Cleanroom Air Monitoring Using Scrubbing and Adsorption Methodologies

by

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BIOGRAPHIES

MARJORIE K. BALAZS, founder and president of Balazs Analytical Laboratory in Sunnyvale, CA and Austin, TX, has worked in the Semiconductor Industry since 1968. She holds degrees from Stanford, University of San Francisco, and Washington University. She is on the Board of Directors of SEMI/SEMATECH, and in 1993 received the SEMI Lifetime Achievement Award for North America.

MARK J. CAMENZIND, Ph.D., joined Balazs in 1987, and is a Research Chemist in the Technology Development Group. He has been developing analytical services for trace organic analysis of chemicals on wafers, and in cleanroom air or mini-environments. He earned his B.S. in Organic Chemistry at Massachusetts Institute of Technology and his Ph.D. in Inorganic Chemistry from the University of California, Berkeley.

JÁNOS FUCSKÓ, Ph.D., Research Chemist, joined Balazs in May 1990, and is engaged in the development of methods for the determination of trace metals from a variety of semiconductor thin films using ICP-MS. Other research efforts include front end injection systems for ICP-MS and development of liquid sensors and monitors. He received his Ph.D. in Analytical Chemistry from the Technical University of Budapest in 1989.

ABSTRACT

In the past, cleanroom monitoring has generally meant the measurement of particles by size and number, and cleanrooms have been rated based on these findings. Now, a new cleanroom monitoring device has been developed to measure volatile acids and bases, and organic contamination. This system uses both scrubbing and adsorption collection techniques to collect the contaminates and concentrate them for analysis. Data that has been collected for the past year will be presented. They illustrate that cleanrooms that are free of particles still contain a considerable quantity of contaminating substances which have been shown to affect sensitive processes.

KEY WORDS

Semiconductor
Cleanroom
Monitors
Sensors
Inorganics
Organics
Contamination

INTRODUCTION

In the past, cleanrooms were only monitored for particles, since they were built to protect wafers from these defect producers. To supplement particle counters as a means to determine cleanliness, other techniques were also used. These included placing witness wafers in several locations, placing collecting vessels of ultrapure water in strategic locations, or wiping an area with something that picked up particles that were on surfaces or walls. In the past few years, however, manufacturers of very sensitive IC devices have come to learn that other agents cause defects during IC production and that the HEPA's themselves can be a contributor of contamination. These agents include airborne chemicals, both organic and inorganic, as well as particles too small to be detected by particle counters. Consequently, a new series of tests were needed to determine the cleanliness of any area that the wafer was exposed to and to measure the efficiency of the HEPA filters themselves.

To meet this need, a cleanroom monitoring system has been developed that utilizes three unique sample collectors. They employ both scrubbing and adsorption techniques. The three units consist of an air scrubbing system, an organic adsorption system, and a new type of particle concentrator that allows for more accurate submicron particle measurements in a reasonable time. Of these three systems, the scrubbing system has been used for the past one and one-half years and the organic system over the past half year The particle unit is still undergoing final testing. It is expected to go into use in 1995. Nonetheless, the principles, construction, and data from all three sampling systems are being presented in this paper.

INORGANICS

The scrubbing unit, called the Cleanroom Air Sampler (see Fig. 1), was developed to measure inorganic anionic and metallic contamination.

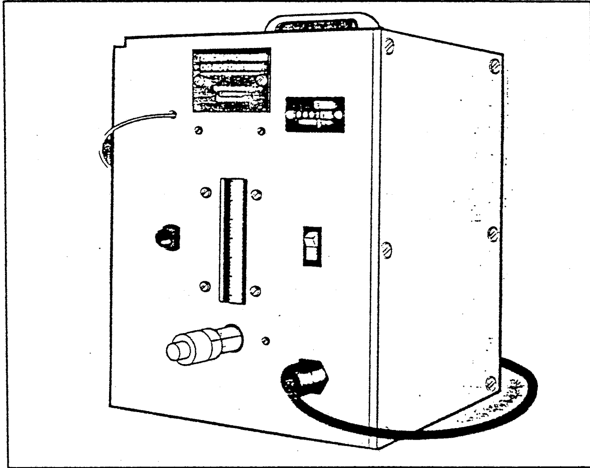

Fig. 1. Cleanroom Air Sampler

It is made up of a pump and a series of tubes through which air is drawn and scrubbed. The unit can be run from a few hours to several days without going dry. Air is scrubbed at a rate of one liter per minute. The resultant solutions are analyzed by ion chromatography (IC) and inductively coupled plasma mass spectrometry (ICP-MS).

It has been found that cleanrooms are so highly contaminated with acid fumes that only 16-24 hour sampling times are required, while 3 to 5 days are needed to measure metal concentration. However, with a more sensitive ICP-MS-HR, the testing time can be reduced by half or better.

Some ways that the Cleanroom Air Sampler has been used include the following:

- Evaluation of cleanroom air for metals, ammonia, and anions (volatile inorganic gases) at various points of the fab.
- The efficiency of HEPA filters and the composition of the air that comes through them, especially for metals and boron.
- Cross-contamination over wet benches.
- Sources of iron, calcium, sodium and boron contamination.
- Cleanliness of new fabs before opening for manufacturing.
- Quality of mini-environments.

Two specific cases where the sampler was used are worth mentioning. The first involved high iron contamination on wafers cleaned at a specific wet station. By testing the water, chemical, and air over the specific unit, it was found that the air had an unusually high concentration of iron in it. By measuring the HEPA performance as compared to other units, it was found that the air that was coming to the HEPA contained a large quantity of iron. This was ultimately traced to a corroding air conditioner. It has become evident in our studies that although HEPA filtration greatly reduces metallic contamination, it doesn't completely prevent it.

The second case involved our own recently built cleanroom at our new laboratory site. Our cleanroom, like all new fab cleanrooms, suffered serious contamination problems from both metallic and non-metallic materials caused by residual particles, sheet rock, new paint, ceiling material, and even our new HEPA's. By using our air sampler, the specific sources of these materials were found and remedied where possible. However, it was also found that new cleanrooms need considerable time to become clean after construction.

Comparisons of cleanroom ambient air within the same building where the same technology is being used showed significant differences in the air quality from site to site and month to month, especially for anion concentrations. These anionic materials exist in cleanrooms in far larger quantities than metallic materials, due to high concentration of mineral acid fumes, particularly HF and HCl. In one case, this type of problem was significant enough to cause fluoride contamination at several wet stations. Such contamination resulted in oxide damage.

An example of an actual evaluation of a cleanroom is shown in Table 1. The air was measured to determine the efficiency of the HEPA for the removal of metals. Metals were measured at three sites within the same cleanroom to determine concentration. The results show that the three HEPA's were passing different quantities of contaminating metals and were not performing as efficiently as others we have measured.

The second study on a fab is shown in Table 2. This study encompassed a range of anionic measurement over time. During the first part of the evaluation, anionic concentrations overwhelmed the collector. Because of the high anionic concentrations measured, especially at the wet bench, the test was repeated and run again three months after air flows were studied and improved.

ORGANICS

It is a known fact that wafers will get three to four atom layers of organic contamination on them as soon as they are exposed to cleanroom air. If these are volatile or oxidize easily, they cause little problem. However, if they are stable or become part of the IC, they can cause failures such as point defects, adhesion problems, residual material in open contacts, and aluminum degradation after encapsulation. Consequently, there has been increasing interest in identifying organics in cleanroom air, carriers, mini-environments, and wafer storage areas.

The organic sampler in this case is a tube containing activated carbon. The material collected is desorbed into a GC-MS where the organics are identified. In many cases, once identified, a quantitative measurement can be made and a quantitative comparison of organic contamination can be done over time. The detection limits are in the ppb range.

Using this system, it has been found that several common organic compounds are found in cleanroom fabs. They include alcohols, aromatics, hydrocarbons, plasticizers, and photoresist and stripper components. Furthermore, our studies show that HEPA filters do not filter out organics and, consequently, those that exist in outside air pass right into the cleanroom. Since many processing chemicals and cleanroom components contribute organics to the cleanroom air, cleanroom air generally contains more organic contaminants that can be deleterious to wafers than is found in air outside of the plant. Fig. 2 is an example of organic materials that were found in a cleanroom.

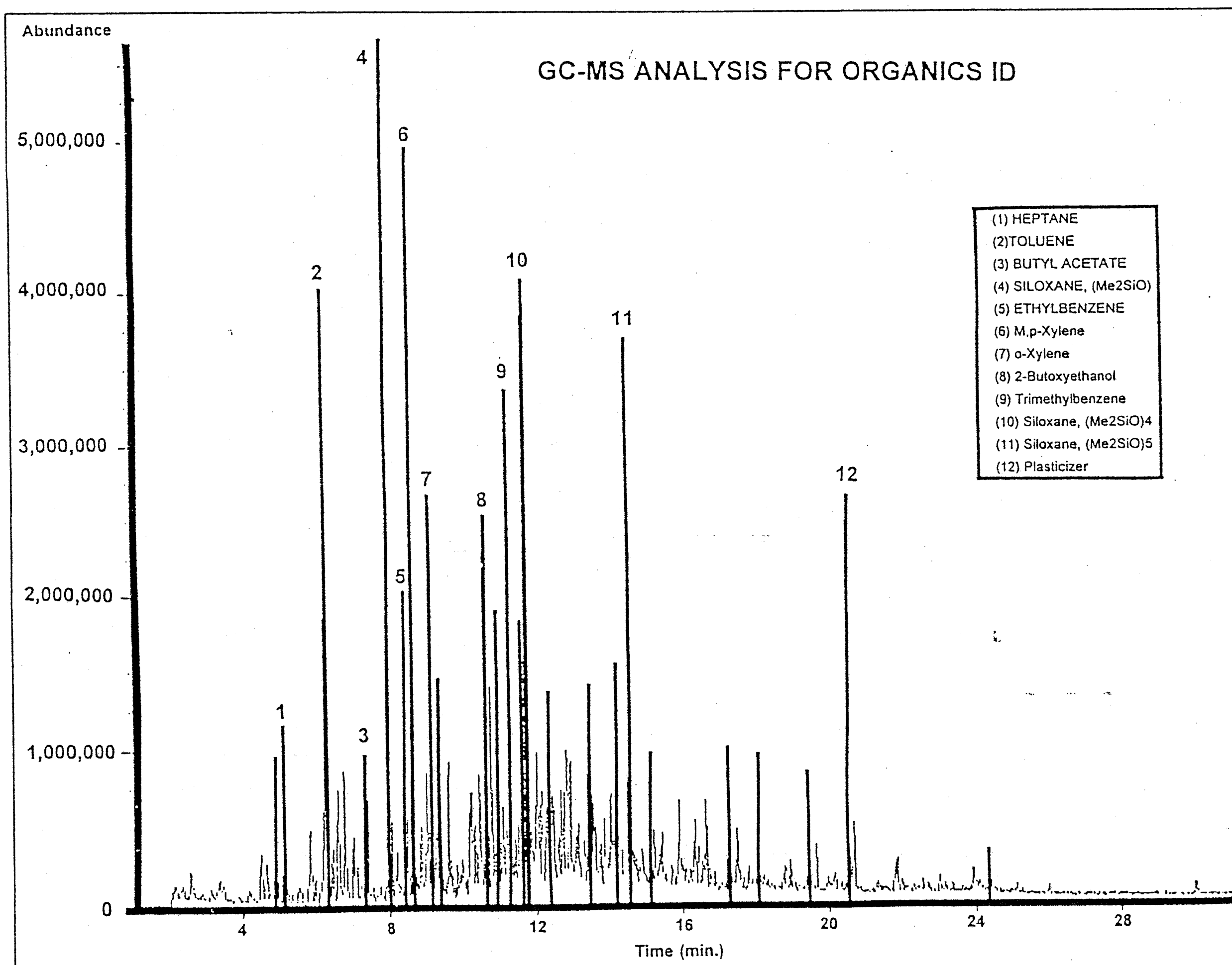

Fig. 2. Organic compounds found in a cleanroom

To help reduce organics from outside air, several manufacturing facilities have replaced their present HEPA filters with organic adsorbing HEPA filters. Although these are effective, they need to be monitored. Their lifetime as organic adsorbers is related to the quantity of organic adsorbent they contain and the quantity of organic materials they encounter. Consequently, rather than being effective for years, their lifetime could be months.

Particle measurements in cleanrooms are becoming increasingly difficult as cleanrooms improve, but they are nonetheless important. A new particle sampling device, which is 30 times more efficient than witness wafers, has been developed and is presently being tested. The sampler, which is a one-stage degenerate particle impactor, uses clean wafers as the collector (see Fig. 3).

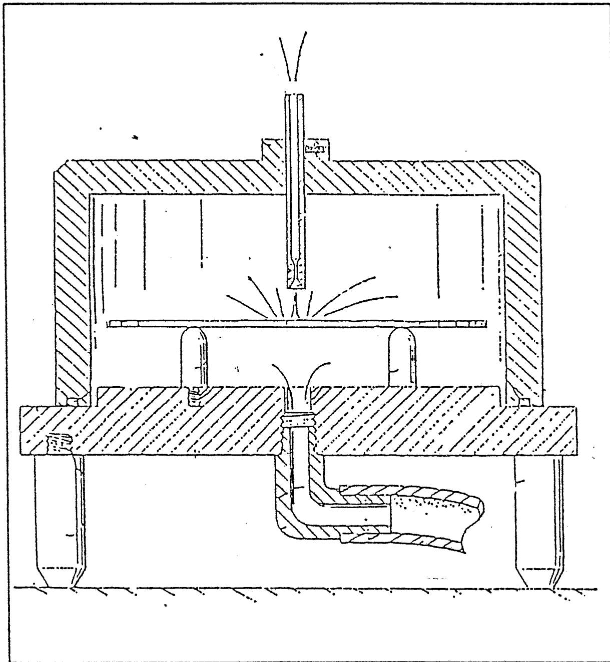

Fig. 3. Particle Sampler

The sampler pulls air into the unit and is run for several hours. The particles, as they enter the collector, undergo selective separation based on their size and density. They consequently impact the wafer at different distances from the center of the wafer. Furthermore, rather than being evenly distributed across the whole wafer, as is the case when using witness wafers, they are concentrated in an area of approximately four square centimeters (see Fig. 4). Analysis of these particles, for both size and elemental composition, can be done both quickly and easily in a SEM because they are concentrated in a small area at a location around the center of the wafer.

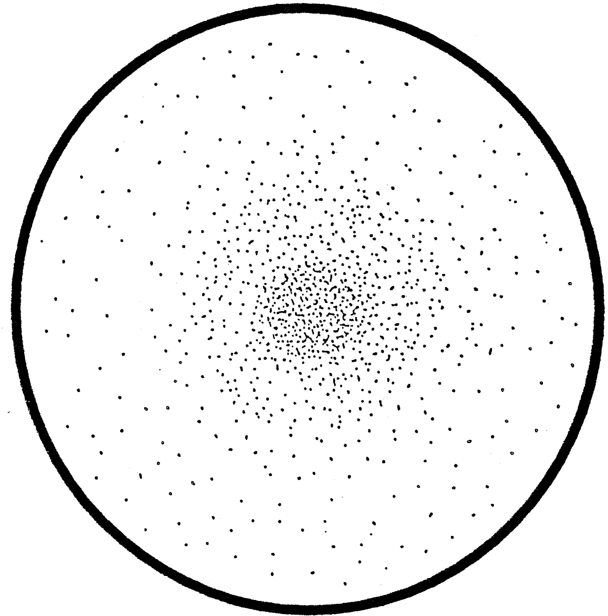

Fig. 4. Particle distribution on wafer used in particle sample

CONCLUSION

Using scrubbing, adsorption, and adhesion technology in three different types of cleanroom samplers, more sensitive measurements and identification of types and levels of contamination are possible. In using these devices in cleanroom fabs, it was found that cleanrooms have, in many cases, significant levels of acid fumes outside of the fume hoods, minor metallic contamination levels, and higher particle contamination than was believed for class 10 and class 100 cleanrooms. It was shown that the air scrubbing unit was especially useful for tracking metallic contamination sources. When utilizing these testers with other kinds of tests such as metals in wet station baths, chemicals and ultrapure water, contamination can be identified at each exposure step whether in a reactor, in air (cassette), or in a wet process. Through proper identification, these contaminants can be removed.

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Table 1. Metallic Concentrations Class 100 Cleanroom

(Element) in µg/m^3	Location 1	Location 2	Location 3
Na	0.001	0.007	<0.001
Mg	<0.001	0.001	<0.001
Al	<0.002	<0.002	<0.002
B	0.056	0.022	0.024
Ca	<0.07	<0.07	<0.07
K	<0.1	<0.1	<0.1
Fe	<0.01	<0.01	<0.01
Ni	<0.001	0.001	<0.001
Si	0.02	0.05	0.04
Cu	0.002	<0.002	<0.002

Table 2 Non-Metallic Concentrations Class 100 Cleanroom

(Element) in µg/m^3	Location 1		Location 2		Location 3	
	Feb. '94	May '94	Feb. '94	May '94	Feb. '94	May '94
Ammonium	61	7.9	12	4.4	3	2.9
Floride	28	1.4	10	1.1	2.9	1.6
Chloride	0.14	<0.01	0.045	<0.01	0.14	0.16
Nitrite	33	3.3	9	1.4	6.9	3.7
Phosphate	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Bromide	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Nitrate	3.1	0.67	0.94	0.4	1.6	0.82
Sulfate	0.14	0.04	0.16	<0.02	0.51	0.25