Advanced analytical spectroscopy for electronic specialty gas

A manufacturer's perspective on 2013 and beyond

By Lisa Bergson

The Semi companies all have their own super-secret development plans," observes Tracey Jacksier, director of Air Liquide's Analytical Sciences Core Global Lab. "Now they're looking at new gases that weren't even on the list 15 years ago." Not just novel gases, but, increasingly, new liquid chemicals are coming into play as Semi's major fabs move to 20nm and lower technology nodes, and prepare for the vast paradigm shift to 450mm wafers. As 'Moore's Law' predicts, the number of transistors on a chip will double approximately every two years.

Accordingly, Jacksier and her rivals are engaged in a vigorous, highly competitive race to find viable, economic means of analysis to keep pace with emerging requirements, ranging from the adoption of sulfur-containing gases to the enhanced hydrogen purity needed for EUV (extreme ultraviolet) lithography, to the development of new organometallics to the growing use of germane and barrier level films.

Much of the work is transpiring on the raggedy edge of invention, where hazards abound, and analytical resources and expertise are limited. Indeed, there is a sizable gap between the capabilities of even the most advanced commercial analyzers and the emerging needs of an industry in rapid transition.

"Technology must evolve to include the ability to analyze liquids at the same level and detail that you analyze gases," remarks Michael A. Pikulin, the senior vice-president for Business & Technology Development at Voltaix LLC. "At DuPont they don't think of chemicals in terms of '5 nines' (99.999 percent) and '6 nines' (99.9999 percent), but Semi requirements call for that."

Today, materials makers and their analytical suppliers enjoy a myriad of opportunities, with so many new applications and technologies coming to the fore. Jacksier notes, "As the industry changes, you always need people to think outside the box."

Taking stock of where we are

Within the world's leading fabs, an almost preternatural calm belies the intense effort that goes into maintaining a consistently stable and immaculately clean environment for the production of their delicate chips. Laser-based analyzers, such as those based on Tiger Optics' Continuous Wave Cavity Ring-down Spectroscopy (CW CRDS), are used for moisture, oxygen and, increasingly, methane detection. So versatile, cost-effective and powerful are the new generation of analyzers that they have fundamentally changed the way measurements are conducted.

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By contrast, traditional techniques, whether FTIR, Raman, electrochemical, quartz-crystal oscillator or gas chromatography suffer variously from severe limitations, be they cost, drift, interferences, operational complexity or truncated dynamic range.

Typically, advanced spectroscopic analyzers offer a multitude of advantages that include:

- Ease of start-up and operation: "Someone with a Bachelors degree can use Tiger Optics analyzers independently, without high-level involvement," says Jacksier. This translates into significant savings, with the ability both to deploy PhDs to more value-added endeavors and to rely upon less trained personnel, who are often more readily available in the remote corners of the world where fabs and their gas suppliers increasingly find themselves.
- Consistency: That's the mantra as more fabs strive to achieve product uniformity by controlling every aspect of their materials and processes. As Bob Ford, the Bulk Specialty Gas manager for Air Products and Chemicals Inc., says, "The top tier of customers have a level of sophistication in their organization, with a whole set of rules, processes and procedures that drive consistency."
- Continuous real-time measurement: When it comes to contaminants, fabs track and react to very small perturbations as they strive to preserve their vulnerable products. Plus, with its wide dynamic range (over four orders of magnitude), CW CRDS allows fabs to monitor the full excursion of a contamination event. "With Tiger Optics technology, you can perform on-line process control continuously," Jacksier says.

Time waits for no-one

Nonetheless, when it comes to evolving Semi processes, there are key points where even the most advanced new commercial analyzers fall short. As the specialty gas companies evolve as specialty chemicals suppliers, they are under growing pressure to meet increasingly stringent specifications on new and exotic materials.

Our research points to the growing need for matching instrumentation that addresses the following critical needs:

- Versatility: "New materials and smaller device geometries require etchant molecules with increasing selectivity and process control," says Paul Stockman, Linde's commercialization manager, who describes the development of analytical methods for the myriad new materials as a Herculean task. At Voltaix, Pikulin concurs. "These are new molecules; you can't just call, say, Agilent and get information," he says.
- Lower measurement levels: On the whole, Semi's move to smaller and smaller integrated circuits accentuates requirements for cleaner materials. With the shift to EUV, for instance, the spotlight will be on hydrogen purity, which is used at a rate of 400 liters per minute to protect the sensitive lenses this process utilizes, according to Terry Francis, president of industry consultancy, TA Francis Associates.
- Multi-species detection: End-users clamor for broader measurement capabilities—the more species the better. Jacksier says she is eager to test Power + Energy's new Hydrogen Elimination Mass Spectrometer, while at Air Products, Suhas Ketkar, manager for Advanced Analytical Technology in that company's Electronics Division, dreams of implementing Quantum Cascade Lasers to "open up new applications."
- Standardization: National metrology institutes and instrument makers face a growing mandate to create more refined standards for an industry beset with conundrums. "Can you get the same answer with different types of equipment, for example, a 17 percent silane mixture, trace phosphine in arsine…?" asks Jacksier.
- New approaches to sample handling: The specialty liquids now in development tend to be toxic, air-sensitive and used in small quantities. At Air Products, Ketkar says he seeks a "sensitive spectroscopic technique in a closed cell" for analytical purposes, particularly when usage of these materials goes online. Otherwise, he and others must rely on complex, lab-based systems, employing gas chromatographs and ICP mass specs, which often pose "a challenge just to get the sample into the instrument," according to Ketkar. "I can't lose things that I want to eventually look for," he says.

Faced with such challenges, developers of new gases and chemicals must improvise.

" 'I don't feel like doing it', is never an answer," states Jacksier. For now, she and her competitors often find themselves contending with analytical technologies that are at a pre-commercial phase, when – in her words – "The user needs to do more because they're new, and the vendor hasn't been able to figure everything out yet."

For experimentalists and makers of advanced spectroscopic instruments alike, the operative word is 'yet'. SGR

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