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DEVELOPMENT OF MEGASONIC CLEANING FOR SILICON WAFERS

Quarterly Report No. 1

By
A. Mayer

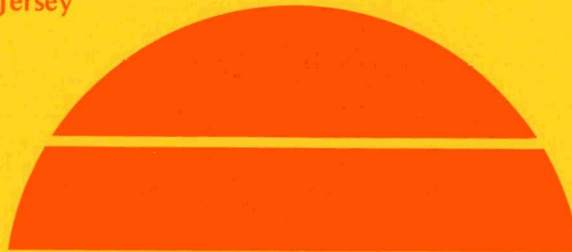
July 1, 1979

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

RCA Corporation
Solid State Division
Somerville, New Jersey

and

RCA Laboratories
Princeton, New Jersey



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Solar Energy

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A. Mayer
RCA Corporation
Solid State Division
Somerville, New Jersey 08876
RCA Laboratories
Princeton, New Jersey 08540

QUARTERLY REPORT NO. 1

Subcontract under NASA Contract NAS7-100
Task Order No. RD-152

July 1, 1979

The JPL Low-Cost Silicon Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic 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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CALIFORNIA INSTITUTE OF TECHNOLOGY
JET PROPULSION LABORATORY
Pasadena, California 91103

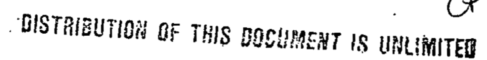

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TABLE OF CONTENTS

Section		Page
I.	ABSTRACT	1
II.	INTRODUCTION	2
	A. Program Objective	2
	B. Background	3
III.	TECHNICAL DISCUSSION	5
	A. Megasonic Station	5
	1. Description	5
	2. Assembly and Initial Checkout	9
	B. SC-1 Recirculation System	9
	1. Choice of Materials and Corrosion Testing	9
	2. Recirculation System	10
	3. Assembly and Initial Checkout	11
	C. Rinse Tank	11
	D. Air-Drying System	12
	1. State of the Art	12
	2. Design of Cold-Air Dryer	12
	3. Preliminary Tests on the Air Dryer	13
	E. Wafer Carriers	13
	1. Quartz Carriers	13
	2. Plastic Carriers	16
	F. Silicon-Wafer Laser Scanner	16
IV.	CONCLUSIONS AND RECOMMENDATIONS	19
V.	REFERENCES	20

LIST OF ILLUSTRATIONS

Figure		Page
1.	Megasonic unit	6
2.	Transducer	7
3.	Top view of Megasonic unit	8
4.	SC-1 recirculation system	11
5.	Cold-air dryer	14
6.	RCA Megasonic wafer carrier. Material: fused quartz. (a) End view. (b) Top view. (c) Side view	15
7.	Laser scanner	18

SECTION I

ABSTRACT

The purpose of the program is to scale up, automate, and improve the existing RCA-invented Megasonic Cleaning System to increase its throughput from about 600 wafers per hour to about 2500 wafers per hour, in preparation for the large-scale production of flat-plate silicon solar-cell arrays.

Design of the dynamic version of the Megasonic station was completed, and the unit was built, by the Fluorocarbon Company. It was delivered and is in the process of being installed. Plans are underway to maximize utilization of the cleaning solution - a mixture of water, ammonium hydroxide, and hydrogen peroxide - by recirculation and filtration. A circulation system was designed, corrosion tests were carried out, and most of the parts have been delivered. Tests showed that polysulfone might be a useful material of construction. The filter membrane and wafer carriers will be fabricated from this. An air-drying module capable of delivering air filtered through a HEPA (high-efficiency particulate air) filter at a speed of about 28.5 m/s was designed and delivered; the unimpeded flow was measured to be 25 ± 2.5 m/s.

Quartz wafer carriers were designed to fit diffusion tubes with 101-mm ID, one with 3/16-in. and another with 3/32-in. spacing. These have been ordered. A design for a plastic carrier, polysulfone or polytetrafluoroethylene (PTFE), was also worked out for use during the production rate, full-scale test of the Megasonic cleaning system.

SECTION II

INTRODUCTION

A. PROGRAM OBJECTIVE

The major objective of this program is to reduce the cost of processing solar cells on 3-in.-diam sliced and etched silicon wafers by use of the Megasonic Cleaning System. This will be accomplished

(a) by a major reduction in the use of chemicals, because Megasonic insonation eliminates the need for heating, and thus the hydrogen peroxide-based cleaning solution is not thermally decomposed;

(b) by introducing continuous filtration of the solution so that its useful life will be extended further;

(c) by removing particles from both solar-cell surfaces, coupled with a chemical cleaning step to remove metallic impurities. This will lead to a greater yield of cells with maximum carrier length and conversion efficiency;

(d) by the reduction in the cost of neutralizing and disposing of the much-reduced volume of spent cleaning solution;

(e) by designing an air-drying system that will permit wafers to be loaded into the quartz carriers to be used during diffusion so that the latter are cleaned and dried together with the wafers, i.e. without the customary transfer from plastic to quartz carriers. This feature not only reduces the probability of contamination, but also saves the cost of a transfer operation and reduces breakage;

(f) by redesigning the stationary, batch-operated existing Megasonic station for continuous-duty high-throughput rates so that low capital investment can be achieved;

(g) by eliminating most moving parts so that a higher than usual equipment utilization rate and reduction in maintenance is to be expected; and

(h) by introducing an RCA-developed laser scanner that can detect the presence of particles; this novel process control method will be explored, as it should further improve the yield of high-quality silicon solar cells.

By virtue of the improved utilization of chemicals, the expected high yield of 99.5% (mechanical), and the improved average cell efficiency, a savings of $0.16/W for a completed module is expected.

The increase in efficiency to an average of 12.5% is expected because it will be possible to clean and remove particles from both sides of the wafer

simultaneously, at no extra cost; this will lessen contamination and hence improve carrier diffusion length.

B. BACKGROUND

During the past six years RCA has evaluated the patented [1] Megasonic cleaning system for use in solid-state device processing. The results in terms of throughput, cleanliness, savings in chemicals, and reduction in breakage have been excellent.

This is a brushless scrubbing system with no moving parts [2]. It removes particles from the front and back of many wafers simultaneously, with a chemically clean solution. The scrubbing action is provided by piezoelectric transducers producing a 0.8- to 1-MHz sonic wave having a length of about 1.5 mm. This effectively removes particles down to at least 0.3-μm diam at an input power density of 5-10 W/cm^2 in contrast to ultrasonic cleaners, which normally operate at 25-80 kHz and at power densities up to 50 times higher.

The system was also coupled with hot-air drying in the same carriers. This is compatible with using quartz as the construction material. Thus, the wafers can be cleaned, dried, and transferred to a diffusion or anneal tube without having to be transferred from a plastic to a quartz carrier.

The preferred process for making flat-plate silicon solar-cell arrays calls for ion implantation in metal carriers, followed by an anneal and diffusion step at 500 and 850°C, respectively. At present the implanted silicon wafers are transferred to PTFE carriers and cleaned in hot sulfuric acid-hydrogen peroxide, rinsed, spun dry, and transferred to quartz carriers for the anneal and diffusion steps. No attempt is made to remove particles that are chemically or electrostatically attached and that may harbor metallic impurities capable of diffusing into the wafer and reducing the carrier diffusion length. RCA's experience with Megasonic cleaning indicated that not only are most particles that are not actually physically embedded in the surface removed in a Megasonic system, but a large number of the undesirable metallic impurities can be dissolved in a suitable solvent. The preferred solution is RCA's Standard Clean 1 (SC-1), consisting of about five parts of water, one of ammonium hydroxide, and one of hydrogen

1. A. Mayer and S. Shwartzman, U.S. Pat. 3,893,868 (assigned to RCA Corp.), July 8, 1975.
2. A. Mayer and S. Shwartzman, "Megasonic Cleaning: A New Cleaning and Drying System for Use in Semiconductor Processing," J. Electronic Mat. (in press).

peroxide solution [3], by volume. Because the solution is not heated, the ammonium hydroxide is volatilized only slowly, and the hydrogen peroxide does not decompose rapidly. This greatly extends the useful life of the solution and permits a reduction of over 80% in the consumption of SC-1. Further improvements in the utilization of the chemicals can most likely be achieved by removal of the major causes for the catalytic or physical decomposition of the peroxide, such as metal surfaces and particles.

---

3. W. Kern and D. A. Puotinen, "Cleaning Solutions Based on Hydrogen Peroxide for Use in Silicon Semiconductor Technology," RCA Rev. 31(2), 187 (1970).

SECTION III

TECHNICAL DISCUSSION

A. MEGASONIC STATION

1. Description

A system for the continuous cleaning of solar-cell wafers was designed and built by the Fluorocarbon Process Systems Div. of Fluorocarbon Company, at Anaheim, California. The photograph (Fig. 1) shows the new unit delivered in May 1979 in the process of being installed.

The design was based on the concept of moving carriers with wafers past pairs of transducers. As the largest commercially available piezoelectric ceramic capable of being driven at about 1 MHz, which is about 1.8 mm thick, has only a 63-mm diameter, two pieces that are offset have to be used to cover 76-mm-diam or 100-mm-diam wafers. Construction as well as repair is then quite simple if modules of such pairs are fabricated; one of these can be seen with cover removed (Fig. 2). When two such pairs, A and B, are incorporated (Fig. 3), they must be staggered (i.e., not opposite each other) to avoid interference of the sonic beam. In the new design a quartz reflector, C, is set into the wall opposite each transducer pair.

The wafers, D, are held in carriers, E, on platen F. The platen hangs from rod G, which engages on and is carried by a polypropylene continuous chain drive H. The speed can be controlled linearly between zero and 65 mm/min and is set by knob K (see Fig. 1). When the platen reaches magnetic switch I (Fig. 3), the drive is stopped. The operator will manually transfer the three carriers to the overflow rinse tank J. Eventually a transfer arm, which is standard equipment with chemical processing stations, can be incorporated. The transducers can be powered only when the drive is moving. This is to avoid excessive exposure of a plastic carrier if the drive is stopped while a carrier is exposed to the sonic beam. The solid-state power supplies O (Fig. 1) are stored underneath the plenum; each is driven by a signal generator, P (Fig. 1). The input power can be read on the wattmeter, L, and the supplies are adjusted by the power control, M. They are kept under constant nitrogen purge to avoid corrosion. The power supplies are interlocked through a level detector that shuts the power off if the liquid level falls below the level of the detector. If the transducers were exposed to air, i.e. not cooled, they would rapidly overheat and fail. The top level detector has to be covered before the unit can be powered.

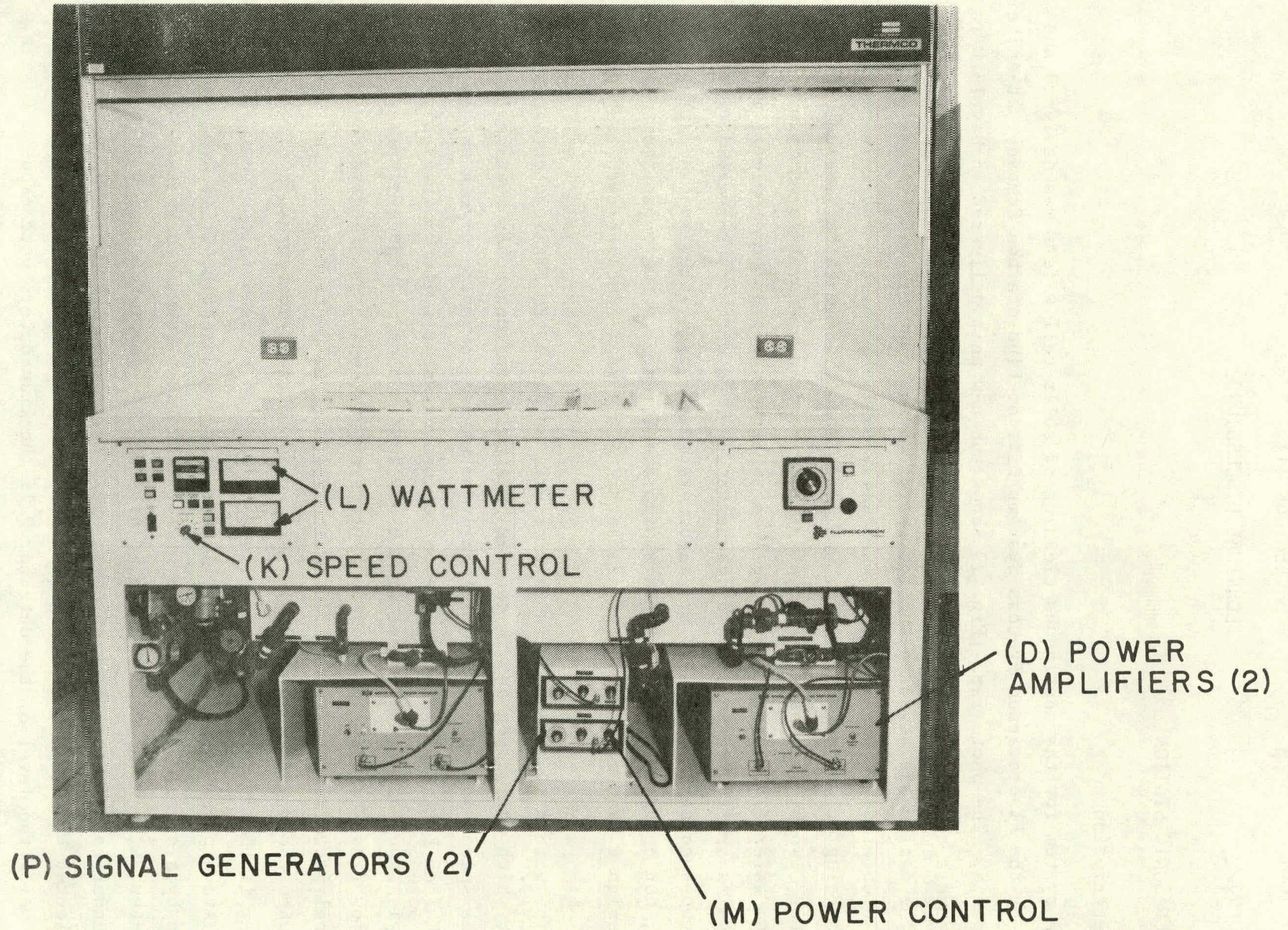


Figure 1. Megasonic unit.

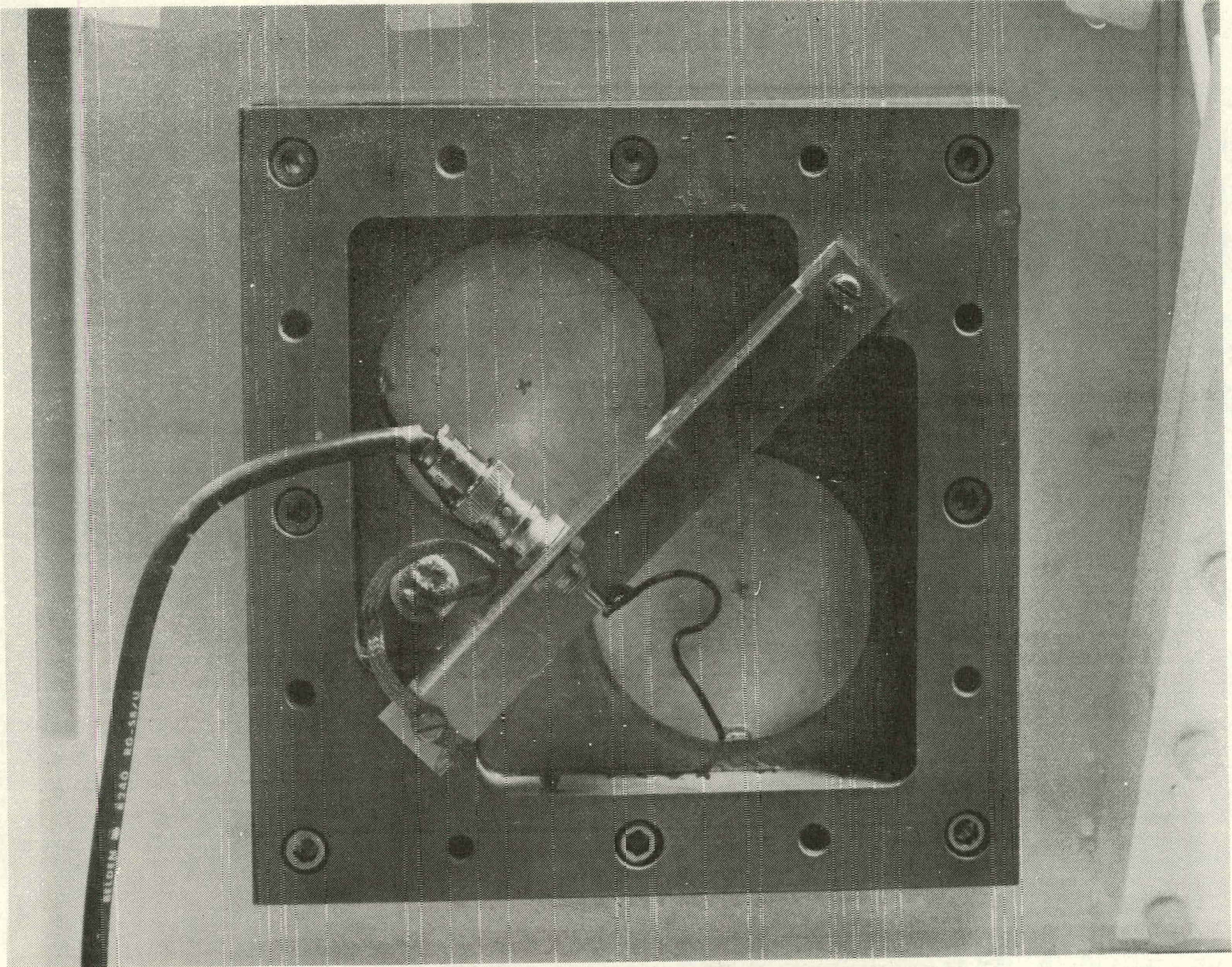


Figure 2. Transducer.

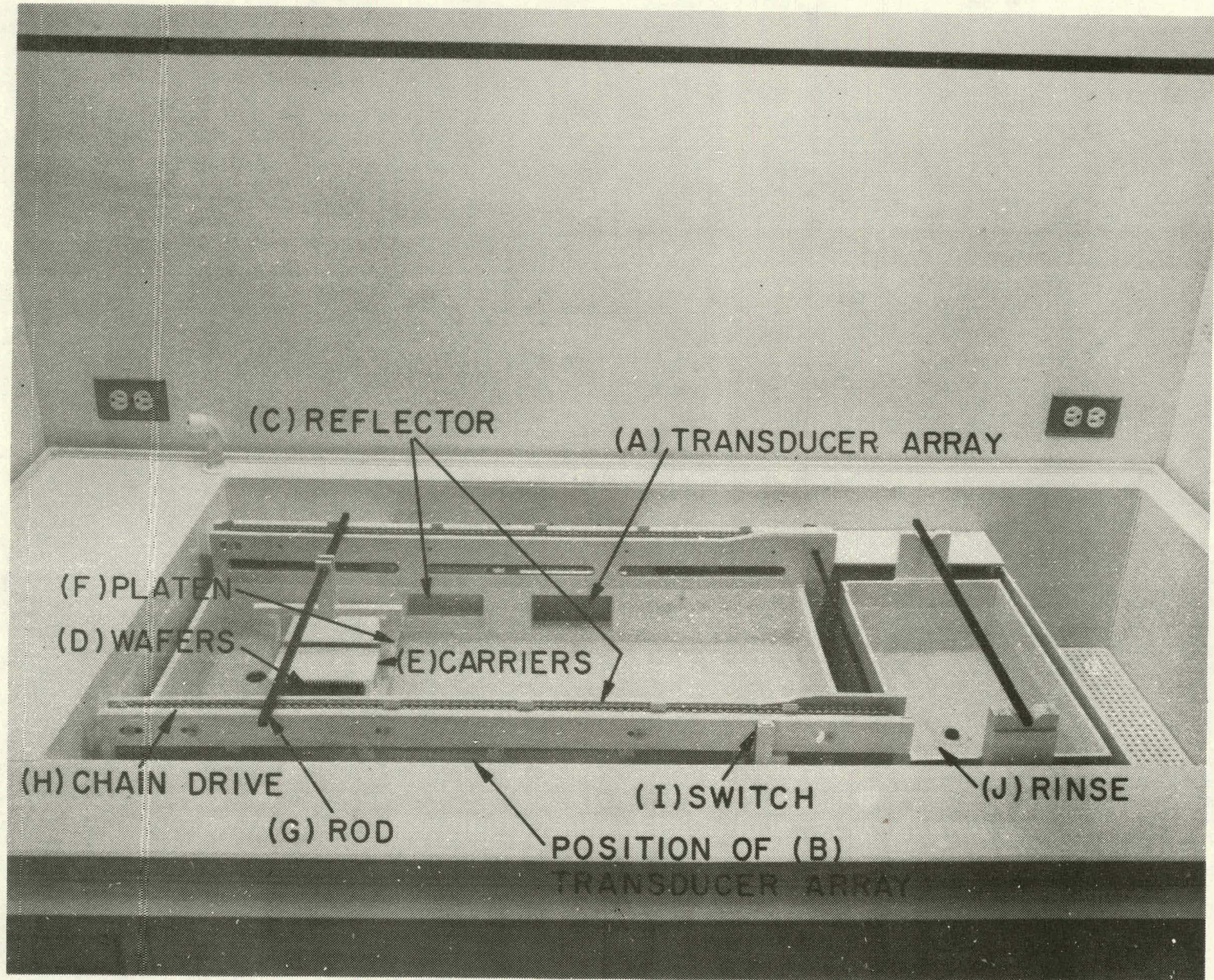


Figure 3. Top view of Megasonic unit.

2. Assembly and Initial Checkout

No problems were encountered in installing the unit in the laminar flow hood. Figure 1 shows this arrangement. The electrical system, interlocks, nitrogen purge, and power valves function as expected.

One transducer module was found to have a leak in the gasket; this was stopped after a change of gasket, but a second leak through the protective foil and backing plate required a replacement, and this was completed in mid-June. The major outstanding problem before the full system can be checked out is that the size of the overflow is too small to permit the circulation system to function at the minimum design rate of over 1 liter/min, i.e. with a tank change approximately every hour. Modifications have been designed and should be completed by the end of June.

Another problem is that the vane power indicators, designed to be deflected by the sonic beam when it is "on," require very careful positioning; it remains to be seen whether this will be acceptable for routine monitoring with a full load of wafers in SC-1 solution at working temperature.

B. SC-1 RECIRCULATION SYSTEM

1. Choice of Materials and Corrosion Testing

The following materials have already been use-tested in the older type of Megasonic system that has been employed at RCA for the past six years: polypropylene, polyethylene, polytetrafluoroethylene (PTFE), polychlorofluoroethylene, polyvinyl chloride (PVC), silicone rubber, quartz, tantalum, and zirconium.

For the recirculation system the use of the gray, asbestos-filled PVC was held to a minimum to avoid a possible source of contamination. The major piping system chosen was made of polypropylene, and all valves, pumps, and connectors were chosen so that no stainless-steel parts or metals other than tantalum were exposed to SC-1, because hydrogen peroxide decomposes catalytically on such metals as nickel, iron and platinum, and because SC-1 attacks copper and titanium and pits aluminum. A few fittings such as pipe size adaptors were obtainable only in the gray PVC; efforts to replace these at a later date will be made.

It should be noted that a 2- to 3-s exposure of PTFE to the sonic beam in SC-1 causes no damage, but that polypropylene melts locally during prolonged exposure, leaving a pinhole pattern. This is the reason for covering the wall of the tank opposite the transducers with a quartz reflector plate.

While the intent is to use quartz carriers for cleaning solar cells it may be expedient on occasion to clean in plastic carriers. PTFE is the first choice there and stands up well to insonation in SC-1, although it eventually shows some surface deterioration, becoming powdery and shedding particles.

Polysulfone, an inert material with a high-temperature tolerance, is used for filter membranes. As it can be molded and has good engineering properties, we also decided to test it for use in the recirculation system and as a possible carrier construction material. Accordingly, corrosion tests were run first on solid sections of polysulfone. No change in appearance, brittleness, or weight change was observed after immersion for 300 hours. Gelman Sciences Co., of Ann Arbor, Michigan, offers a wettable polysulfone membrane filter in a polypropylene housing. The filter is attached to the acetal copolymer core with polyurethane, and gaskets are either of neoprene or polyfluorochloroethylene.

Another test was made on the Gelman ASC 121 Acroflow cartridge. This was exposed in SC-1 for 35 hours at 50°C, for 85 hours at 23°C, and for 2 hours at 75°C. It showed no change except for a slight yellowing, and was sent to Gelman for detailed examination. The membrane was extracted from the filter and tested for water flow and bubble point integrity. Gelman's Quality Department reported no change in properties, compared with those of a new filter.

Although several filter manufacturers recommended PTFE membranes for use with SC-1, it was found that one manufacturer had problems sealing these membranes to the headers, while another warned about the danger of not prewetting the membranes before use, preferably in alcohol, because otherwise they might rupture when water was first admitted. Further, at about $300 each, these cartridges are quite expensive. The polysulfone cartridges, on the other hand cost about $90 each, have the same filtration area as the PTFE ones, and require no special handling at installation because they are wettable - good reasons to test these further in the recirculation systems.

2. Recirculation System

This was designed and all parts were ordered and received. The system is piped in polypropylene, the filter is the 0.2-μm polysulfone cartridge Acroflow 121 (Gelman) in a polypropylene housing, the pump is all PTFE, and all the diaphragms are made of polychlorofluoroethylene.

The recirculation system as shown schematically in Fig. 4 is self-explanatory. As mentioned in Section III.A.2, the overflow return hole is too small and a new design has been made and will be incorporated shortly.

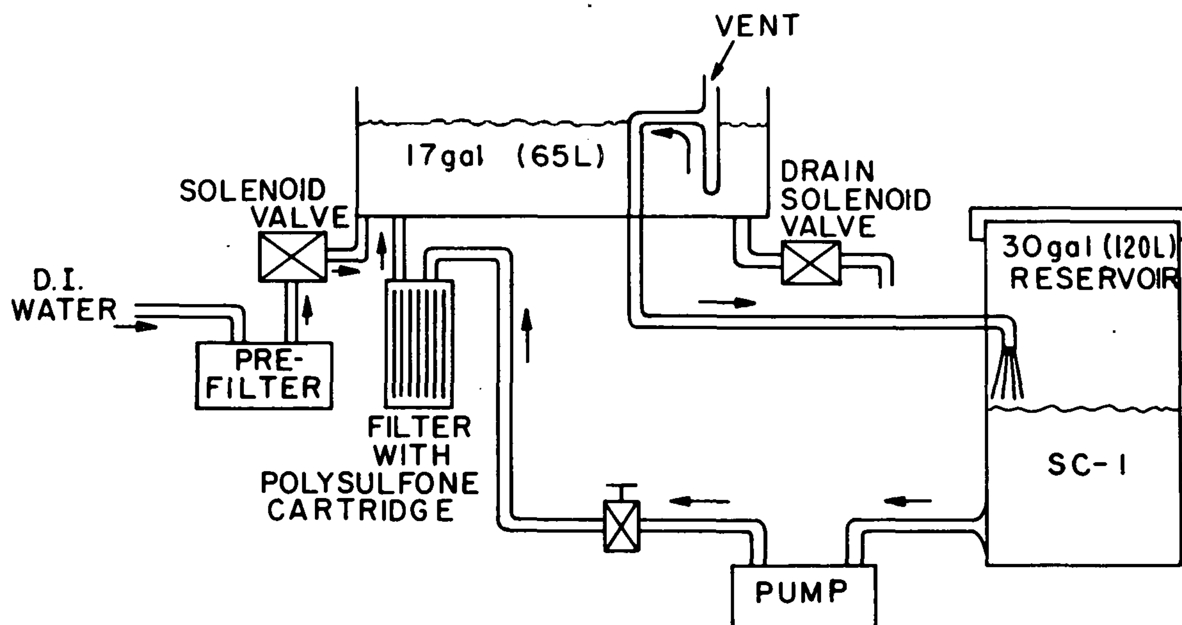


Figure 4. SC-1 recirculation system.

The Megasonic tank holds approx. 65 L; the circulation hold tank can hold up to 120 L. A pressure gage and flow meter were ordered and are due for delivery at the end of June.

3. Assembly and Initial Checkout

No immediate problems were observed. Another level detector has been ordered as a safeguard to avoid pumping SC-1 into the Megasonic tank at a rate faster than the overflow can handle. This detector will switch off the circulation pump and sound an alarm if the level is close to overflowing.

C. RINSE TANK

The rinse tank can be seen in Fig. 3. It is supplied by filtered deionized (DI) water with a resistivity higher than 12 MΩ·cm. The tank has room for one platen with three carriers. The platen rests on the yoke and magnetically activates a microswitch that increases the water flow whenever a load is present. Past experience has shown that 2 minutes of overflow rinse is usually sufficient to ensure adequate removal of surface impurities. The rinse tank appears to function as designed.

D. AIR-DRYING SYSTEM

1. State of the Art

It is customary in the semiconductor industry to dry silicon wafers by spinning them in a centrifuge. This has a number of disadvantages: the material of construction of the carrier cannot be quartz because quartz is not strong enough, and this implies that the wafers must be cleaned and dried in a plastic carrier and then transferred to quartz (which in turn has to be cleaned separately) before annealing, diffusion, or oxidation; another problem is that centrifuging is essentially a batch operation and does not fit in well with continuous processing; also the downtime of a centrifuge is relatively great because, like all highly stressed mechanical systems, a centrifuge requires maintenance and, besides, is difficult to clean if it gets contaminated by insertion of an improperly handled, dirty carrier, by the inevitable breakage of a wafer that showers debris, or by particle-laden air being dragged into the chamber.

The Megasonic systems in current use at RCA rely on a high-speed air dryer to first remove all the large water drops from the surface, then raise the temperature to about 100°C to ensure complete removal. This takes about 3 minutes and requires about 6 kW for heating the filtered air needed to dry one carrier with twenty-five 76-mm-diam wafers. The details of this system are fully described in Ref. 2. It should be noted that no streaks develop if the water used in rinsing is clean; conversely, if streaks are visible it is immediately clear that the water supply is dirty and needs attention. This self-indicating feature is most valuable in detecting a problem long before device electrical tests would show that it exists.

The best utilization achieved so far is the drying within 5 minutes of the contents of two carriers, one stacked over the other and each holding 25 wafers. Conceivably the number of wafers could be doubled by closer spacing, but even so hot-air drying would require 5 Wh/wafer plus the cost of the high-velocity air.

2. Design of Cold-Air Dryer

An alternative appeared to be to increase the airflow sufficiently to achieve drying with room-temperature air by physical displacement of the surface water and evaporation of the few surface layers of adsorbed water. This implies that drop formation should be avoided, i.e. that the surface energy of the water should be smaller than that of the wafers so that the drops can spread. As SC-1 naturally

makes the wafer surface hydrophilic, the water can be displaced in a relatively mild air stream. However, experimental work is required to determine how fast this stream has to be.

Preliminary experiments indicated that a stream with a velocity of more than approximately 12 m/s (= 2837 ft/min = 32 mph) may be acceptable for this purpose. An experimental air dryer was designed by RCA and constructed, delivered, and installed by Atmos-Tech Industries of Eatontown, New Jersey. It is capable of delivering air filtered through a large HEPA (high-efficiency particulate air) filter rated 99.97% efficient for particles down to 0.3 μm in diameter. The maximum velocity was designed to be about 23 m/s. This unit is shown in Fig. 5. It consists of the HEPA filter underneath a plenum chamber. Air is supplied by a 3/4-HP squirrel cage fan regulated by a speed control mounted on the side. The air is delivered through an opening that is just wide enough for one and long enought for two carriers, i.e., 90 mm x 305 mm. Eventually a duct will connect the laminar flow station, the dryer, and the inspection station. The latter consists of another laminar flow station and the laser scanner (see Fig. 7 in Section III.F); the latter, which has not yet been moved into place, will fit on the right of the drying station.

3. Preliminary Tests on the Air Dryer

The air velocity was measured with a Peto gage and found to be about 25 ± 2.5 m/s without a load. There appeared to be little turbulence, and the noise level was quite acceptable. Several designs are being studied at present for moving the carriers under the dryer after they are removed from the rinse tank.

E. WAFER CARRIERS

1. Quartz Carriers

Although quartz wafer carriers are commonly used, a special design for use in the Megasonic unit is necessary for these reasons:

(a) The structure must be as open as possible to avoid edge effects and to prevent obstruction of the sonic beam, which would create a shadow for the posterior carrier arranged in series with the anterior one.

(b) The existing furnace tubes for the fabrication of solar cells have a 101-mm-ID quartz tube, into which the carriers must fit.

(c) The spacing should be compatible with that used in normal processing.

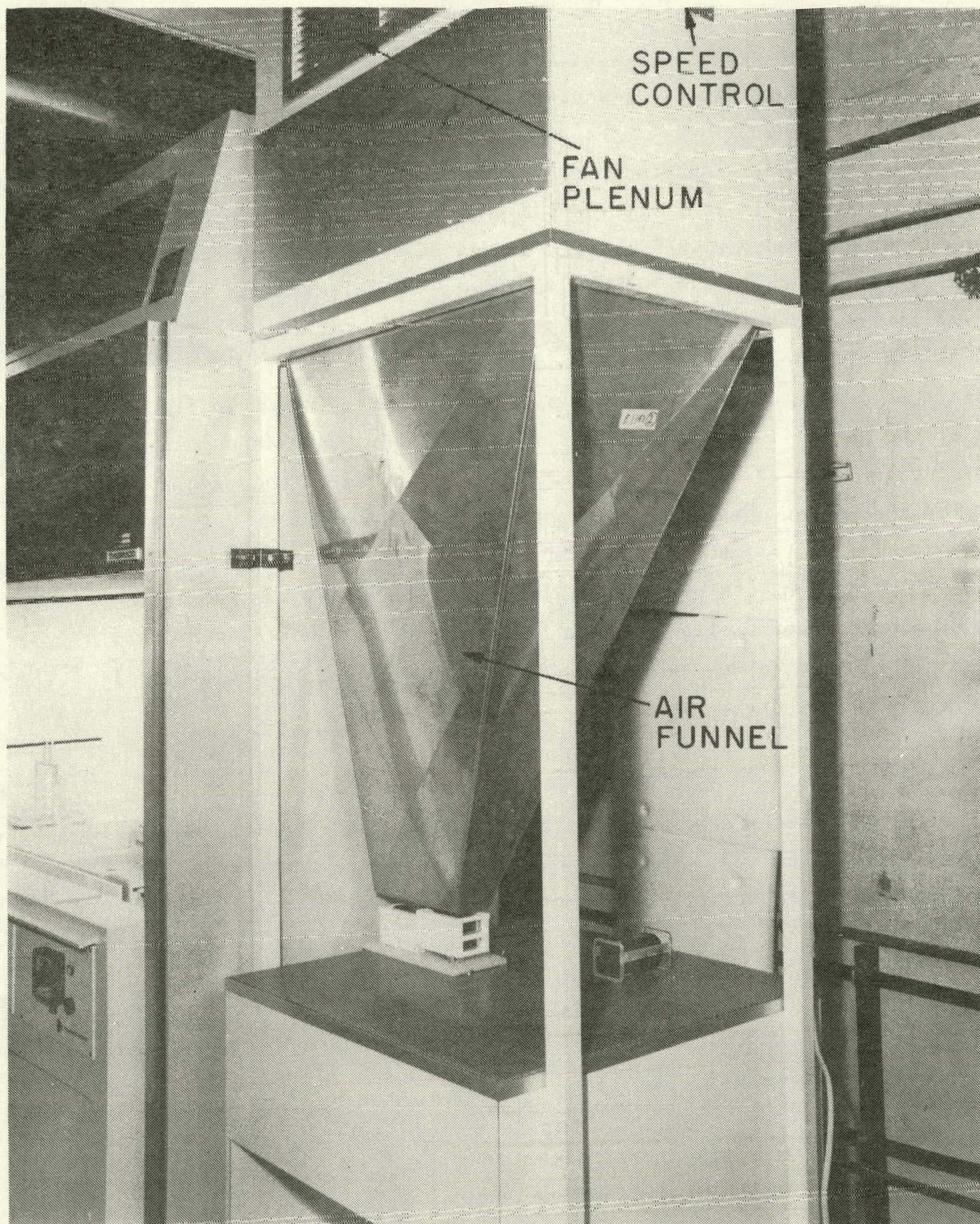
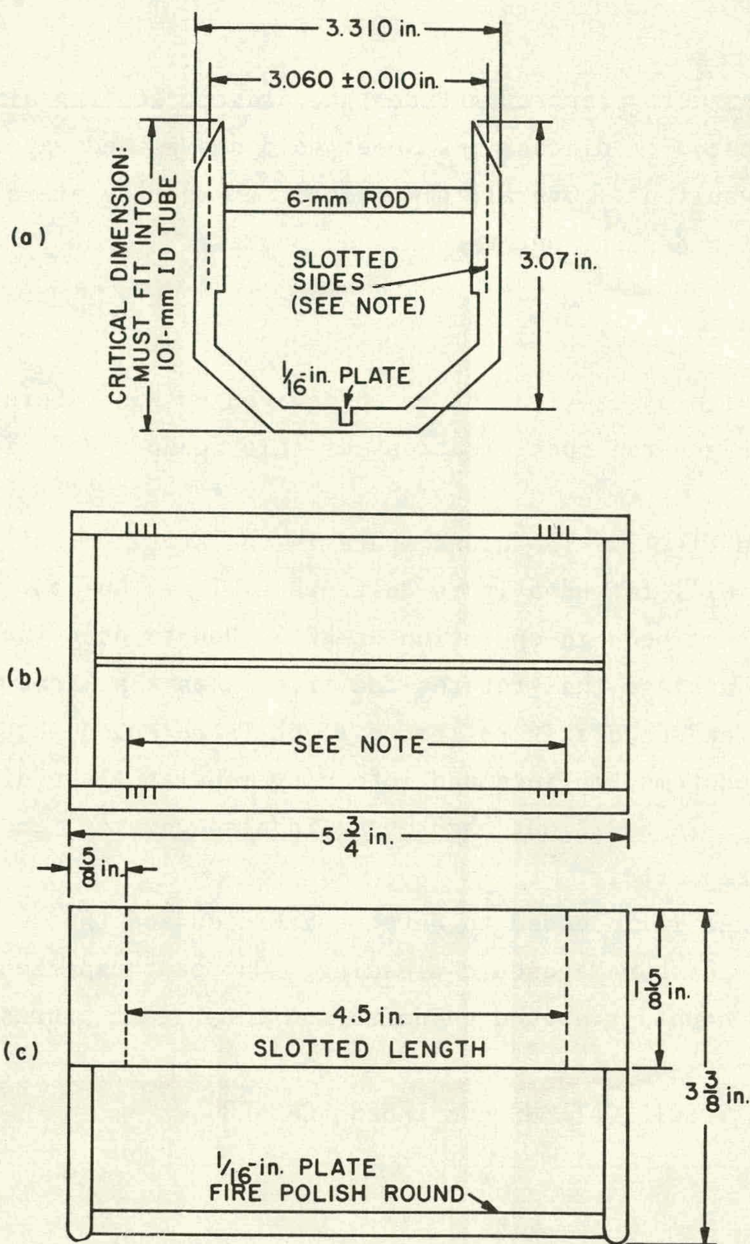


Figure 5. Cold-air dryer.

These considerations led to the designs shown in Fig. 6. An order was placed with U.S. Fused Quartz Company of Fairfield, New Jersey, to fabricate some quartz carriers with the standard 3/16-in. spacing, and some with a 3/32-in. spacing. Delivery is due at the end of July 1979.



NOTE:

NO.	SLOT SPACING
1	24 SLOTS AT 0.1875 in. = 4.50 in.
2	48 SLOTS AT 0.09375 in. = 4.50 in.

Figure 6. RCA Megasonic wafer carrier. Material: fused quartz.
(a) End view. (b) Top view. (c) Side view.

2. Plastic Carriers

Only conceptual sketches were prepared and sent to Plastic Tooling Aid Company of Southbury, Connecticut, for discussions about mold design and experimental fabrication from molded polysulfone. Overall dimensions and spacing are similar to those of the quartz carrier design, but the ends are square.

F. SILICON-WAFER LASER SCANNER

This instrument was designed, fabricated, and delivered by RCA Laboratories of Zurich, Switzerland. The general specifications of this scanner are given in Table 1.

The scanner is shown in Fig. 7. The blank space in the storage oscilloscope is where the plug-in module will fit when it is delivered. It is due on 18 June 1979. A similar instrument has been in operation at RCA's Mountaintop facility. That instrument consists of a stage that rotates and translates a silicon wafer under a He-Ne laser beam. The specularly reflected light is rejected, but scattered light is detected by photomultipliers and intensity modulates the display on the storage oscilloscope. In addition, each event is also counted, and the total counts are displayed separately.

The silicon-wafer scanner is intended to detect particles and thus provide a guide to the effectiveness of the Megasonic cleaning. Our past experience indicates that the particle count is a good guide to monitor "clean processing."

TABLE 1. SPECIFICATIONS FOR LASER SCANNER

1. GENERAL

Wafer size	3 in. standard; 4 in. max possible
Scan time	6 s (for 3-in. wafer)
Laser spot size	250 μm
Scan spiral pitch	200 μm
Spot overlap	50 μm
Detectability	
(at 3-in. wafer circumference)	about 7 μm
(at 3-in. wafer half-circumference)	about 3.5 μm
Scan spiral length	21.5 m (for 3-in. wafer)

2. POWER

Supply	115 V, 60 Hz
Turntable motor speed	1800 r/min
Scan motor speed	300 r/min

TABLE 1. (Continued)

3. OPERATING MODES

Auto : Automatic scanning from center to edge, return with beam off to center
Manual: Manual scanning with switches IN/OUT, BEAM ON/OFF

4. INTENSITY AMPLIFIER

Gain	:	1700
Bandwidth	:	dc to 600 kHz
Threshold	:	Adjustable sensitivity
Invert/Noninvert	:	Intensity modulation positive/negative
Test	:	Test pattern displayed, of 6-μs optical square wave pulses; to be used with laser turned off

5. DISPLAY

Counting	:	9999 events max
Threshold	:	Adjustable sensitivity (up to a maximum value given by setting of intensity amplifier threshold)
Invert/Noninvert	:	Intensity modulation positive/negative

6. COORDINATE TRANSFORMATION POLAR TO RECTANGULAR

Arranged so that picture of wafer on storage oscilloscope appears upright to permit 1:1 correspondence.

7. LASER (incorporated in system)

He-Ne Spectrophysics Model 145 P 2 mW
Power Supply 248

8. STORAGE OSCILLOSCOPE (proposed model)

Tektronix 5115 Storage Scope
Frequency range (intensity): dc to 1 MHz (Note: Square pulse of 2 V and length of 1 μs can turn storage on)
External intensity input (at back): Positive, i.e. +5 V turns display ON from OFF level
Setting: X : 0.5 V/div dc calibrated
Y : 0.5 V/div dc calibrated
Beam intensity: Vertical position
Enhance : OFF
Brightness : min
Storage : ON

9. VACUUM PUMP (proposed model)

Membrane pump equivalent to Reciprotor 506 R
Specifications: Endvacuum - 240 Torr
$0.5\ m^3/h$ at 360 Torr
$0.9\ m^3/h$ at 460 Torr
Power typically: 50 W

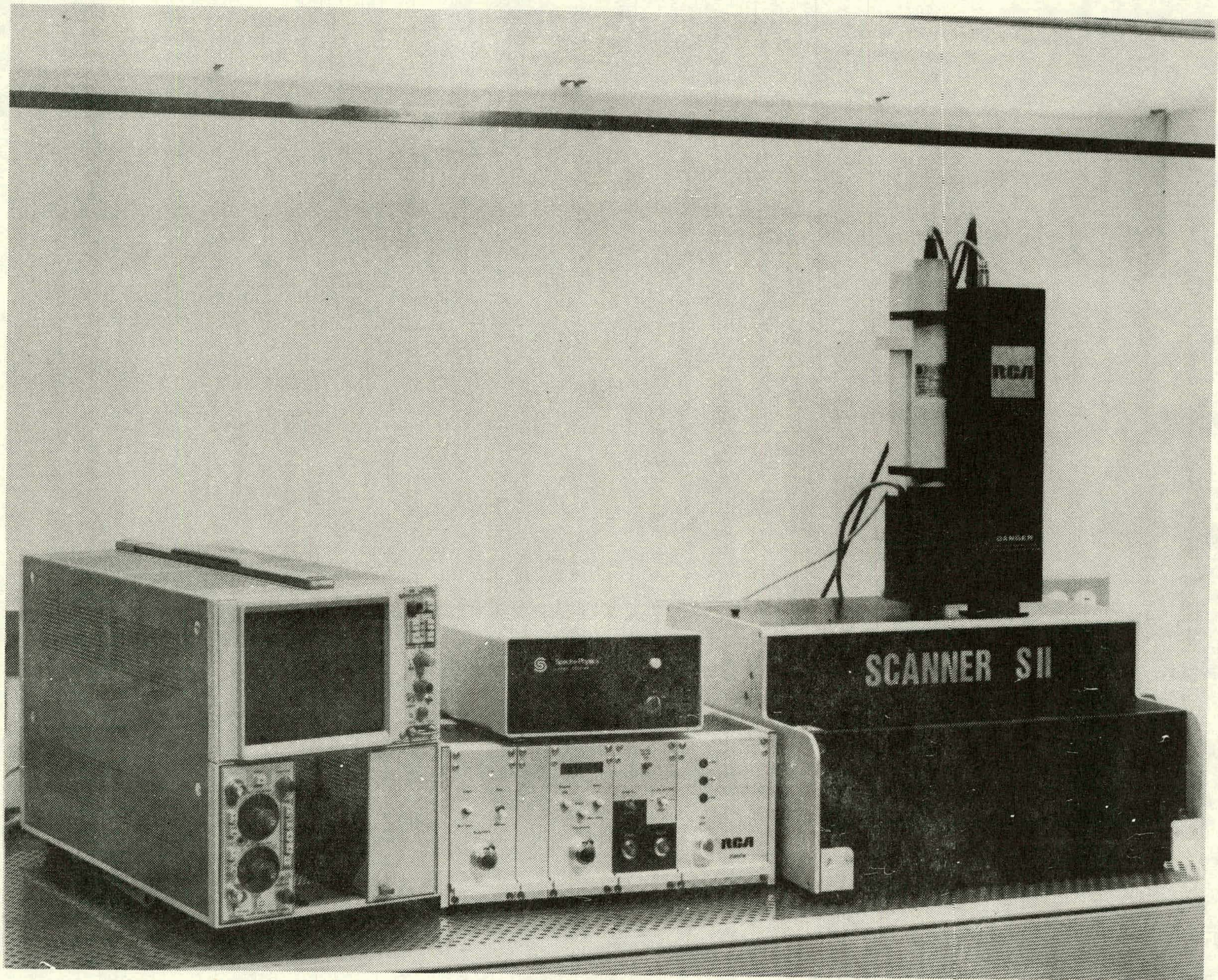


Figure 7. Laser scanner.

IV. CONCLUSIONS AND RECOMMENDATIONS

All program schedules have been met ahead of time. The only part of task I to be completed is the integration of the Megasonic cleaning station with the air dryer and the wafer scanner.

All parts appear to function as designed, but have not been tested in detail.

The program plan should be followed as set out.

V. REFERENCES

1. A. Mayer and S. Shwartzman, U.S. Pat. 3,893,868 (assigned to RCA Corp.), July 8, 1975.
2. A. Mayer and S. Shwartzman, "Megasonic Cleaning: A New Cleaning and Drying System for Use in Semiconductor Processing," J. Electronic Mat. (in press).
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