared by dissolving 20 grams of CrO₃ 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 1 shows the structure of an IBM silicon ribbon after chemical polishing. Figure 2 and 3 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 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 4).

ETCHING SOLUTION II

The second variation from the Sirtl etch was prepared by dissolving 10 grams of CrO_3 in 40 ml of deionized water, and adding an equal volume of concentrated HF.

Figure 5 is a photomicrograph of the chemically polished surface. Figure 6 is a photomicrograph of the same surface after 30 second etch by Etching Solution II. Figure 6 shows all dislocations, twin boundaries and grain boundaries present in the sample. Variations in contrast of dislocations observed is due to focusing on a slightly curved surface.

The silicon surface in Figure 6 was etched for an additional 30 seconds. This resulted in deeper etching of dislocations and overlapping of twin boundaries (Figure 7). An additional 30 second etch (i. e. a total of 90 seconds) on the same surface resulted in significant overlapping of dislocations and twin boundaries. (Figure 8).

ETCHING SOLUTION III

The third variation from the Sirtl etch comprises 10 grams of CrO_3 in 60 ml of deionized distilled water; and an equal volume of concentrated HF.

Figure 9 is a photomicrograph of a chemically polished silicon surface. Figure 10 is a photomicrograph of the same area after 60 seconds etch by Etching Solution III. Figure 11 is another etched area on the same surface.

The etching treatment by Etching Solution III will result in an optical resolution of 10^{-4} cm for twin boundaries and an optical density resolution of 10^7 dislocations per cm^2 . A higher resolution, however, can be achieved if a higher magnification is used for observation.

The Etching Solution III has also been used to etch chemically polished Wacker samples. Figure 12 is a photomicrograph of chemically polished silicon surface of Wacker sample. Figure 13 is a photomicrograph of the same area after 50 seconds etch by Etching Solution III.

It has been observed on many silicon surfaces that an optimum etching time of approximately 50 seconds by Etching Solution III is sufficient to distinctly reveal grain and twin boundaries and dislocations. Both IBM and Wacker samples were etched with Etching Solution III. High quality defect structures, without overlapping and without wide variation in contrast of each defect type, were always obtained.

ETCHING PROCEDURE:

The silicon sample is placed into a teflon beaker with the surface to be etched facing up. Enough freshly prepared etching solution is poured so as to cover sample surface with a 5 mm head of etching solution. The test sample is left in the etching solution until desired contrast is achieved. Etching rates may vary slightly depending on the internal energy associated with the various types of defects, and the manufacturing/fabrication/processing variables associated with each sample type. The etching reaction was quenched using a volume of deionized water twice that of the etching solution. The test sample was removed from the etching solution, and it was immersed in another beaker containing a 5% NH_4OH solution for 30 seconds or less. The sample was then immersed in deionized water for 5 minutes. The sample was then rinsed in pure ethyl alcohol and the surface was dried by blowing Nitrogen gas.

(Note: The purpose of using NH_4OH solution is as follows. Even a small amount of residual HF left in the sample can attack and corrode the very expensive objective lenses of the metallograph or QTM. Therefore trace amounts of any left-over HF should be removed by immersing the sample in NH_4OH . Longer than 30 seconds exposure to NH_4OH will result in etching of the sample by NH_4OH and, therefore, should be avoided.

INPUT MATERIALS:

Deionized water
Trichloroethylene
Acetone
Ethyl alcohol
Cotton balls
Nitrogen gas

Apiezon Wax (W)
Hydrofluoric acid
Nitric acid
Acetic Acid
Chromium trioxide (CrO₃)
NH₄OH solution

EQUIPMENT AND FACILITIES

Fume hood
Baking oven capable of reaching 125°C
Air Brush
Hot plate
Rubber gloves
Face mask
Pyrex beakers; teflon beakers and tweezers

TWO - STEP ANALYSIS OF SAMPLES:

The etching technique, as described above, produces surfaces which are quite satisfactory when observed under an optical metallograph, but not entirely satisfactory and reproducible when observed under the QTM 720 system at a magnification of 800X. It was found that many dislocation etch pits were touching the grain and twin boundaries. This overlapping of defects creates problems for separate detection and measurement of twins, grain boundaries, and dislocation pits. The problem was serious, especially in the areas where the twin density was high. Therefore, effort was made to prevent such association whereby dislocation pits were joined with twin or grain boundaries. One way of achieving this goal was to etch silicon samples for a shorter time to develop and bring in sharp contrast only grain boundaries and twin boundaries, but not dislocation etch pits. This is possible, since etching rates vary slightly depending on the internal energy associated with each types of defects.

Several samples were then etched for different time durations ranging from 5 seconds to 30 seconds, and examined in the QTM 720 system. It was found that an initial 15 second etch was required to distinctly etch only the grain and twin boundaries, and produce a good contrast for detection and measurement with the QTM 720 system. An additional 35 seconds etch brings the dislocation pits in sharp contrast. Therefore, these two different etching times (separately for grains and twins on the one hand, and dislocation pits on the other) require the silicon samples to be analyzed in two separate steps as shown below:

Analysis of Defects on the QTM 720		
Defect Types	First Step	Second Step
	Chemically polished and 15 seconds etching	Same Sample with additional 35 second etch
Grains	Counted	not counted
Twins	Counted	not counted
Dislocation etch pits	not counted	counted

SECTION V

AUTOMATED IMAGE ANALYSIS

Preliminary measurements have been made on grains, twins, and dislocation pits of silicon samples from different manufacturers as reported earlier². These measurements were performed with a field area of about 0.46mm^2 and included: 1) the number of features and their area density, 2) mean free path between features (both horizontally and vertically), 3) the length of features per unit sample area, and 4) the average feature area.

We have now accomplished performing these measurements automatically while scanning the entire sample surface (fields for measurement are chosen at fixed increments in a square array). This automated analysis is accomplished by using the Programmer, Automatic Stage, and Automatic Focus optional modules in the QTM 720 System. Those allow the basic measurements (area, perimeter, vertical projection, horizontal projection, and count) to be made on each field, followed automatically by scanning to the next field, then focusing, and then performing the measurements again. The basic measurements from the QTM 720 are transferred to a Hewlett-Packard Model 9810 programmable calculator (HP9810) by way of a Field Data Interface module (optional QTM module). A block diagram of the system is shown in Fig. 14.

A program (on magnetic card) has been written for the HP9810 to convert the basic QTM parameters into the desired measurements for each field and print out the results on a standard teletype. In addition, the average of the data (based upon all previous fields) may be obtained at any time during a sample run by accessing a subroutine in the program. The HP9810 program and the storage register information is given in Appendix A.

PROCEDURE FOR RUNNING PROGRAM

1. Select proper objective for desired magnification.
2. Adjust optics for "Kohler illumination," following steps in Reichert Microscope Manual, if necessary. It is important that the field of view be uniformly illuminated.
3. Adjust 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. Sensitivity should read 0.4.

4. Place sample on a blank field of view and perform shade correction, setting the RANGE at about 10-11 o'clock. This is an important step. Be certain that entire standard frame can be detected uniformly.

(Light sensitivity switch must be in AUTO to perform shade correction.)

5. Place sample at origin of scan. This will be the lower left-hand corner of the sample. Make certain that sample is firmly held to 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. Any time you wish to have manual control of stage, switch from AUTO to MANUAL. When returning to AUTO mode, stage must be at ORIGIN. Always set ORIGIN after pushing AUTO. At this time, set 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 pp) will be the frame area called for at the beginning of the program.

7. Set proper detection of the features in the field using the "flicker method" and the 1-D Standard Detector Module.

8. The Standard Computer MS-3 should be switched to PATTERN RECOGNITION. Function Computer #1 will generally be set to AREA if area sizing is being used on the Classifier-Collector Module. Function Computer #2, the Classifier-Collector, and the Field Data Interface Modules should be set to AUTO. The Form Separator Module should be OFF. The Programmer Module should be set to STOP and AUTO/0-15. The Programmer board on left should be setup according to Fig. 15.

SETTING UP THE HP9810 DESK CALCULATOR TO RUN THE QTM720

1. With the appropriate magnetic card in hand, push RUN-END-LOAD on the HP9810 and load side A of magnetic card followed immediately by side B.
2. Push RUN-END-CONTINUE to begin Program. The HP9810 will print out on its tape "Magnification?" Type in the objective being used. It will

then ask for the calibration factor in whatever units/pp are being used. Later, you will type in the units on the teletype sheet which records the results. The frame area (in units of pp) is the product of the horizontal and vertical settings of the variable frame. If the Standard Frame is used, the frame area is 500,000 pp. (Note: After responding to each command on the HP9810, push CONTINUE).

The teletype will type out "Units -" and the operator types in the appropriate response. Push CONTINUE on the HP9810 and the heading will be typed out. Note that all units will be in those specified by the calibration factor. In the example of Fig. 1, the frame area is in units of mm^2 .

The meaning of each heading on the teletype printout is described below:

Field: The number of the field.

NO: The absolute number of features detected in the field.

NO./AREA: The number of features detected per unit sample area. (in the example, number of features/ mm^2).

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

MFPH: Mean free path (Horizontal): This is the horizontal analogue of MFPV.

L/A: Length (L) of twins per unit area (A) of sample. In the example, it is in units of mm/mm^2 . This quantity is equal to $(\text{perimeter}/2) \div \text{frame area}$, where the perimeter is the total detected feature perimeter. As long as the length \gg width of the feature (true for twins), $\text{perimeter}/2$ is approximately equal to the length.

Note: In the case of dislocation pits,

$$\frac{1}{\text{NO. / AREA}} \cdot \frac{L}{A} = \frac{P}{2A} \cdot \frac{1}{\text{NO. / AREA}} = \frac{P}{2(\text{NO.})}$$

$$\frac{2\pi \bar{r}(\text{NO.})}{2(\text{NO.})} = \pi \cdot \bar{r}$$

$$\bar{r} = \text{mean pit radius} \approx \frac{1}{\pi} \left(\frac{L}{A} \right) \left(\frac{1}{\text{NO. / AREA}} \right)$$

AFETA: Average feature area. This quantity is the total detected feature area in the field — the number of detected features. Note that for dislocation pits,

$$\bar{r} \approx \sqrt{\frac{\text{AFETA}}{\pi}}$$

When the HP9810 screen goes blank, it is ready to accept data from the QTM.

Push STEP on the QTM Programmer Module. The following QTM measurements on the detected features will be made (in order): Area, perimeter, vertical projection, horizontal projection, count. As these measurements are made they are printed out (in units of pp) on the HP9810 immediately following the FIELD number. Following all measurements the results for that field are typed out on the teletype in the specified units. The QTM automatically steps to the next field and focusses on it. The operator then examines the new field and performs the appropriate image editing or detecting. Do not change the variable frame size. It must remain constant throughout the run. Note that the HP9810 screen goes blank, signifying it is ready to accept QTM data for the new field.

DELETING DATA

If the last field measured is determined to be in error for any reason, the operator may perform the following key sequence on the HP9810. STOP, GO TO, LABEL, D (for Delete), CONTINUE. The teletype will respond immediately with:

LAST FIELD DELETED

AVERAGING DATA

Averaging of the data can be performed at any time by the following HP9810 key sequence. STOP, GO TO, LABEL, A (for average), CONTINUE. This will not affect data determined in the last field and is always an average of all of the data accumulated from field number one through, and including, the last field.

SECTION VI

PRELIMINARY ANALYSIS OF DATA

In the Mobil Tyco and IBM samples, almost all the twin boundaries are parallel to the longitudinal axis of the silicon ribbon. In the 1 inch x 1 inch samples examined in the QTM, these twins were found to run from one edge of the specimen to the opposite edge. There are few grain boundaries in each sample, and these grain boundaries are approximately parallel to the twins. Therefore, only a small number of twins intersect the grain boundaries. Therefore, counting of these defects by QTM is not too tedious. However, in the Wacker samples there are larger number of grain boundaries in each of the 1 inch x 1 inch samples. Moreover, the twins within different grains are oriented in different directions (not parallel to one another as in IBM and Mobil Tyco samples). To further complicate the counting of these defects, all the twins intersect the grain boundaries, and there are large number of such intersections in each field of view. The QTM counts all features which touch one another as one feature. As an example, if one grain boundary is touching several twins these will be counted as one feature. Therefore, each of the twins which touches a grain boundary must be made to separate on the TV screen image. This is done by manually using a light pen. This becomes very tedious, since each field of view on the TV screen must be manually edited quite extensively. Because of these complications, Wacker samples were chosen as the first ones to be analyzed by QTM. It was felt that if Wacker samples could be successfully analyzed, the other samples would be easier to examine by QTM.

Wacker sample No. 7 was the first sample to be analyzed on the QTM. This sample had a surface area of 40.32 mm^2 . As shown in Table 4, 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 magnification

of the image on the QTM screen was 311.2 X using a 12.5 objective. A calibration standard was used to calibrate the TV screen, and it was found that 0.4 mm was equal in length to 415 picture points (pp). In terms of area, $0.16 \text{ mm}^2 = 172, 225 \text{ pp}$. The total area of the image on the TV screen was 500,000 pp, which represented an area of 0.46 mm^2 on the specimen surface. For 50 fields this represents a total area analyzed by QTM of 23.23 mm^2 of 57.6% of specimen surface. Table 4 also shows average values (listed as AVERAGE), standard deviation (SD) and standard error (SE). The values are for all the data points preceding such listing. In other words, the data for Average, SD, SE at the bottom of Table 4 is for all the 50 fields.

The data in table 4 was analyzed statistically to get an idea of the minimum number of fields to be analyzed for precise measurements. The minimum value is that which gives a mean within 95% confidence (two standard deviations) of the mean determined from the 50 fields. Length of twins/unit area (L/A) in Table 4 was chosen as the test measurement, and was grouped into 2 mm class intervals. These are shown in seven histograms in Fig. 16 where the length of twins/unit area is plotted against 50, 25, 17, 13, 10, 10, and 10 fields. The data for the above fields were picked uniformly from the 50 fields listed in Table 4. It may be observed from the Figure 16, that the principal mode in all the cases as the fields are decreased from 50 to 10 lies in the 2 to 4 mm class. The distribution may be considered unimodal to a first approximation.

The arithmetic mean (\bar{X}), variance (σ^2), and standard deviation (SD) were calculated for each group of data. These are given in Table 5 and also listed to the right side of the graphs shown in Fig. 16. Figure 16 also shows the range of mean (\bar{X}) at 95% confidence level calculated by using the formula:

$$\bar{X} \pm t_{0.975} \left(SD / \sqrt{N-1} \right)$$

where N = Number of fields, and
 $t_{0.975}$ represents the 97.5 percentile value.

It indicates that if the data is meaningful and significant at 95% confidence level, the value of the true mean (\bar{X}) must lie within the range of the calculated mean shown for each of the graphs in Fig. 16. As the number of fields decreases from 50 to 10, the mean (\bar{X}) in Fig. 16 lies within the range of 95% confidence level. Values of $t_{0.975}$ (95% confidence level) for different degrees of freedom were taken from the standard table given in statistical texts.

The student's "t" test was also applied to check the validity of the data at 95% confidence level. The formula used for these calculations is:

$$t = \frac{\bar{X} - \mu}{SD} \sqrt{(N-1)}$$

Results obtained down to the level of 10 fields using the above formula are significant at the 95% confidence level. In other words "t" tests indicate that ten (10) data points or 11.52% of the surface area of a test specimen is enough to obtain 95% confidence level. However, this is preliminary analysis and at the present time we intend to continue taking 50 measurement fields per sample. After more data is collected, the number of measurement fields will be decreased in the near future, if this lesser number of fields can still give 95% confidence level.

SECTION VII

CONCLUSIONS

Polishing and etching procedures have been perfected for Mobil Tyco, Wacker, and IBM group of silicon samples for obtaining proper contrast of structural defects suitable for QTM analyses.

A computer program for automated quantitative image analyses of grain boundaries, twin boundaries, and dislocation pits using Quantimet 720 Image Analyzer has been perfected. Preliminary data on a Wacker sample is discussed. In the future, it is planned to interface a Terak 8510 A minicomputer to the Quantimet 720 for an improved data collection capability.

SECTION VIII

REFERENCES

1. R. Natesh, "Quantitative Analysis of Defects in Silicon", Quarterly Progress Report No. 1, DOE/JPL 954977, Materials Research, Inc., Technical Report: MRI-255, June, 1978.
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T A B L E 1

CHEMICAL POLISHING OF WACKER SAMPLES

Polishing solution: mixture of HNO_3 : HF: CH_3COOH = 1: 2: 3 by volume

<u>Temperature ($^{\circ}\text{C}$)</u>	<u>Time (sec.)</u>	<u>Surface Conditions</u>
50	30	slight smoothening of surface; but no polishing
50	45	underpolishing of surface, Facets join together to form subgrain type structure.
50	60 to 75	slight underpolishing. Subgrain type structure (due to facets) becomes larger, and, in some places, becomes faint and starts disappearing. Get staining and pit formation inside subgrain type structure.
50	80 - 85	Good even polishing. Subgrain type structure, and pits within subgrains completely disappear. -
70	45	slight underpolishing
80	55	reasonably good polish

- Note: (1) Time of polishing is to be increased or decreased depending on how soon and how fast bubbles evolve from sample surface.
- (2) For each polishing operation, a fresh solution must be used since the strength of solution decreases drastically after just one use.

T A B L E 2

CHEMICAL POLISHING OF IBM SAMPLES

Polishing solution: mixture of HNO_3 : HF: CH_3COOH = 1: 2: 3 by volume

<u>Temperature (°C)</u>	<u>Time (sec.)</u>	<u>Surface Condition</u>
50	30	growth lines persist. Faceting persists.
50	45	growth lines disappear, but facets join together to form subgrain type structure.
50	60	surface appears very even and bright, however, faint remnants of subgrain type structure still persists.
50	85 to 90	Good even polishing

- Note: (1) Time of polishing is to be increased or decreased depending on how soon and how fast bubbles evolve from sample surface.
- (2) For each polishing operation, a fresh solution must be used since the strength of solution decreases drastically after just one use.

TABLE 3

CHEMICAL ETCHING OF IBM SAMPLES

<u>Composition of Sirtl Etch</u>		<u>Etching time (Sec)</u>	<u>Surface conditions</u>
<u>Sol A</u>	<u>Sol B.</u>		
20 g CrO ₃ in 60 ml deionized water	60 ml HF	15	Revealed dislocations twins and grain boundaries. Resolution limited by the optical equipment
		additional 15	Revealed higher density of defects and less variation in contrast between twin boundaries.
<u>Sirtl etch Solution II</u>			
10 g CrO ₃ in 40 ml deionized water	40 ml HF	30	Revealed all dislocations, twin and grain boundaries.
		additional 30 sec.	Resulted in deeper etching of dislocations and over- lapping of twin boundaries.
		additional 30 (a total of 90 sec)	Resulted in significantly more overlapping of dislocation, twins.
<u>Sirtl etch Solution III</u>			
10g CrO ₃ in 60 ml deionized water	60 ml HF	50	Resulted in optical resolu- tion of 10 ⁻⁴ cm for twin boundaries and optical density resolution of 10 ⁷ dislocations per cm ² .

MAG-311.2 UNITS-MM CAL. FACTOR- 7.300964 UNITS/PP
 FRAME AREA- 0.464648

FIELD NO.	NO./AREA	MFPV	MFPH	L/A	AFETA	
1	10	21.522	0.217	0.143	9.294	7.303712
2	8	17.217	3.315	3.156	7.913	7.302969
3	6	12.913	3.154	3.446	7.216	7.302275
4	14	30.130	0.147	3.156	10.287	7.303014
5	7	15.065	3.263	3.261	5.794	7.303378
6	5	10.761	0.527	3.264	4.626	7.303872
7	1	2.152	8.033	4.869	3.268	7.303623
8	5	10.761	3.587	3.544	2.442	7.303127
9	8	17.217	0.147	1.578	7.476	7.302395
10	15	32.283	3.185	3.253	7.147	7.300941

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AVERAGE						
	NO.	NO./AREA	MFPV	MFPH	L/A	AFETA
	7.97	17.132	1.757	3.767	6.245	7.302415
SD	4.01	8.633	2.339	1.376	2.999	7.301111
SE	1.27	2.730	3.737	3.435	3.929	7.300751

11	10	21.522	3.296	0.214	7.129	7.302877
12	10	21.522	0.272	3.234	5.723	0.001069
13	1	2.152	4.595	2.274	3.574	7.301311
14	9	19.370	0.448	3.186	6.377	7.302773
15	3	6.457	3.663	2.919	1.618	7.301462
16	6	12.913	1.511	3.960	1.492	7.303027
17	8	17.217	3.183	3.112	11.278	7.306544
18	11	23.674	0.167	1.252	8.224	7.303195
19	3	6.457	0.692	3.343	3.499	7.304491
20	7	15.365	3.296	3.267	5.481	7.302625

AVERAGE						
	NO.	NO./AREA	MFPV	MFPH	L/A	AFETA
	7.35	15.818	3.976	3.767	5.716	7.302563
SD	3.71	7.974	1.076	1.172	3.167	7.301426
SE	0.93	1.783	3.119	3.262	1.798	7.30319

21	2	4.334	2.052	3.493	1.593	7.304867
22	7	15.365	3.286	1.237	6.228	7.303353
23	8	17.217	0.248	0.167	8.293	0.003452
24	6	12.913	3.473	3.237	6.172	7.304253
25	4	8.639	3.841	3.323	3.546	7.302734
26	8	17.217	0.287	3.315	5.135	7.301777
27	4	8.639	1.346	3.575	2.824	7.304983
28	4	8.639	3.413	3.261	4.813	7.303373
29	5	10.761	5.671	1.701	7.472	7.303347
30	9	19.370	3.172	3.499	3.538	7.301130
31	5	10.761	1.358	1.328	1.137	7.301285
32	4	6.609	3.349	3.568	3.721	7.303367

AVERAGE						
	NO.	NO./AREA	MFPV	MFPH	L/A	AFETA
	6.66	14.325	1.162	3.485	5.371	7.302061
SD	3.30	7.113	1.758	3.969	2.948	7.301924
SE	0.58	1.257	0.311	3.171	3.521	7.300347

33	8	17.217	0.355	3.275	4.734	7.302418
34	4	8.609	0.931	3.035	1.053	7.301612
35	5	10.761	0.998	3.288	3.023	7.302412
36	8	17.217	3.433	1.488	2.613	7.301451
37	3	6.457	0.623	3.316	3.915	7.302233
38	9	19.370	3.354	3.287	4.171	7.301634
39	10	21.522	1.168	3.103	11.071	7.302671
40	5	10.761	0.297	3.304	2.144	7.304112
41	9	19.370	0.375	0.194	6.540	7.303714
42	5	10.761	1.328	3.932	1.449	7.301724
43	12	25.826	3.477	3.491	3.307	7.300679
44	9	19.370	0.376	0.142	3.755	7.301319
45	19	40.891	0.101	3.368	18.617	7.303497
46	9	19.370	0.579	3.325	2.159	7.301573
47	18	38.739	3.140	3.142	11.193	7.302860
48	8	17.217	0.424	3.283	2.713	7.302526
49	7	15.065	0.644	3.404	3.337	7.301100
50	8	17.217	3.345	3.304	3.517	7.302181

AVERAGE						
	NO.	NO./AREA	MFPV	MFPH	L/A	AFETA
	7.38	15.883	0.862	3.573	5.247	7.302754
SD	3.75	8.363	1.443	3.801	3.447	7.301852
SE	0.53	1.140	0.204	3.113	3.487	7.30262

TABLE 5. "t" Test for Data in Table 4

Number of fields	Mean (X)	Variance	Standard Deviation SD	"t" test at 95% confidence level
50	5.247	11.88	3.447	significant
25	5.658	18.90	4.348	significant
17	4.116	7.628	2.762	significant
13	5.9255	25.39	5.039	significant
10	5.5403	13.91	3.73	significant
10	4.8481	5.577	2.3616	significant
10	5.4948	11.99	3.4629	significant
6	5.8869	3.508	1.873	significant
5	6.677	37.34	6.112	not significant

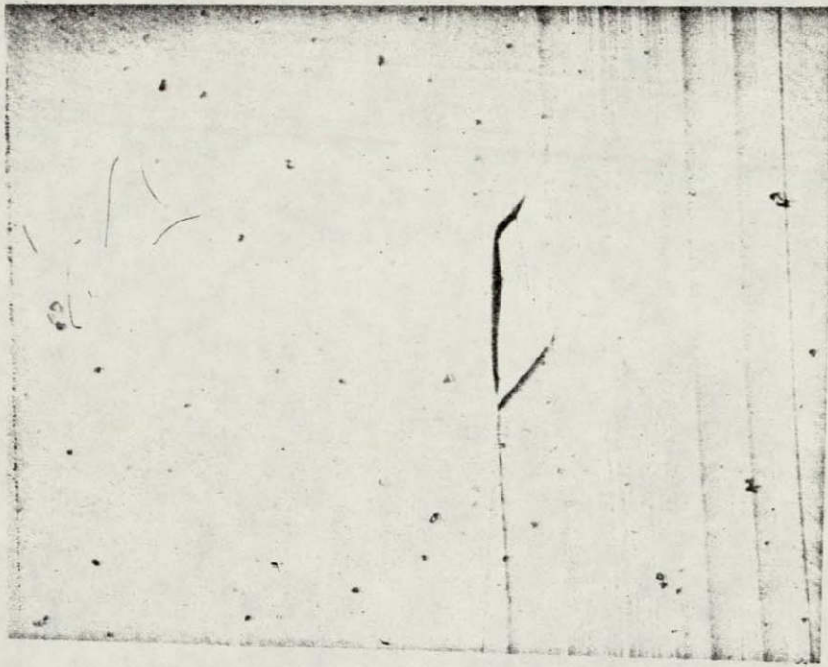


Figure 1. IBM #6 - Section 1 - Side 1, micrograph of ribbon surface after chemical polishing.
Mag 200X

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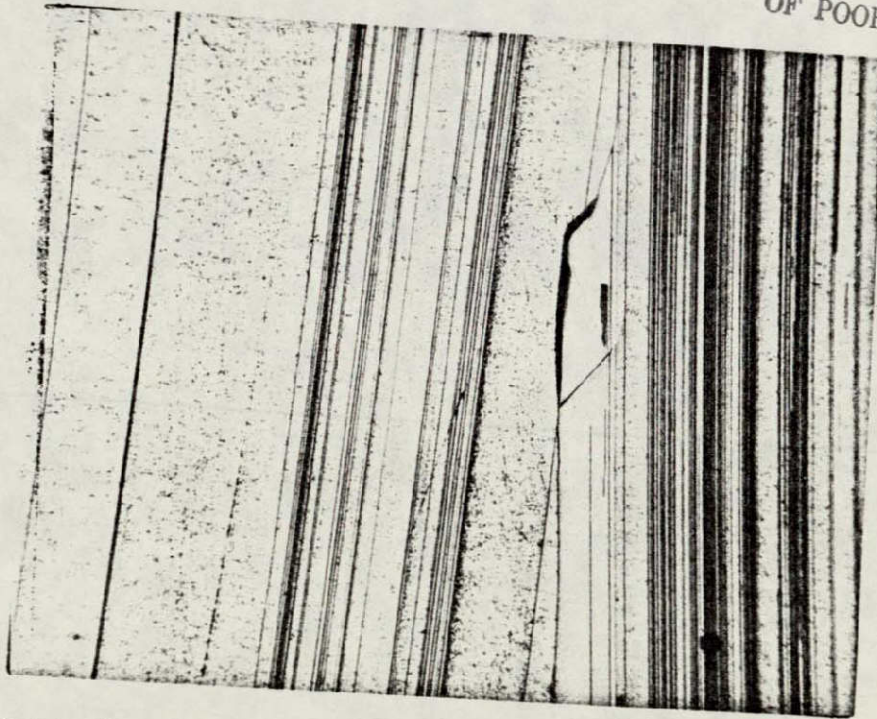


Figure 2. IBM #6 - Section 1 - Side 1, micrograph of ribbon surface after a 15 second etch by Etching Solution I
Mag 200X

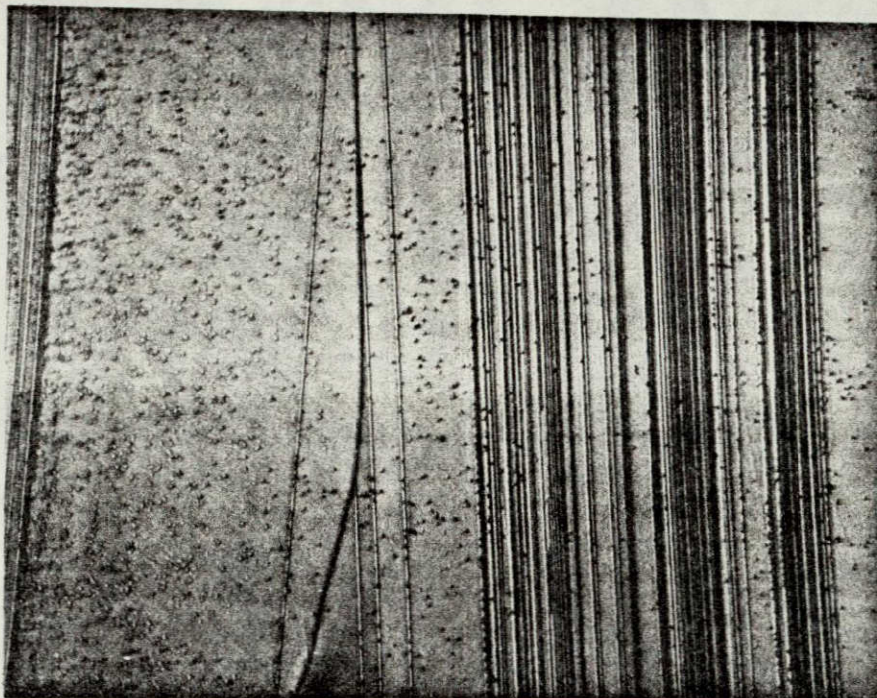


Figure 3. IBM #6 - Section 1 - Side 1, micrograph of ribbon surface after a 15 second etch by Etching Solution I.

Mag 500X

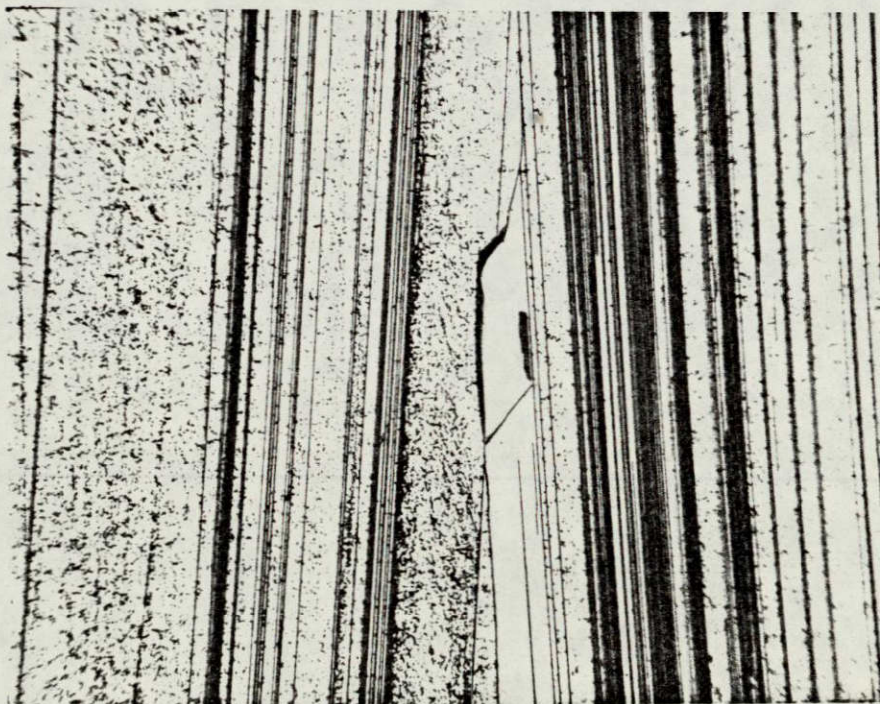


Figure 4. IBM #6 - Section 1 - Side 1, micrograph of ribbon surface, shown earlier in Fig. 1, 2 and 3, after a 30 second etch by Etching Solution I

Mag 200X

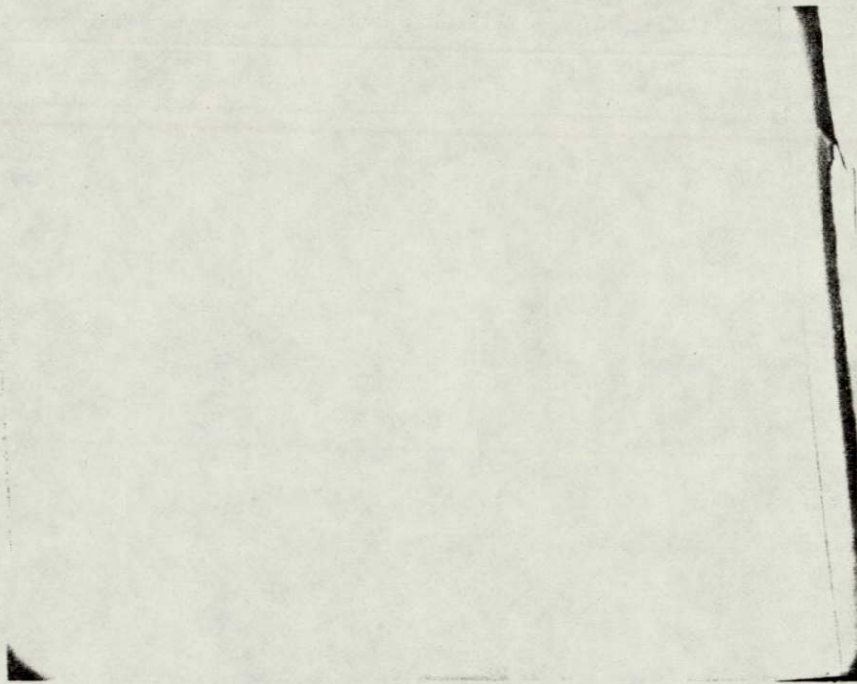


Figure 5. IBM #6 - Section 1 - Side 2, micrograph of ribbon surface after chemical polishing.
Mag 200X

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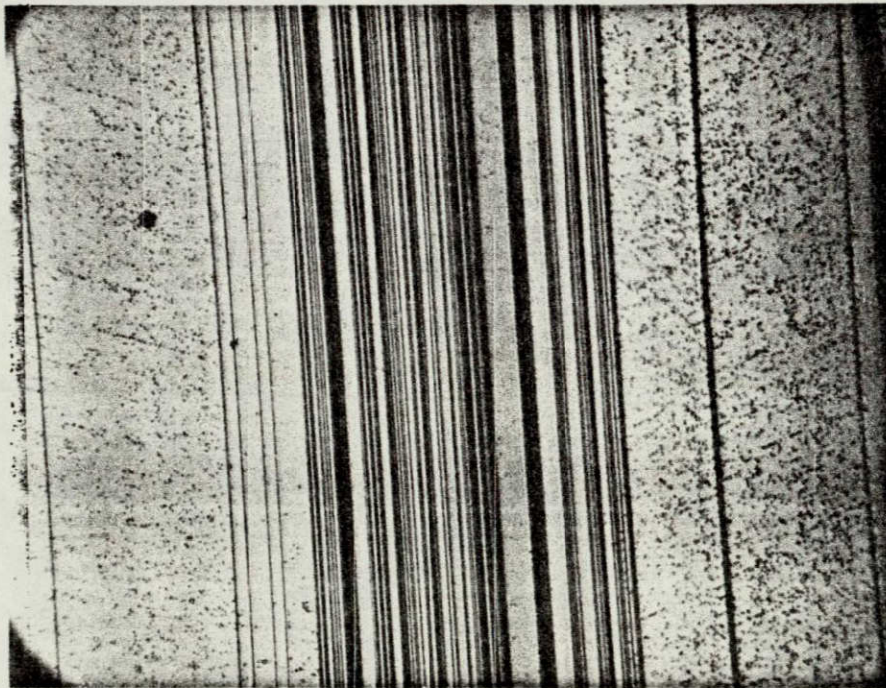


Figure 6. IBM #6 - Section 1 - Side 2, micrograph of ribbon surface, shown earlier in Fig. 5, after a 30 second etch by Etching Solution II.
Mag 200X

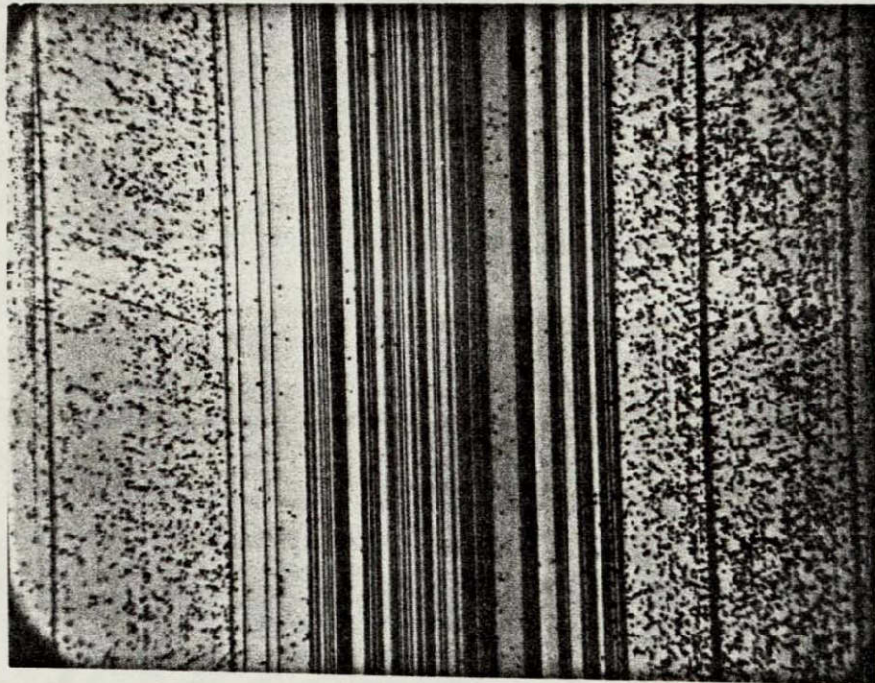


Figure 7. IBM #6 - Section 1 - Side 2, micrograph of ribbon surface, shown earlier in Figs. 5 and 6, after a 60 second etch by Etching Solution II.

Mag 200X

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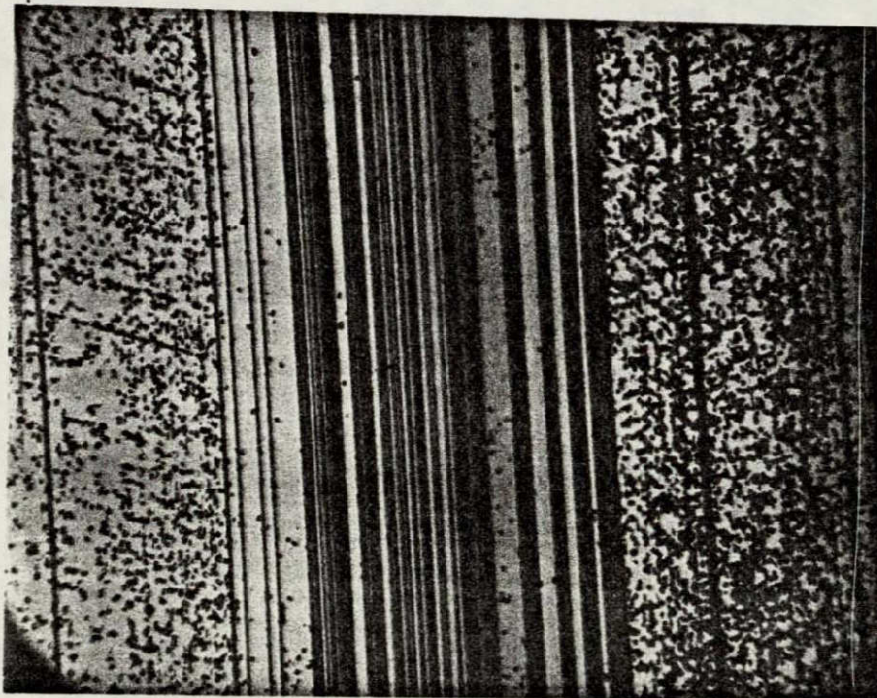


Figure 8. IBM #6 - Section 1 - Side 2, micrograph of ribbon surface, shown earlier in Figs. 5, 6, and 7, after a 90 second etch by Etching Solution II.

Mag 200X

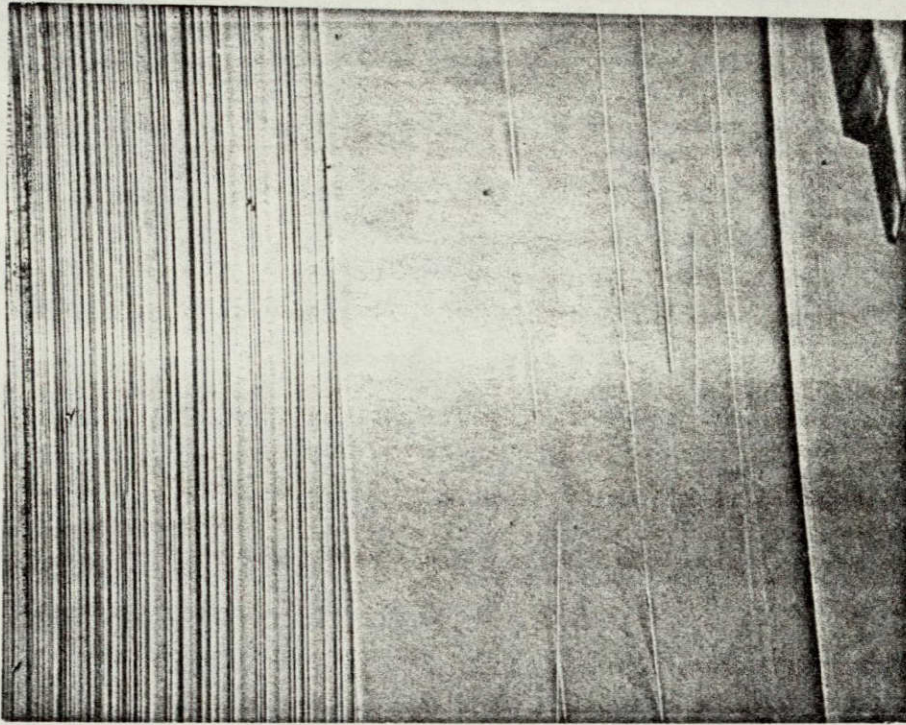


Figure 9 IEM #6 - Section 3 - Side 2 - Area 1,
micrograph of ribbon surface after chemical
polishing.

Mag 200X

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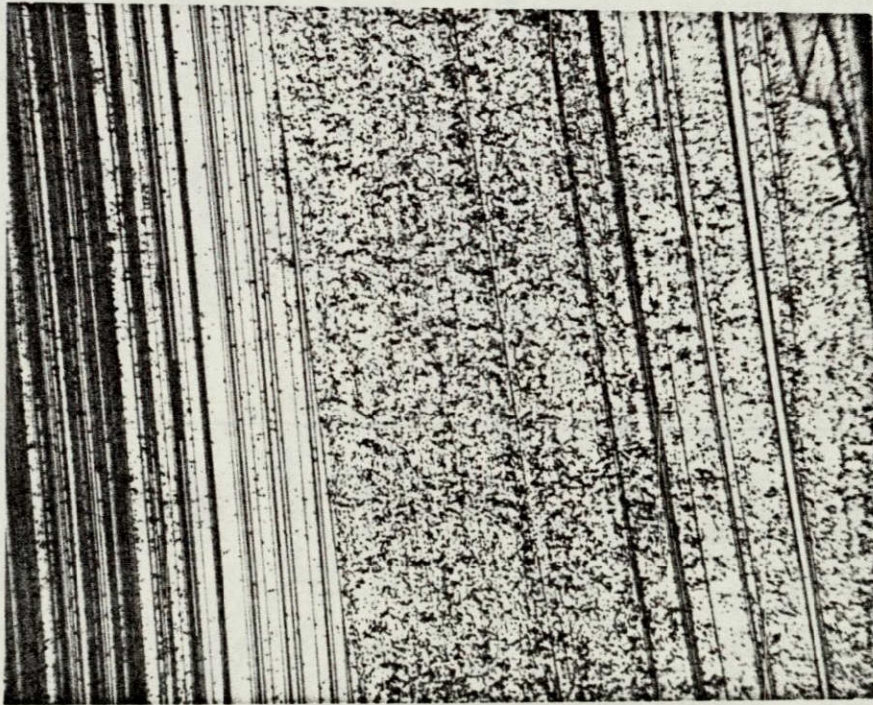


Figure 10. IEM #6 - Section 3 - Side 2 - Area 1, micrograph
of ribbon surface, as shown in Fig. 9, after a
60 second etch by Etching Solution III

Mag 200X

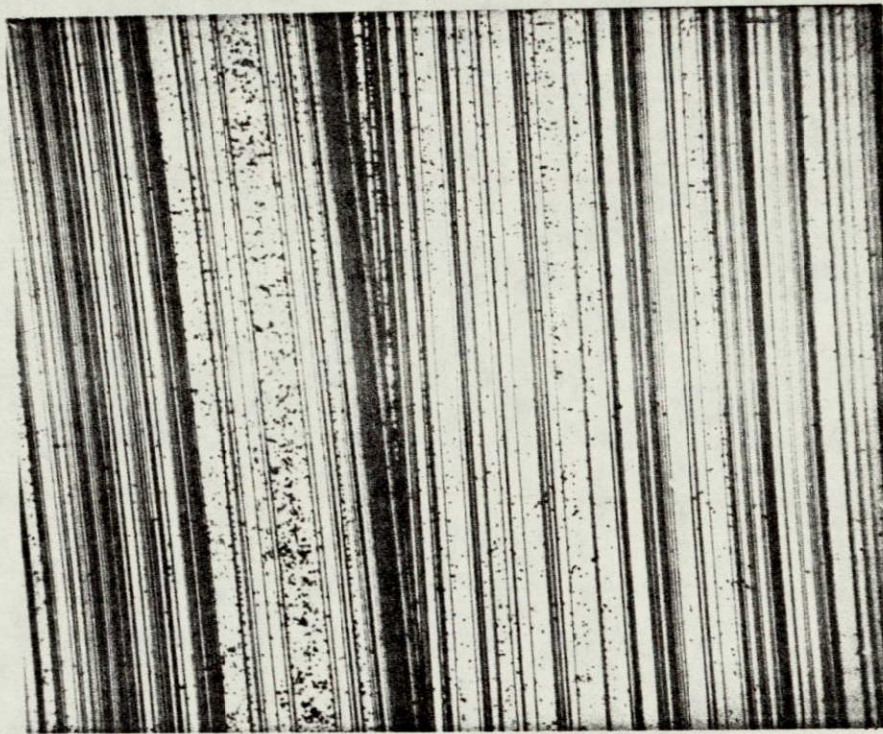


Figure 11. IBM #6 - Section 3 - Side 2 - Area 2, micrograph of ribbon surface after a 60 second etch by Etching Solution III.

Mag 200X

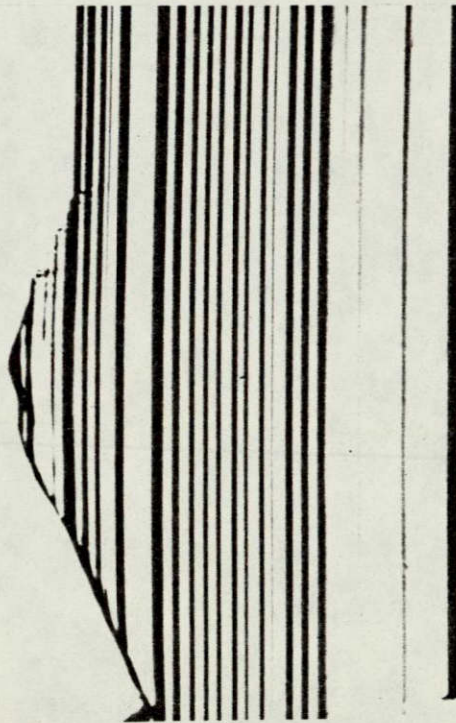


Figure 12. Wacker #7 - Section 1 - Area 1, micrograph of ribbon surface after chemical polishing.

Mag 210X

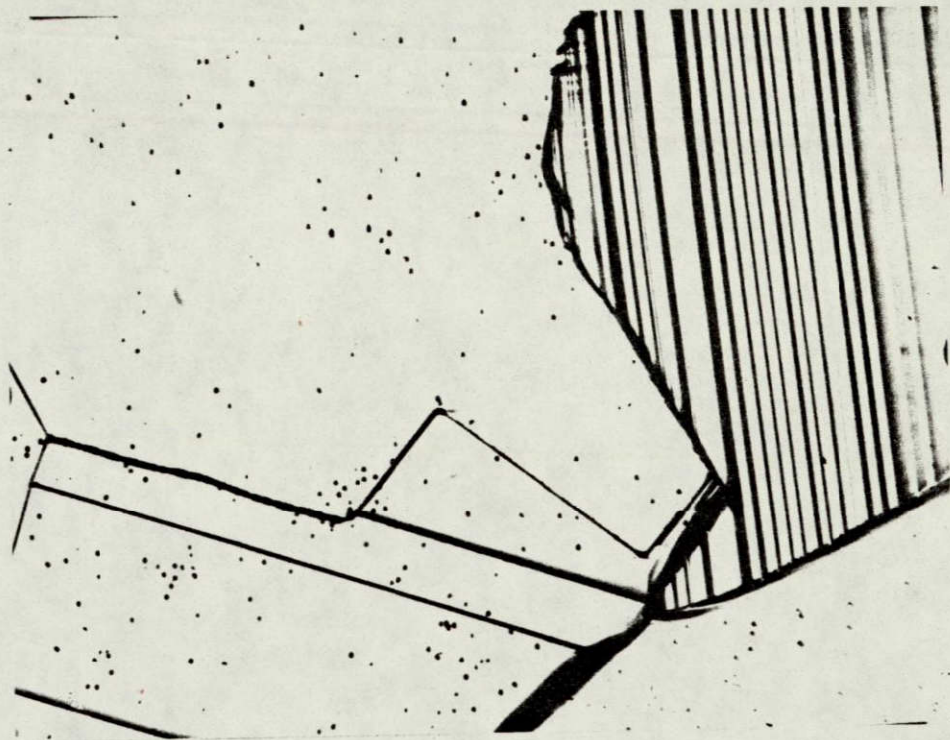


Figure 13. Wacker #7 - Section 1 - Area 1, micrograph of ribbon surface, shown earlier in Fig. 12, after 50 second etch by Etching Solution III.
Mag 210X

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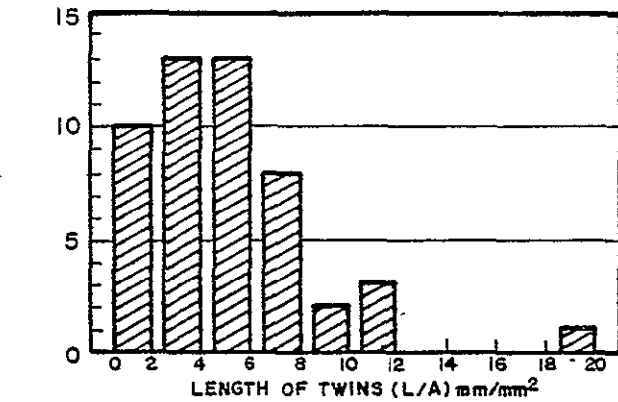
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FIGURE 15

QTM PROGRAMMER BOARD SET-UP FOR DEFECT
CHARACTERIZATION OF SILICON

<u>Column</u>	<u>Switches ON</u>
0	A, M, R, X
1	A, M, P, X, Y
2	A, M, P, R, W
3	A, M, N, W, Y
4	A, J, M, N, R

WACKER SAMPLE

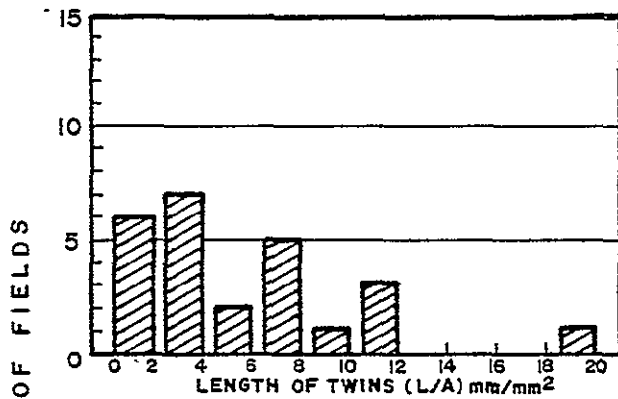


57.6% IN 40.32 mm²

N = 50 FIELDS
 \bar{X} = 5.247
 SD = 3.447

AT 95% CONFIDENCE LEVEL THE TRUE
 VALUE OF \bar{X} WILL LIE BETWEEN 4.2557
 6.236

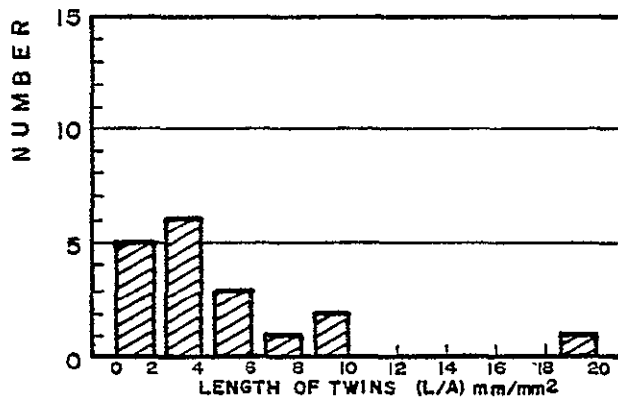
THE 95% CONFIDENCE LIMITS ARE
 GIVEN BY $\bar{X} \pm t \cdot 975 (6/\sqrt{N-1})$



28.81% IN 40.32 mm².

N = 25 FIELDS
 \bar{X} = 5.658
 SD = 4.348

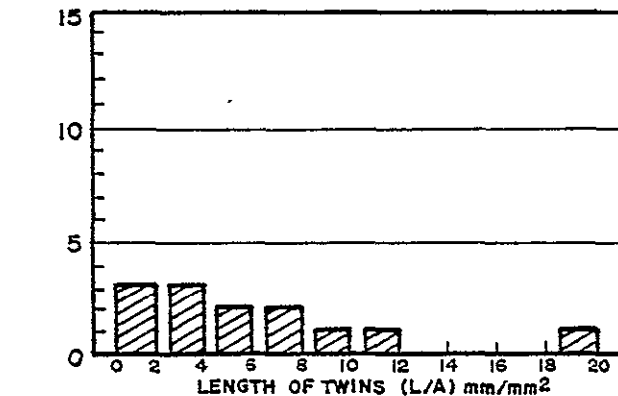
TRUE MEAN VALUE AT 95% CONFIDENCE
 LEVEL BETWEEN 3.8300
 7.486



19.59% IN 40.32 mm²

N = 17 FIELDS
 \bar{X} = 4.116
 SD = 2.762

TRUE MEAN VALUE AT 95% CONFIDENCE
 LEVEL BETWEEN 2.645
 5.558



14.98% IN 40.32 mm²

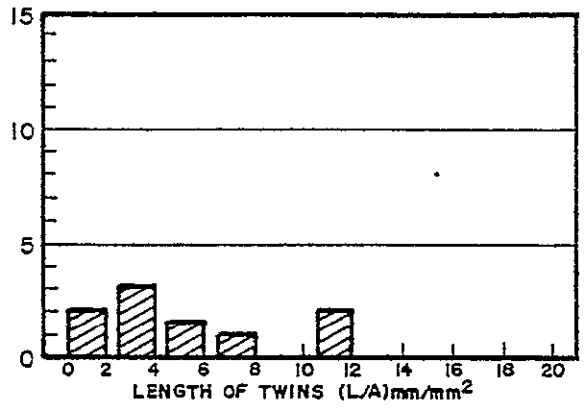
N = 13 FIELDS
 \bar{X} = 5.9255
 SD = 5.039

TRUE MEAN VALUE AT 95% CONFIDENCE
 LEVEL BETWEEN 2.7545
 9.0965

Fig. 16 Histograms showing distribution of L/A as Number of Fields is changed

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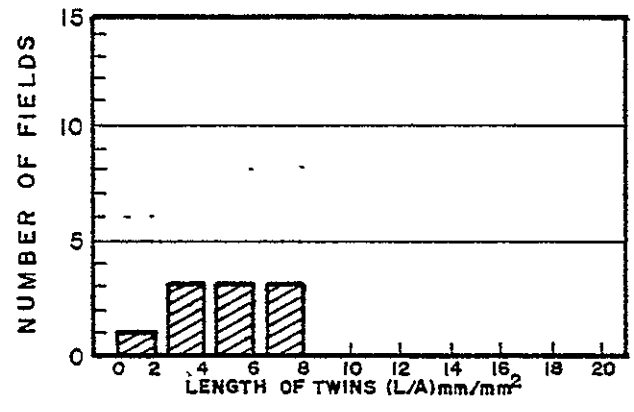
WACKER SAMPLE (CONT.)



11.52% IN 40.32 mm² AREA

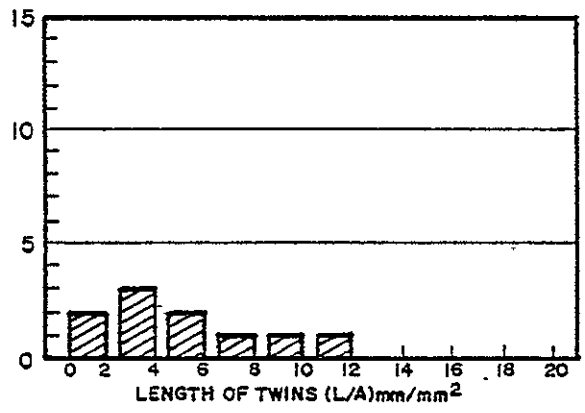
N = 10 FIELDS
 \bar{X} = 5.5403
 SD = 3.73

TRUE MEAN VALUE AT 95% CONFIDENCE
 LEVEL BETWEEN 8.3493
 2.7313



N = 10 FIELDS
 \bar{X} = 4.8481
 SD = 2.3616

TRUE MEAN VALUE AT 95% CONFIDENCE
 LEVEL BETWEEN 6.6271
 3.0691



N = 10 FIELDS
 \bar{X} = 5.4948
 SD = 3.4629

TRUE MEAN VALUE AT 95% CONFIDENCE
 LEVEL BETWEEN 8.1028
 2.8868

Fig. 16 (cont.)

APPENDIX A - HP9810 Program for Measurement
of Structural Defects in Silicon
(pages 40 thru 51)

<u>Register</u>	<u>Parameter</u>
001	Area of features in field (pp)
002	Perimeter of features in field (pp)
003	Vertical projection of features in field (pp)
004	Horizontal projection of features in field (pp)
005	Count of features in field
006	Frame area (pp)
007	Calibration Factor (units/pp)
008	Frame area (units)
009	Magnification
010	Cumulative area (pp)
011	Cumulative perimeter (pp)
012	Cumulative vertical projection (pp)
013	Cumulative horizontal projection (pp)
014	Cumulative count
015	Cumulative No. of fields

0101 21
 0102 01
 0103 01
 0104 00
 0105 00
 0106 09
 0107 FMT
 0108 4
 0109 1
 0110 5
 0111 FMT
 0112 H
 0113 A
 0114 G
 0115
 0116 FMT
 0117 FMT
 0118 FMT
 0119 4
 0120 1
 0121 5
 0122 FMT
 0123 CNT
 0124 1
 0125 H
 0126 I
 0127 YTO
 0128 YTO
 0129
 0130 FMT
 0131 STP
 0132 JFP
 0133 0
 0134 0
 0135 7
 0136 FMT
 0137 4
 0138
 0139 1
 0140 25
 0141
 0142 26
 0143 FMT
 0144 4
 0145 1
 0146 5
 0147 FMT
 0148 CNT
 0149 C
 0150 A

0151 L
 0152
 0153 CNT
 0154 F
 0155 A
 0156 C
 0157 YTO
 0158 0
 0159 0
 0160
 0161 FMT
 0162 FMT
 0163 FMT
 0164 4
 0165 1
 0166 5
 0167 FMT
 0168 CNT
 0169 1
 0170 H
 0171 I
 0172 YTO
 0173 YTO
 0174 DIV
 0175 A
 0176 A
 0177 FMT
 0178 JFP
 0179 0
 0180 0
 0181 8
 0182 JFP
 0183 JFP
 0184 9
 0185 9
 0186 7
 0187 YSO
 0188 7
 0189 YTO
 0190 8
 0191 JFP
 0192 FMT
 0193 4
 0194 1
 0195 5
 0196 FMT
 0197 STL
 0198 CLR
 0199 F
 0200 12

0331-- 1 ---01
 0332-- 3 ---03
 0333-- .ITO---23
 0334-- 1 ---01
 0335-- 4 ---04
 0336-- .ITO---23
 0337-- 1 ---01
 0338-- 5 ---05
 0339-- LBL---51
 0340-- 0 ---00
 0341-- 1 ---01
 0342-- .ITP---67
 0343-- + ---33
 0344-- 0 ---00
 0345-- 1 ---01
 0346-- 5 ---05
 0347-- PNT---45
 0348-- CNT---47
 0349-- CNT---47
 0350-- CNT---47
 0351-- FMT---42
 0352-- 3 ---03
 0353-- 3 ---03
 0354-- . ---21
 0355-- .ITO---40
 0356-- 0 ---13
 0357-- .ITO---23
 0358-- IND---31
 0359-- 0 ---13
 0360-- PNT---45
 0361-- .ITO---23
 0362-- + ---33
 0363-- 1 ---01
 0364-- 0 ---00
 0365-- FMT---42
 0366-- 3 ---03
 0367-- 3 ---03
 0368-- . ---21
 0369-- .ITO---40
 0370-- 0 ---13
 0371-- .ITO---23
 0372-- IND---31
 0373-- 0 ---13
 0374-- .ITO---23
 0375-- + ---33
 0376-- 1 ---01
 0377-- 1 ---01
 0378-- PNT---45
 0379-- FMT---42
 0380-- 3 ---03

0381-- 3 ---03
 0382-- . ---21
 0383-- .ITO---40
 0384-- 0 ---13
 0385-- .ITO---23
 0386-- .ITO---31
 0387-- 0 ---13
 0388-- .ITO---21
 0389-- 4 ---33
 0390-- 1 ---01
 0391-- 2 ---02
 0392-- PNT---45
 0393-- FMT---42
 0394-- 3 ---03
 0395-- 3 ---03
 0396-- . ---21
 0397-- .ITO---40
 0398-- 0 ---13
 0399-- .ITO---23
 0400-- IND---31
 0401-- 0 ---13
 0402-- .ITO---23
 0403-- 4 ---33
 0404-- 1 ---01
 0405-- 3 ---03
 0406-- PNT---45
 0407-- FMT---42
 0408-- 3 ---03
 0409-- 3 ---03
 0410-- . ---21
 0411-- .ITO---40
 0412-- 0 ---13
 0413-- .ITO---23
 0414-- IND---31
 0415-- 0 ---13
 0416-- .ITO---23
 0417-- + ---33
 0418-- 1 ---01
 0419-- 1 ---01
 0420-- PNT---45
 0421-- FMT---42
 0422-- 3 ---03
 0423-- 3 ---03
 0424-- . ---21
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 0431-- + ---33
 0432-- 1 ---01
 0433-- 1 ---01
 0434-- PNT---45
 0435-- FMT---42
 0436-- 3 ---03
 0437-- 3 ---03
 0438-- . ---21
 0439-- .ITO---40
 0440-- 0 ---13
 0441-- .ITO---23
 0442-- IND---31
 0443-- 0 ---13
 0444-- .ITO---23
 0445-- + ---33
 0446-- 1 ---01
 0447-- 1 ---01
 0448-- PNT---45
 0449-- FMT---42
 0450-- 3 ---03

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040099
040100

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040503--PHT--49
040504--CHT--50
040505--CHT--51
040506--CHT--52
040507--CHT--53
040508--CHT--54
040509--FR--55
040510--G--56
040511--G--57
040512--G--58
040513--UP--59
040514--FR--60
040515--G--61
040516--G--62
040517--FR--63
040518--G--64
040519--FR--65
040520--G--66
040521--G--67
040522--FR--68
040523--G--69
040524--FR--70
040525--DIV--71
040526--FR--72
040527--G--73
040528--FR--74
040529--G--75
040530--FR--76
040531--CHT--77
040532--PHT--78
040533--FR--79
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040536--CHT--82
040537--CHT--83
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040539--CHT--85
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040548--CHT--94
040549--CHT--95
040550--CHT--96
040551--CHT--97
040552--CHT--98
040553--CHT--99
040554--CHT--100

050001	+	04
050002	UW	03
050003	WY	02
050004	WT	01
050005	+	04
050006	I	03
050007	S	02
050008	WT	01
050009	WT	04
050010	WT	05
050011	WT	06
050012	WT	07
050013	WT	08
050014	WT	09
050015	WT	10
050016	WT	11
050017	WT	12
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0005	--	P	---	00
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0013	--	0	---	00
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0015	--	DIV	---	35
0016	--	FP	---	67
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0019	--	5	---	05
0020	--	DIV	---	35
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0024	--	1	---	01
0025	--	5	---	05
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0032	--	CNT	---	47
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0060	--	4	---	04
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0075	--	CNT	---	47
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0080	--	CNT	---	47
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1017	--- . ---	51
1018	--- 2 ---	58
1019	---GTO---	44
1020	---LBL---	51
1021	--- b ---	14
1022	---END---	46

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