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DEVELOPMENT OF MEGASONIC CLEANING FOR SILICON WAFERS

Quarterly Report No. 4

By
A. Mayer

April 1, 1980

Work Performed Under Contract No. NAS-7-100-955342

RCA Corporation
Solid State Division
Somerville, New Jersey

and

RCA Laboratories
Princeton, New Jersey

U.S. Department of Energy

Solar Energy

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PREFACE

This quarterly report covers the work performed by RCA Corporation, Solid State Division (SSD), Princeton and Somerville, New Jersey, for the subcontract under NASA Contract NAS-100, during the period of 15 December 1979 and 14 March 1980.

The contract is being administrated under the direction of Dr. A. H. Firester, RCA Laboratories, Princeton. The program is directed by A. Mayer, SSD, Somerville, New Jersey. Technical assistance is being provided by personnel at RCA Laboratories, including R. V. D'Aiello, R. E. Daniel, J. Murr, Jr., G. E. Nostrand, and D. T. Tarangioli.

The Technical Monitor for Jet Propulsion Laboratory is Dr. David Moffett.

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SECTION I

ABSTRACT

The Megasonic system consisting of the cleaning and rinse tank, the air dryer with the belt drive, and the laser defect scanner, has now been completely assembled, debugged, and tested in the laboratory at various power levels and belt speeds. The results indicate that wafer-throughput design targets have been met and exceeded. At the recommended power input of 160 W per transducer about 4500 wafers per hour can be cleaned and at least 2600 wafer per hour can be dried in 3/32-in.-spaced quartz carriers. As experience accumulated, several sources of contamination were identified. These included turbulence in the laminar-flow air stream, causing unfiltered room air to be inducted into the working area; the temporary aluminum supports for the quartz carriers; wash water contaminated with colloid, leading to slow and inadequate rinse rate; and leaks in the seal between the laminar-flow station and the belt drive. The laser scanner was found to be an excellent diagnostic tool: the pattern of scattering centers displayed on the storage 'scope is almost always indicative of the cause of the problem, be it dust, poor water, or surface defects on the wafer.

The solar-cell evaluation test carried out with 22 Megasonically cleaned and 18 system-Z-cleaned solar cells has been completed. It confirms that the Megasonically cleaned cells have higher average efficiency. However, about 17% of the Z-cleaned cells are considerably poorer than the rest; if these are not included, the distribution of the remaining 83% is statistically indistinguishable from that of the Megasonically cleaned ones. It is tentatively concluded that Megasonic cleaning produces a more uniform product with fewer really poor cells, but not cells with an efficiency greater than the maximum attained by Z-cleaned cells.

Another 25 test wafers were cleaned Megasonically and 25 control wafers were cleaned by system Z, just before the system was disassembled. These 50 wafers are being made into solar cells at RCA Laboratories.

The system was transferred to an RCA production facility for the production-environment testing, operation, and evaluation required by Task 3. Detailed instructions for its reassembly, operation, and maintenance have been written.

SECTION II

TECHNICAL DISCUSSION

A. MEGASONIC CLEANING STATION

1. Megasonic Tank, Transducers, Power Supplies, Carriers, and Platens

Although during this quarter the system was in regular operation nearly every day, no major problems developed. However, one power supply showed a significant instability 1-2 hours after being first turned on, observable as a drop in peak power output. The spare supply has now been used as a replacement while the defective power supply is being repaired.

The platen design shown in Fig. 2 (p. 4) of Quarterly Report No. 2 (Oct. 1, 1979) proved to be effective. The main novel feature that was tested was the nylon cord used to retain the wafers when the carrier is placed on the platen on its side. The cord is so thin that it does not present an obstacle to the sonic beam, yet effectively prevents wafers from rolling out. The second feature is the removable stop and support for the quartz carriers, necessitated by their design. These supports can be removed in a few seconds, leaving the platen ready to accept conventionally shaped carriers such as the ones made of polytetrafluoroethylene (PTFE), which are to be used for the manufacturing test.

The PTFE carriers with straight grooves need a wafer retainer across the bottom; in the past this had been made from a PTFE-coated thin stainless-steel rod. Experience has shown that during continued use some cleaning liquid can penetrate the coating and cause corrosion of the stainless steel. To avoid this, PTFE carriers used in the past were fitted with PTFE support rods. These have to be at least 3/16 in. thick and could cause some shadowing of the sonic beam. They have now been replaced by nylon cord and were found to give good service in continued use.

As discussed under drying (Section II.B), the sides of the quartz carrier were shortened from about 40 to 25 mm to increase the drying rate.

2. Recirculation System and Standard Clean Solution (SC-1)

The system continues to function as designed. The Gilmont flowmeter could not be plumbed in for reasons of space. The valve setting for the return

flow was therefore calibrated with water, which was collected in a calibrated vessel in the storage tank. Water was used to avoid exposing the operator to ammonia fumes. We have operated at recirculation rates of either one or two tank changes (68 or 136 L per hour). On no occasion did the quality of the SC-1 solution affect the cleanliness of the wafers. The SC-1 solution was not replaced (except when excessive water was inadvertently admitted). It was successfully refreshed daily by the addition of ammonia and hydrogen peroxide solution; this incidentally balanced the loss of liquid caused by drag-out and evaporation.

The 0.2- μ m polysulfone filter cartridge continues to function. Even the introduction of a relatively large amount of alumina as a deliberate contaminant during the cleaning ability studies did not cause buildup of excessive pressure. When the unit was shipped to Mountaintop, the filter was sent to Gelman. Bubble-point tests showed that it had in no way deteriorated.

Back pressure was judged by the Ametek* pressure gage. This gage has a PTFE diaphragm and transmits pressure via oil to a dial gage, which has continued to read between 1 and 2 psi. The dial gage was sent to the manufacturer once for a minor repair, a loss of oil to the outside, and has functioned well since then.

3. Rinsing

As mentioned in Quarterly Report No. 2, contamination of the deionized water with iron had been detected. Since then an absolute filter with 0.2- μ m pore size, made by Nucleopore Corp., Pleasanton, CA, has been inserted. Samples from the deionized water were taken before and after filtration. In all the cases that were tested, atomic absorption analysis indicated that any iron, copper, or sodium present must have been in amounts below the detection limits of 0.2 μ g/L for each iron and copper, and less than 0.025 μ g/L for sodium. During a four-week period, no discoloration was visible on the rinse tank. In analyses of the SC-1 solution iron and copper also were not detected, both initially and after continuous use for four days. During that period the sodium level for the freshly prepared solution increased from 0.6 to 1.7 μ g per L.

*Made by Truesdell Co., Skillman, NJ.

We concluded that the iron contaminant found in previous work derived either from a sporadic contamination or from the iron present in amounts below the detection limit of atomic absorption analysis. To become visible as a deposit on the side of the rinse tank, this iron contaminant may require a period of operation much longer than four weeks.

As the incoming deionized water later became heavily contaminated with colloidal matter, which rapidly blocked the 0.2- μ m filter, we became more convinced of the accuracy of the "sporadic" contamination model. The intrusion of colloid appears to have been caused by the local water company's changing to a new system of pipes, coupled with a problem in dispensing the coagulant prior to the first filtration of the raw water entering the plant.

The direct result of this contamination was a severe reduction in the flow of rinse water and a clear indication, conveyed by the streaky nature of the laser scanner display of the scattering centers, that the wafers had not been rinsed adequately. The situation improved immediately when the 0.2- μ m filter was changed, and deteriorated as soon as the filter plugged up again.

Analysis of the filtered deposit showed that major amounts of alumina, iron, calcium, and magnesium were present. These findings agree with the assumption that the contamination source was external to the plant.

It is also worth noting that the laser scanner was able to spot the deterioration of the cleaning ability of the system and permitted corrective action to be taken rapidly.

B. AIR-DRYING SYSTEM

In the cleaning tests made at the beginning of this reporting period, we fed the carriers manually through a Lucite* tunnel. To compensate for the shape of the quartz carriers, an aluminum sled had been constructed. It soon became clear that the aluminum constituted a source of particulate contamination. When a polypropylene sled was used instead, this contamination during the drying cycle disappeared (see Fig. 1).

The belt mechanism for the dryer was received at the end of January and required extensive engineering to make it operable. It is shown schematically

*E. I. du Pont de Nemours & Co., Inc., Wilmington, DE.

ALUMINUM SLED
COUNT = 1024

POLYPROPYLENE SLED
COUNT = 117

Fig. 1. Cool-air dryer - choice of material for sled.

in Fig. 2 and, as it is installed in the cleaning system, in Figs. 3-5. Its reliability has yet to be proved. It jammed on two occasions and broke the belt drive. The clearances have now been increased, and slip clutches have been ordered to be fitted between the motor and the belt drive.

Fig. 2. Schematic for belt-driven air dryer.

Care was taken to seal all the openings other than the ports so that no room air can be sucked into the clean duct. This effort was monitored with a Climet* II dust counter. A maximum of 16 particles of 0.3- μ m diameter or greater were found in a sample period of 10 minutes in the main air stream and in the tunnel. However, it became quite clear that any object that could cause turbulence at the ports could also cause contaminated air to intrude. For example, to achieve rapid drying when the wafers are only 3/32 in. apart in the quartz carriers, it is essential for them to be separated from each other and not leaning toward each other. Otherwise the air stream is cut off and

*Climet Instruments Co., Redlands, CA.

Fig. 3. Megasonic cleaning system with Megasonic cleaning and rinse station (left), dryer (center), and laser scanner inspection (right).

Fig. 4. Quartz carrier with silicon wafers being carried into the drying section by the belt drive.

Fig. 5. Dried wafers being pushed out into the inspection station by the belt mechanism.

wet spots remain for a long time. Initially we introduced deflector plates into the air stream that made the wafers lean first in one direction and then in the other. More recently, we have found that a filtered nitrogen stream delivered by a 3-mm-diam nozzle to the wafer edge was more reliable than the deflector plates. When this nitrogen stream was introduced in such a way that it caused turbulence, the scatter-center count increased immediately. To prevent this problem, the nitrogen jets are now being introduced from above the wafers and directed to the edge.

After the drive belt regulator was calibrated, drying tests were made at various belt speeds. It was found that the drying rates could be increased by approximately 25-30% when the side of the quartz carrier was shortened from about 40 to about 25 mm; those dimensions still provide ample wafer support. Complete dryness was achieved in such carriers at belt speeds up to about 18 cm/min. Above that speed one or two wafers per carrier had wet spots, usually at the edge. As can be seen from Fig. 6, this belt speed corresponds to a drying rate of about 2600 wafers per hour in 3/32-in.-spaced quartz. This figure also shows the corresponding cleaning rates. Whereas the cleaning rates are a function of only the wafer spacing, the drying rates are also affected by the nature of the carrier. In general, wafers in PTFE carriers dry only at about half the maximum rate achievable in quartz carriers.

I = PRACTICAL MAXIMUM BELT SPEED

Fig. 6. Comparison of cleaning and drying rates at various belt speeds.

In the course of the tests carried out in Somerville during the past months, the laboratory temperature has ranged from about 65 to 76°F and the relative humidity from about 20 to 76%. No major effect on the drying rates was observed, but it has to be borne in mind that most of the tests were made before the belt dryer was available. As wafers were pushed through manually, the reproducibility and statistical validity is questionable. Also, the system did not yet have to perform under the most adverse conditions of really high relative humidity as might occur during wet spring weather.

C. SYSTEM CLEANLINESS AND SCATTERING CENTER OBSERVATIONS

As was pointed out in earlier sections, problems which cause high scattering-site counts derive from sources that include poor rinse water, the use of aluminum supports for quartz carriers, and turbulence of the air stream. In addition, scratches, handling tool marks, and subsurface defects also show up as high counts under the laser scanner. Most of these are readily diagnosable by the patterns seen on the storage 'scope screen.

However, high counts in the range of 200-600 per wafer were observed on occasion. The particle counter was used to search for the cause of these sporadic contaminant sources. It was known from previous work that a linear relationship exists between the logarithms of particle concentration and their diameter, similar to that observed in liquids. The latter serves as the basis of classification in ASTM Standard F 575-78.* If this relationship holds for the laboratory where these tests were made, then it is necessary to count the particles of only one diameter; the concentration of particles with other diameters is predictable. This is shown in Fig. 7 for room air. The next line shows the distribution in the laser scanner hood: although this has a much lower particle concentration, the relationship still holds and the two slopes are similar.

The area between dotted lines, assumed to follow the same shape, indicates the particle concentration encountered in the Megasonic station when it is quiescent (lower boundary) and during maximum activity (upper boundary). This

*F 575-78, "Particle Concentration in Liquids," 1979 Annual Book of ASTM Standards 43, 1015, American Society for Testing and Materials, Philadelphia, PA.

Fig. 7. Dust particle distribution.

shows how critical careful fixturing is in avoiding the introduction of room air through turbulence. Note also that the air in the dryer is extremely clean.

An attempt was made to cross-correlate the diameter of scattering sites with a grid pattern etched into silicon. The lines and dots of this pattern ranged from 3 to 10 μm . When displayed on the storage 'scope at the sensitivity setting normally used in this work, lines with diameters of 5 μm were just visible. In a recent private communication by the inventor of the laser scanner it was pointed out that the optical properties of most scattering centers are such that the scattering power of small particles is at least 10-80 times greater than that of the etched lines. From the contamination studies in which we used 0.3- and 0.03- μm -diam alumina on the silicon wafers, it would appear that we can readily see clumps of particles having a diameter of 1-1.5 μm , as measured by phase-contrast microscopy.

D. MEGASONIC CLEANING ABILITY

By connecting one power supply to each transducer it was possible to double the power density. To ensure complete exposure of all wafers, the platen was turned around after it passed the transducer pair powered in the above-described way and was passed through it again. This simulated the normal condition of cleaning with two transducer pairs. The wafers were contaminated with alumina and dried for various times up to 20 hours. Rates up to 20 cm/min and power levels of 160, 220, and 300 W per transducer were examined by scanning the cleaned wafers and taking photographs of the distributions.

A rate of 15 cm/min is marginally worse than a rate of 20 cm/min. However, the results obtained at 15 cm/min, at the various power levels, were not markedly different. Figure 8 illustrates a typical test result that shows that at a belt speed of 15 cm/min the deliberately contaminated wafer can be cleaned. This speed corresponds to a throughput rate of 4500 wafers per hour. Figure 9 shows a somewhat scratched wafer which, before each cleaning step, was contaminated with alumina. It was then cleaned in the Megasonic unit at 160, 220, and 300 W per transducer. As virtually the same final scattering-center count was reached, we conclude that an input of 160 W per transducer provides adequate power density.

BEFORE

(0.5 g/L 0.03 m μ Al₂O₃)

COUNT=3619

AFTER

(160 WATT, 15 cm/min)

COUNT=248

Fig. 8. Megasonic cleaning at a belt speed of 15 cm/min,
equivalent to 4500 wafers/h.

Fig. 9. Transducer power vs cleaning ability.

We also conclude that the power supplies now being used are adequate for the existing system and the designed rate. Only long-term device-related and statistically significant tests could show whether larger power supplies would be cost effective for cleaning solar cells in mass production.

E. SOLAR CELLS

Twenty-four test and 18 control cells have been completed. This test confirms that the Megasonically cleaned cells have a higher average efficiency

than the ones cleaned in system Z (see Fig. 10). For example, the Megasonically cleaned cells averaged 8.46% efficiency (uncoated) and ranged from 7.3 to 9.3%, while the Z-cleaned cells averaged 8.2% and ranged from 7.1 to 9.1%. It appears that about 17% of the Z-cleaned cells are considerably poorer than the rest; if these are excluded, the distribution of the remaining cells becomes statistically indistinguishable from that of the Megasonically cleaned ones (see Fig. 11). It was therefore concluded that Megasonic cleaning produces a more uniform product with fewer really poor cells, rather than cells whose efficiency is greater than the maximum attained by Z-cleaned cells.

Another 50 cells were ion-implanted, 25 of which were cleaned Megasonically and 25 by system Z, just before the Megasonic system was shipped to Mountaintop, PA. These cells are being completed for measurement at the RCA David Sarnoff Research Center.

F. LARGE-SCALE TEST

The entire system has been shipped and reconnected at the RCA Mountaintop plant. It is planned to operate the unit in a regular production environment prior to the epitaxial deposition. This is a 24-hour operation and involves four operators, as well as the normal maintenance personnel for each shift. For the specific goals of this contract, plans have been made to record Megasonic system data on numbers of wafers, yield, chemicals usage, and normal maintenance. These factory environment data are particularly relevant for the near-term application of the Megasonic system to solar-cell production.

G. CONCLUSIONS AND RECOMMENDATIONS

1. The cool-air drying system was assembled and tested. It was found capable of drying at a rate of at least 2600 wafers per hour when quartz carriers with 3/32-in.-spacing were used. Some means are needed to separate wafers that lean toward each other in 3/32-in.-spacer carriers. These could be deflectors or air or nitrogen jets.

2. As the total system was being operated it became clear that the major cause of contamination was room air inducted into the cleaning area. The laser scanner continues to serve as an excellent process control tool, capable of rapidly detecting contamination problems.

Fig. 10. Preliminary solar-cell data.

Fig. 11. Solar-cell efficiency.

3. Up to 4500 wafers per hour can be processed through the Megasonic cleaning tank with 160-W input per transducer. Two modules with two transducers each were used.

4. The cells recently completed showed that Megasonically cleaned solar cells have a higher average efficiency than system-Z cleaned cells. Another 50 cells were cleaned and are being processed to firm up the statistical significance of the data.

5. The system was shipped to RCA Mountaintop.

6. It is recommended to continue the program as planned by operating the Megasonic cleaning and drying system as part of the RCA Mountaintop manufacturing plant processing sequence.