Beta Test Proves Mass Spec's Mettle

Robert J. Thomas

How a collaborative process verified performance and ruggedness.

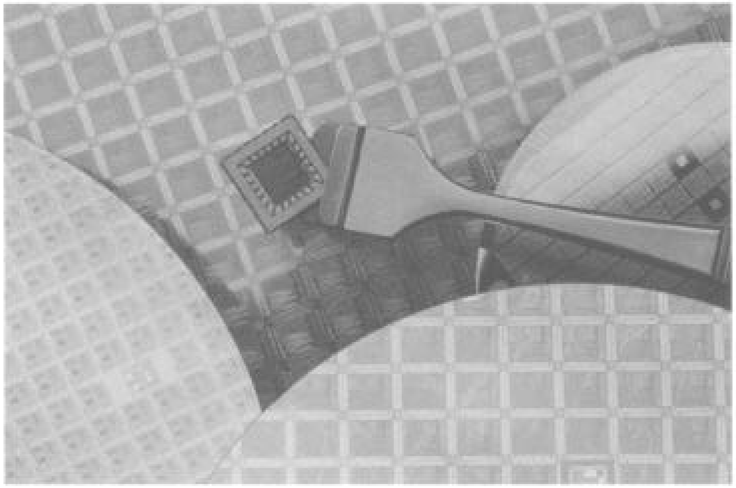
Beta testing has been a well-proven method of fine-tuning and improving the design of a product before it is introduced to the market. Instead of just "throwing a product over the wall," a company hand-picks a few strategic customers to put an instrument through its paces, normally about 6–12 months before it is introduced. The main purpose of beta testing is to see how a piece of equipment stands up to the demands of a real-world laboratory. A great deal of valuable information can be gathered, such as the robustness of the hardware, the ease of use of the software, and an instrument's strengths and weaknesses in solving certain application problems.

A recent collaboration between the Chemical Analysis Division of PerkinElmer (Norwalk, CT) and Air Liquide America Corp. (Dallas), a manufacturer of high-purity chemicals and gases used in the semiconductor industry, shows the benefit of beta testing. PerkinElmer, in conjunction with its joint venture partner, SCIEX (Concord, Ontario, Canada) had developed an inductively coupled plasma (ICP)-mass spectrometer for carrying out trace element determinations. The instrument, which was designed primarily for the semiconductor industry, used a dynamic reaction cell (DRC) to eliminate polyatomic spectral interferences and improve detection capability. Although they knew that this technology gave exceptional detection limits, PerkinElmer scientists wanted to make sure that it would perform in a routine laboratory environment, running real-world semiconductor-type samples. They felt it was important not only to test the ruggedness of the system but also to understand how the DRC technology would handle the application demands presented by semiconductor samples. This was critical because of the analytical problems associated with the determination of ultratrace levels in concentrated and corrosive electronic-grade chemicals by ICP-MS.

Contamination Is a Concern

Trace element contamination is a major problem for the semiconductor industry. Any contamination in the high-purity chemicals used in the semiconductor manufacturing process can dramatically affect the performance of the semiconductor device. This ultimately affects product yield and the profit margin for the device manufacturer. The problem is further compounded by the lengthy sample preparation steps involved in readying the chemicals and reagents for analysis.

Air Liquide faced this problem with its existing technology. According to Anthony Schleisman, manager of the Atomic Spectroscopy Development Lab of Air Liquide, "At the purity levels we work with in the semiconductor business, any sample handling, whether it is pipetting, evaporation, sample transfer, or addition of reagents, can lead to contamination and false test results." Air Liquide's problem is typical of manufacturers of the semiconductor devices and suppliers of high-purity chemicals to the industry. The semiconductor industry is demanding purer chemicals to produce larger wafers and narrower line widths, and the analytical instrumentation used to measure the contamination levels is struggling to keep up with them.



Lower Detection Levels Needed

However, the semiconductor industry is caught in a dilemma, because the *National Technology Roadmap for Semiconductors* produced by the Semiconductor Industry Association predicts only a small demand for increasing the purity of these process chemicals. But as Schleisman explains, "Even though some of the industry trends suggest that the chemical purity won't need to improve for the next few years, we have seen a number of problems in our industry regarding purity levels below the 'roadmap' levels." He adds, "Fifteen years ago, the purity level of chemicals was not as critical, and chemical specifications near the parts-per-million level were sufficient. Over the years, however, the purity requirements have increased such that laboratories must be able to detect contamination at the low parts-per-trillion to parts-per-quadrillion range."

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In fact, several critical chemicals used today have trace element guidelines at the 10-ppt level. The Semiconductor Equipment and Materials International (SEMI) Standards organization, in its *Book of Semiconductor Standards (BOSS)*, states that before a guideline can become a specification, there must be a documented spike recovery study showing a recovery of 75–125% at 50% of the specified level. This means that if a concentrated chemical has to be diluted 10× to be introduced into the instrument, a 10-ppt guideline level is equivalent to 500 ppq in solution. The challenge for instrument manufacturers is developing instrumentation that can routinely measure these contamination levels.

Another reason for the increased need to measure higher purity levels is that the semiconductor industry is switching to less concentrated acids and chemicals. Semiconductor manufacturers are mixing the reagents with ultrapure deionized water, a less contaminated ingredient, to obtain a higher purity chemical. "This leads the industry to falsely believe that purity levels of the chemicals do not need further improvement," says Schleisman. "But I have seen cases of contamination in the low parts-per-trillion to parts-per-quadrillion levels in the dilute solutions that adversely affect the semiconductor wafers."

"Our customers constantly tell us of the pressures they are under to qualify and specify reagents used in the semiconductor manufacturing process at lower and lower levels," says Eric Denoyer, technology manager for ICP-OES and ICP-MS at PerkinElmer. "To maintain their competitiveness, our customers must measure trace element impurities at the low-ppt to high-ppq levels. Although the levels measured today are probably sufficient to produce high-quality electronic devices, we see reagent manufacturers pushing levels even lower by providing higher purity chemicals than the SEMI Standards organization currently specifies. This drives the chemical manufacturers to provide purer reagents in order to maintain a competitive edge. So, whether for technical or competitive market pressures, the semiconductor industry sees trace element impurity requirements dropping to the sub-ppt range."

DRC Technology

DRC technology provides semiconductor laboratories with the ability to detect and measure elements such as iron, calcium, potassium, arsenic, chromium, and other critical elements at ppt and ppq levels—a capability historically degraded by interferences from argon-derived species. By removing plasma and solvent- and matrix-based polyatomic species before they enter the mass spectrometer, some of the most problematic spectral interferences associated with ICP-MS are eradicated, allowing significantly lower detection capability than traditional instrumentation. In addition, the system uses a normal-temperature plasma to eliminate the drawbacks of lower temperature approaches and to provide increased sample throughput and more efficient multielemental analysis. A new quartz sample introduction system also ensures quality measurements at ppt and ppq levels by greatly reducing sources of contamination.

"The DRC will help reagent manufacturers supply a better product by allowing them to institute more strict quality assurance/quality control [QA/QC] specifications. This not only makes their product offerings more competitive, but also benefits the industry overall by increasing product yield," says Denoyer. "Because this technology detects impurities at lower levels than before, it allows closer inspection of incoming raw materials into a semiconductor fabrication [FAB] plant. This closer inspection will help improve FAB output yield and, as importantly, help avoid extremely costly plant shutdowns due to process contamination."

The Beta Test

A large part of the success of the development of this instrument can be attributed to the beta site agreement between Air Liquide and PerkinElmer. The instrument was tested for several months at Air Liquide's Technical Center. "Before the instrument was shipped, both companies agreed on well-defined goals and milestones, which is absolutely essential for a collaboration of this type to work," explains Denoyer.

"We agreed to be a PerkinElmer beta site because we had all the lab capabilities, the clean room, and the necessary experience with ultratrace analyses," says Schleisman. "But the driving force for me," he continues, "was the increased capabilities and competitive edge that the instrument would give our own company." He adds, "Our goal from this beta site test was the removal of polyatomic interferences so we could run most of our analyses using an ICP-MS under normal plasma conditions. The DRC would thus eliminate the need for the determination of elements like sodium, calcium, potassium, and iron by graphite furnace atomic absorption."

The beta site experience began smoothly with an efficient instrument installation process that took less than three hours from delivery to the time it was operational. "Electricity was hooked up to it and it was pumping down by 5 o'clock that afternoon. When the instrument was started up the next morning, the detector immediately saw ions. The installation was very, very fast," Schleisman said.

The Results

To supply its customers with the necessary raw material purity levels, the Air Liquide laboratory analyzes several matrices, including deionized water, hydrochloric acid, sulfuric acid, ammonium hydroxide, and hydrogen peroxide. Many of these matrices produce polyatomic spectral interferences that cause problems in determining elements such as iron, nickel, and chromium. The goal is to eliminate the polyatomic interferences to achieve better detection limits and faster analyses. Though many scientists acknowledge that there are various ways to circumvent the interferences, Schleisman prefers to remove them with the least amount of sample preparation or instrument manipulation. "To improve detection limits using our current ICP-MS technology, we have to eliminate polyatomic interferences by removing the matrix and concentrating the analytes. Unfortunately, as all laboratories know, sample preparation can lead to sample contamination," he explains. "The DRC technology is suited for the semiconductor industry because it can reduce sample preparation, thus minimizing the possibility of contamination. We have used the DRC to determine ultratrace and trace metal contaminants at the low-ppt to ppq levels in concentrated hydrochloric acid with just a simple dilution. The instrument has more than met our expectations."

PerkinElmer introduced the DRC technology on its ELAN ICP-MS platform at the 1999 Pittsburgh Conference where it won the Pittcon Editor's Gold Award for the most innovative product at the conference. The beta testing of the instrument by Air Liquide went a long way in helping PerkinElmer to better understand the DRC, in particular its application capabilities related to the analysis of high-purity semiconductor chemicals.

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