Chemical Sensors

A Perspective of the Present and Future

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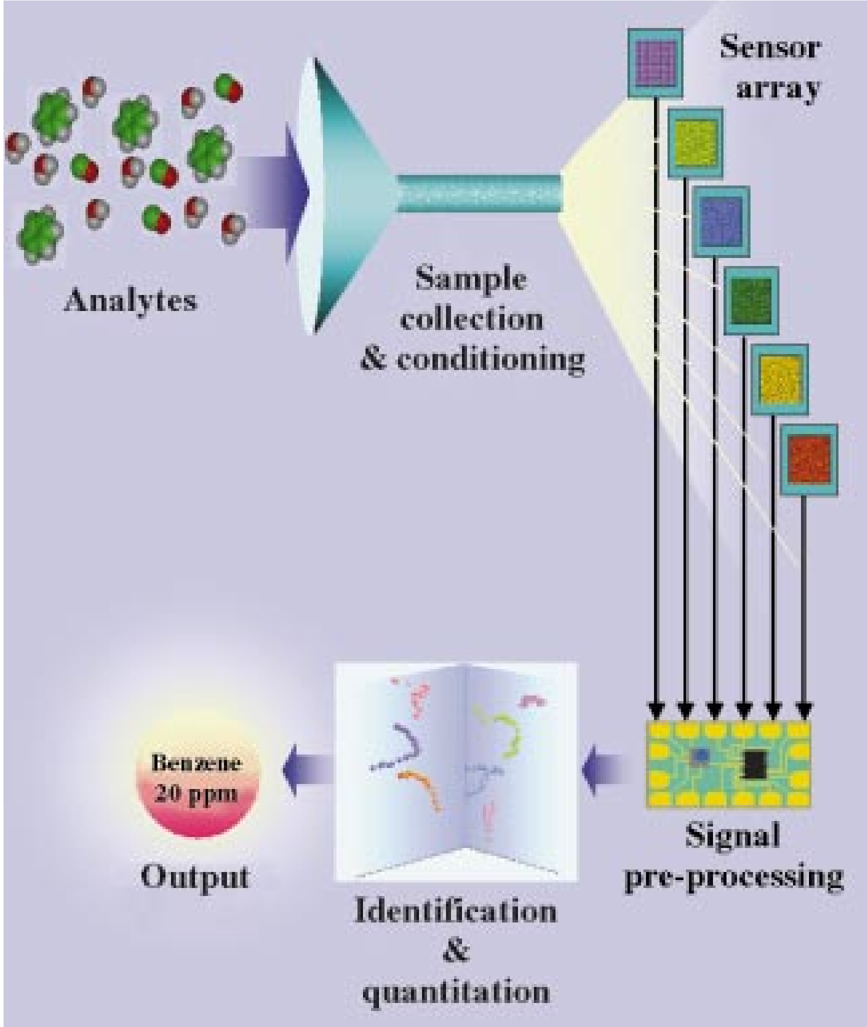

Schematic representation of a chemical sensor array system, tracing the steps from analyte collection to response output. The "sample collection and conditioning" step may include preconcentration and/or preseparation. The "sensor array" can include from a few to tens of sensors, often based upon the same physical transducer platform, although multiple platform types can be advantageous. To provide greater accuracy and robustness of response, the array may include some proportion of intentionally redundant (nominally identical) sensors. In addition to such functions as analog-to-digital conversion, filtering of noise, and multiplexing, the "signal pre-processing" stage can streamline the output from the sensors by averaging identical elements, eliminating "out of range" responses, normalization, scaling, etc. The "identification & quantitation" steps typically utilize some form of pattern recognition to classify the response as one of the "known" (previously measured and calibrated) analytes; better methods can also identify a response pattern that does not match any known analytes, rather than making an incorrect identification. Depending on the method chosen, quantitation can be performed simultaneously, or as a separate step following analyte identification. "Output" can take many forms, from a simple indicator light or alarm to the display of analyte identities, concentrations, probability that the identification is accurate, and related information.

To a greater extent than in many areas of endeavor in the chemical sciences, successful chemical sensors require a high level of interdisciplinary collaboration and effort, along with an unusually close coupling between the ultimate application and the R&D process. The tremendous growth in chemical sensor R&D over the past ten years has been spurred by everything from fundamental advances in interfacial chemistry, to new microscale engineering technologies, to a demand for cleaner, more efficient, better-controlled industrial processes. Figure 1 presents the numbers of refereed papers published (in English) in the chemical sensors field from 1988 until the present, as collected by Janata and colleagues in the course of several reviews.[1] To better understand the underpinnings of such growth and, more importantly, to anticipate the critical needs and potential rewards of the future of this dynamic field, groups of 20-30 scientists met in May 1997 and in July 1998, at National Science Foundation-sponsored workshops, to critically evaluate the chemical sensor field.

This article summarizes and presents trends based upon the first-hand experiences of the participants in those two workshops as they grapple with transferring the fundamental research of chemically sensitive interfaces from concept, to laboratory, to the shelves of commercial vendors in the form of marketable products. We believe that this is an important paradigm for much of the research in chemistry that will be pursued as we begin the next millennium—research that expands our knowledge of chemistry at the most fundamental levels, while simultaneously coupling it to the needs of real-world applications and to a broad range of scientific and engineering disciplines outside the traditional confines of chemistry. We hope this article will help to answer such critical and oft-asked questions as "Who cares about chemical sensors?", "Where does the funding come from?", and "What's the best sensor?".

Practical Issues: Commercial Aspects of Chemical Sensors

There are a number of chemical and biochemical sensors that have been successfully developed. These include ion-selective electrodes, glucose sensors for monitoring diabetics, amperometric sensors for toxic gases such as Cl_2 and CO, industrial ISFET pH sensors, high-temperature zirconia oxygen sensors used in automobiles, and semiconducting oxide sensors and catalyst-loaded ceramic beads for combustible gases. More recent developments have made use of microfabrication technology to manufacture a variety of sensors, including many of those described above.[1,2]

A clear requirement for the successful commercialization of these devices is a need, either established or successfully predicted, for the sensor. In addition, the sensor technology selected has to offer an advantage over previous technologies. Successful implementations have been those where the use justified the development cost. From a technical perspective, the successful sensors were those that were developed with a firm understanding of the basic underlying science. In contrast, less successful commercialization efforts have been those in which development cycles have been long because of a lack of understanding of the necessary materials or technologies.

Among the barriers to successful development are technological issues, technology transfer, funding in the early stages of development, communication among the various stakeholders, and establishment of the required, effective interdisciplinary partnerships. In both technology transfer and the development of interdisciplinary teams, the wide gap between academic and industrial modes of sensor development needs to be effectively bridged, as does the gap between the different technical and non-technical disciplines that must collaborate. Often another technological gap separates a successful laboratory sensor from the field application. Present experience indicates that even in the research stage, it is important to consider how a sensor might be packaged and manufactured if it is to reach the marketplace quickly. Commercial experience further shows that academic research directed toward improving sensor subcomponents (e.g., a particular type of chemically sensitive interface material), rather than attempts to devise and construct completely new types of sensors, more often proves to be commercially useful. Nevertheless, the fact that sensors are functional parts of larger systems, not isolated components, must be kept in mind if development is to be rapid. In many cases, it is the overall system that must meet cost and performance issues, not just the chemically selective coating or the transducer.

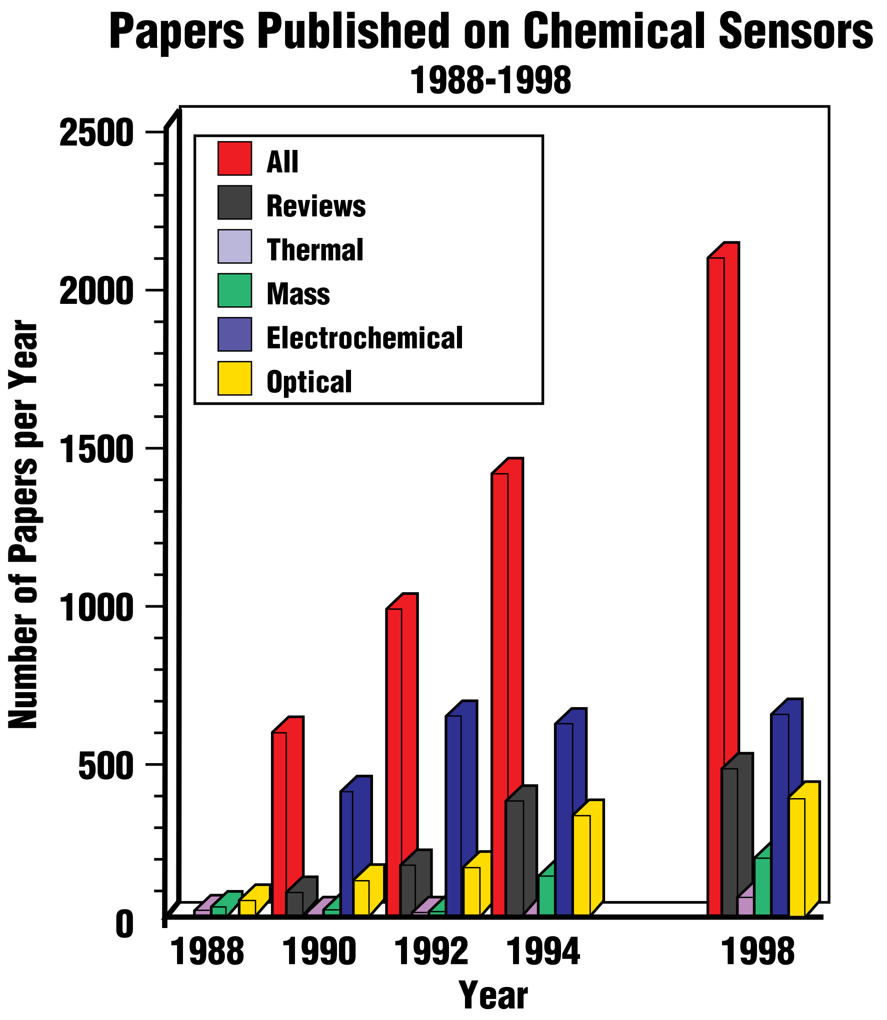

FIG. 1. Number of papers published in English, organized by year and by topic area, in the field of chemical sensors. The year of the published review is given on the abscissa, and the data in each case are from the two years preceding. (No review was done in 1996.) The details of the search and selection criteria used to obtain these statistics are reported elsewhere.[1]

An issue that often delays commercialization is acquisition of the intellectual ownership of all the technologies incorporated in the complete system. Academic and other non-commercial researchers should strive for a stronger appreciation of what constitutes an invention. In addition, they should be aware that many of the enabling technologies used in their own research and development efforts may involve intellectual property owned by others. Successful commercialization can be impeded until all the ownership issues are resolved.

In the process of transferring technology from an inventing institution to the product developer, there are significant cultural, technical, personnel, and resource differences. Moving ideas between entities works best when accompanied by the transfer of personnel and the availability of funding. Developing mechanisms for completion of the projects or transfer of people is critical. The establishment of formal technical collaborations is a powerful means to break down the barriers between partners in sensor development. In these partnerships, specifications, practical goals, and problems are raised; students receive training that includes a practical perspective; new scientific and technical skills are transmitted; and a direct connection is made between industry and academia.

The issue of understanding the windows of opportunity for bringing a product to market is complex and expedient. If a technology possesses an obvious advantage that can be exploited, commercial developers will move quickly to turn it into a product. Formation of product-development teams at an early stage, bringing together technologists and business experts, allows a group to move forward quickly once the business opportunities emerge; this can help the developers retain the competitive advantage they have worked so hard to gain. Within the academic fora, inclusion of industrial expertise within interdisciplinary teams at an early stage in technology development provides needed market savvy.

Funding

Sensor research is intrinsically an applied science and its ultimate success is indicated by commercialization of the technology. Accordingly, much of the funding for sensor research has been need-driven, with the exception of fundamental research directed toward the development of chemically-sensitive interface materials. Small companies have typically been the vehicles by which sensors are brought to the marketplace, a consequence of the fact that a sensor may have utility only in a niche, low-volume market that is not of interest to a major company. As a result, competitiveness in chemical sensor commercialization is largely dependent upon the creation and support of small companies. By virtue of their size, small companies often lack the necessary financial resources to bring a sensor system to market independently; funding by government programs and transfer of expertise from academic or government laboratories is often critical. General awareness of the existing funding modes, which are designed to facilitate collaboration between academic and government laboratories and industry, needs to be enhanced.

In the U.S., there are three major groups of government programs that support R&D in this area. One primarily supports industry, and includes SBIRs, STTRs, and ATP. Another group focuses on military applications, supporting industry, academia, and government laboratories, and this includes DARPA, ONR, ARO, AFOSR, and DoD. The third group also supports academia, government laboratories, and industry; it includes NSF, and NIH (these two having a largely academic focus), as well as NASA, DoE, EPA, OSHA, USDA, DoJ, the intelligence agencies, DoT, and the Technology Support Working Group (an interagency body). At present there is much more financial support from government agencies than from the private sector for fundamental research; the highest risk research is almost entirely supported by government agencies. For applied research and particularly for development, the total amount of support from the private sector is significant, though difficult to quantify.

One reason that small companies have not been more successful in commercializing chemical sensors is that they typically are not capable of developing complete systems independently. The FIFTH Framework, the European Union’s R&D funding program (http://www.cordis.lu/fifth/home.html) , has addressed this issue by encouraging joint collaborations and ventures between academia and small and medium-sized enterprises throughout the EU. In the U.S., the only government funding program encouraging such joint ventures is NIST’s Advanced Technology Program.

Key to Acronyms

AFOSR	Air Force Office of Scientific Research
ARO	Army Research Office
ATP	Advanced Technology Program (administered by NIST)
CMOS	Complementary Metal Oxide Semiconductor
DARPA	Defense Advanced Research Projects Agency
DoD	United States Department of Defense
DoE	United States Department of Energy
DoJ	United States Department of Justice
DoT	United States Department of Transportation
EPA	United States Environmental Protection Agency
EU	European Union
NASA	National Aeronautics and Space Administration
NIH	National Institutes of Health
NIST	National Institute of Science and Technology
NSF	National Science Foundation
ONR	Office of Naval Research
OSHA	Occupational Safety and Health Administration
R&D	Research and Development
SBIR	Small Business Innovative Research
STTR	Small Business Technology Transfer
USDA	United States Department of Agriculture

Communication and Effective Technical Meetings

The field of chemical sensing is inherently interdisciplinary, and consequently effective communication is absolutely critical to enhancing the success of chemical-sensor research, development, engineering, and commercialization; technical meetings and workshops play an important role in this exchange of information. In addition to having a positive impact on the economy and industrial competition, enhancing communication within the chemical-sensor discipline contributes to interdisciplinary educational efforts.

The ever-growing number of technical meetings with symposia related to chemical sensors is a well-recognized problem: the subject areas and speakers often overlap significantly, causing moderate-to-extreme dilution of new technical results, lower average paper quality, missed opportunities for key researchers to communicate face-to-face, and costing much time for those active in the field. In addition, the objectives of a symposium are not always clear, the users of sensor technology are typically under-represented, and many symposium proceedings papers report already-published results. The interdisciplinary nature of chemical sensors exacerbates many of these problems, because many separate technical disciplines can (rightly) claim to be “owners” of an interest in the sensor field. More effective communication within the sensor community can address a number of these concerns.

Technical Issues: Chemically Sensitive Materials

There is no single, best approach for developing a selective chemical sensor or sensing system. The application and conditions of use dictate the most suitable approach. The environment in which the sensor must operate plays a strong role in determining appropriate materials and transducing principles. For example, environmental conditions that must be considered include temperature; presence or absence of possible chemical interferences; fouling, harsh or corrosive conditions; and electromagnetic interference. The application needs include the desired limit of detection, dynamic range, precision, stability, selectivity, and lifetime. In addition, power requirements, size, weight, cost, ease of use, and safety in the measurement environment will also be considerations. The key is to match the appropriate material or set of materials to the appropriate sensor platform(s), based on a well-founded scientific understanding of material properties, analyte-material interactions, application environment/material interactions, and transduction mechanisms.

Candidate materials for chemically sensitive interfaces include polymers, organic monolayers, ceramics, metals, semiconductors, composites, organic receptors, porous materials (molecular sieves, sol-gels, aerogels, and composite aerogels), biomolecules, and combinations thereof, such as supramolecular architectures. Materials deemed unsuccessful in molecular recognition chemistries (because they are insufficiently selective) or oxidative catalysis (because they are poor partial oxidation catalysts) offer a vast base of potential chemically sensitive materials for sensors: the selectivity requirements for multielement sensor arrays are significantly less stringent than for "perfect" molecular recognition or high-selectivity catalysis.

Adequate physical and chemical characterization of the sensing material, as prepared on the sensing platform, is essential because understanding of the bulk material structure and properties may or may not be relevant to the materials interfaced to transducer platforms. In addition, all surfaces that the sample encounters prior to the chemically sensitive material should be assessed as potentially reactive interfaces. Approaches for enhancing selectivity and/or sensitivity include: (a) highly selective molecular recognition of the analyte(s), which may involve polyvalent association; (b) sample pre-treatment to remove potential interferents and preconcentrate the analyte; (c) manipulation of interfacial architecture on the molecular scale, including both physical and chemical site heterogeneity (an increase in surface area is one example); (d) monitoring the kinetics of the reaction in situations where kinetic control is operative; (e) the use of sensor arrays involving multiple coating/transducer sets; and (f) so-called "higher-order systems," such as a sensor array whose response is recorded and analyzed as a function of temperature.

Single, perfectly selective sensors may not be feasible or desirable for all applications. While single-element sensors provide only a single data point, sensor arrays provide multiple data points per sample (a vector of data) that can provide additional chemical information to differentiate multiple analytes and discriminate against interferences. Sensor array systems can provide two (or more) vectors of data per sample, as in the example of measuring an array of sensor responses as a function of temperature. Information content can sometimes be further enhanced using modulation techniques, in which, for example, the temperature of the sensor or the input concentration is deliberately modulated and the effect upon sensor response recorded. Arrays and other higher-order sensing systems can be based on a single material class on a single type of transducer platform, or various types of materials combined with one or more sensor platforms. Whatever the approach, fabrication of sensing materials in a reproducible manner that is compatible with the chosen transduction platform, as well as the analyte and application, is mandatory. Furthermore, it should not be assumed that the response of a zero-order sensor predicts its behavior in a multiorder sensing system. Therefore, it is necessary to characterize all materials used in an array or higher-order sensor over the entire range of potential operating conditions.

The use of a sensor array is a potential solution to a variety of problems associated with using chemical sensors in viable, practical systems. In contrast to, e.g., image processing, using more than one chemical sensor in a sensing system does more than merely enhance the discrimination capability or resolution of the array. Multiple sensors can be used to address the following issues.

Robustness of concentration information.—Multiple sensors that are fabricated under the same conditions and then operated under the same conditions should ideally produce identical outputs. However, due to drift, fabrication mismatch, irreversible reactions on the sensor surface, and similar effects, the actual sensor outputs from a homogenous group can vary widely. Placing replicate sensors of each type in an array of chemical sensors allows imperfections in fabrication, operation, and aging to be quantified and often compensated using signal processing.

Chemical discrimination.—Due to nonideal selectivity and overlapping specificities among sensors, higher order sensor arrays allow pattern-recognition engines to detect a variety of analytes in the environment. Ideal materials candidates for use in arrays should have moderate but complementary selectivity to the target analytes, which enables an array of sensors with overlapping selectivities to be fabricated. This radical change in selectivity requirements (compared to the near-perfect selectivity required for a one sensor/one analyte system) must be communicated to researchers specializing in the synthesis of molecular recognition materials, because "poor" recognition materials that may have a selectivity in the range of "only" 5-500 can be ideal candidates for use in arrays. The ideal number of heterogeneous (different) sensors in an array has been suggested to be between three and eight for the various chemical sensing applications addressed to date.

Distributed sensors.—Multiple arrays consisting of both heterogeneous and homogeneous subgroups of sensors can be distributed at multiple locations in an environment to locate chemicals, evaluate gradients, and determine chemical identity.

Transducer Platforms and Materials Integration

Whether the sensor system is based upon individual devices or an array, transducing approaches and devices can include mechanical (acoustic wave, micromechanical), electrochemical, optical, thermal, and electronic types. Each has strengths and weaknesses relative to the particular application. Each transduction principle can be implemented in a variety of configurations, and fabricated by multiple approaches, resulting in many different platforms. If the market is large enough to justify development costs, a single-analyte sensor may be suitable in applications where the analyte has properties that permit selective recognition in the environment to be probed. For needs with smaller markets, a sensor or sensing instrument may need to be more versatile so that it can be successfully applied in multiple markets. A sensor array provides a flexible approach for developing a sensing instrument that can be adapted to many applications. Regardless of the approach chosen, the engineering design of the entire sensing system must be considered at the beginning of the platform design process.

The application of chemically-sensitive films to sensor array platforms can be accomplished in one of two ways: "pick and place" and "local definition." In the former, several wafers, each bearing a distinct chemically-selective coating material, are prepared and diced. The resulting chips bearing the different coatings are then individually integrated into the sensor array system by attachment to a common substrate; one defective chip can be replaced without sacrificing the entire array. Each chip exposes an edge which, in the case of conductive platforms, is electrically live when diced, and therefore must be encapsulated for liquid-phase applications. The second approach involves selected-area delivery or definition of each of the chemically sensitive materials via microdispensing, screen printing, stenciled evaporation or spraying, selective-area chemical-vapor or electrochemical deposition, lithography, photochemical reaction, or localized doping, onto the active region of a single sensor element, of which there may be many in an array. The second approach is less demanding of the platform, since there is no need to integrate (i. e., physically, optically, or electrically interconnect) separate chemically sensitive "subchips" onto the main platform. However, the requirement for yield is more stringent, since a single defective coating may render an entire multi-coating array chip useless. Regardless of the approach used to provide the chemically sensitive interface, as much encapsulation as possible should be done at the wafer level.

Data Analysis

Many applications may be best addressed using chemical sensor arrays. The advantage of using multiple sensor devices hinges on relaxed selectivity of the individual chemically-sensitive coatings. However, the cost of simpler coatings comes in the form of more complex mathematical analysis requirements and the increased power required to run recognition algorithms on suitable processors. The objectives of mathematical methods as they relate to chemical sensor arrays are two-fold. First, mathematical methods can be used to select optimal sensor elements from a menu of many possibilities. Second, suitable pattern-recognition algorithms are required to interpret the results of multielement arrays. The sensor array configuration, particularly the number of similar and diverse array elements, must be compatible with the chosen processing and analysis system.

There are four general classes of mathematical methods that are useful for chemical sensor applications: (1) error minimization-based methods, (2) neural nets, (3) cluster analysis methods, and (4) principal components analysis (PCA). Most of these methods are used for pattern recognition. Current pattern-recognition methods rely on training sets: the sensor system is exposed to different concentrations of all analytes anticipated to be present in the application matrix. These data are then available to compare against unknown samples. However, the presence of unknown materials can lead to incorrect identification of an analyte. In the future, processing methods must evolve for use in unconstrained environments. Consider, for example, the airport security issue. The chemical signatures of many explosive devices are known and it is straightforward to train a pattern-recognition algorithm to look for these materials. However, the background matrix is far too variable to include in the training set: there are numerous chemical interferences such as perfumes, colognes, deodorants, and the like, to which such a screening device would be exposed. The effects of noise, drift, and history of sensors operating under field conditions must be considered and compensated by the data analysis system as well.

Of course, pattern recognition cannot compensate for poorly designed chemical sensor array elements. It follows that elements should be as "chemically independent"[3] as possible and complementary information from different transduction platforms used where practical. Similarly, the effectiveness of pattern-recognition systems can be enhanced if designed to take advantage of more of the information content available in the sensor response transient, in addition to the steady-state response. Software should also be able to select the optimal set of array elements for a particular application from a library of potential candidates, and it should be able to discover the amount of drift and error the system can handle without recalibration. All of this should be accomplished with speed, accuracy, and a convenient human interface.

Packaging and Integration of Control Electronics

Sensor packaging involves connecting transducers to the outside world, isolating the sensor system from aggressive ambients, and applying protective coatings. At present there is little overlap between the transducer and coatings and packaging communities, presumably as a consequence of the paucity of sensor systems that have climbed the development ladder to this crucial stage. Over the next decade, as more sensor systems are steered toward the marketplace, efficient packaging must become a major focus of the sensor community. Relevant packaging issues include the development of better packaging materials, the ability to process them outside ordinary silicon foundries, and their consideration from the start of the development process, rather than as an afterthought.

For many applications, the integrity of the package, which must be partially open to allow access of the analyte, is more crucial and more difficult to insure than for standard electronics fabrication (for example, for in-vivo use or for explosion hazards). As for all aspects of sensor fabrication, packaging issues are application-driven. In some cases, it may be possible to check each sensor in a pass/fail manner for leaks, while other packaging protocols require destructive testing of a small but statistically significant fraction of each "batch" of devices, the results from which are used to infer the integrity of the balance of the lot.

The design of a chemical sensor system requires careful consideration of the extent to which control electronics should be integrated with the sensing elements. There is a general, but not all-pervasive, sense in the chemical-sensor community that as little of the control electronics as necessary should be integrated onto the same chip with the sensor, particularly in the case of sensors destined for use in small, niche markets. The reasons for this are the significant cost of designing and fabricating a custom integrated circuit that includes both chemical sensing and conventional microelectronic elements, and the fact that the materials and methods used in silicon-based microelectronics are often incompatible with the fabrication of chemical sensors. For example, aluminum and, more recently, copper are used for metallization in the CMOS fabrication process, whereas gold, platinum, and palladium are common for chemical sensors. Similarly, silicon oxide is the insulator used for CMOS processing, but its barrier properties are not adequate for liquid-phase sensing applications; in this case, thick layers of high-quality silicon nitride are often preferred. Many of the polymers, molecular films, and ceramic materials common to chemical sensors are completely foreign to modern semiconductor processing facilities as well. The bottom line is that the materials associated with chemical sensors are likely to be incompatible with those used by silicon foundries. The pace of chemical sensor development would be spurred by a few existing and/or new foundries taking a more tolerant and creative approach to diverse materials and processes.

When production volumes for a given sensor are relatively large and there are no major obstacles in compatibility of materials or processing parameters, integration of some or all of the electronic circuitry directly on the sensor chip offers the potential of greater reliability, lower cost, better signal-to-noise ratios, and, in some cases, simpler packaging. In those cases where materials and process incompatibilities are difficult to overcome, however, a most promising alternative to electronics integration is "flip-chip" technology. Here, the chemical sensor and silicon microelectronics are fabricated separately on two different chips, which are subsequently fused by bonding the top sides of both chips to one another via electrical interconnects. The two chips can differ in size, so that the region of the sensor chip destined to interact with the ambient environment can protrude away from the area in which the two chips are bonded, and the area with contacts for connections to external circuitry can protrude similarly (typically away from the sensing region). The interconnects are made using solder, hence both chips must be heated to temperatures in excess of 200°C—a potential problem for many chemically sensitive materials, although the use of lower-melting solder alloys, or even conductive epoxies, is a distinct possibility. The interchip region, including interconnects, is then encapsulated; the nature of the encapsulant is dictated by the sensing environment (chemical composition, temperature, exposure time, etc.). The boundary between exposed and unexposed regions of the sensor must be resilient and must not delaminate. Both interconnection and encapsulation are areas of contemporary engineering R&D, primarily in the microelectronics industry.

Summary

The need for chemical sensor systems is expanding at a rapid clip. Chemists and materials scientists have designed a plethora of chemically selective materials, but only a handful of these are sufficiently selective, sensitive, cost- effective, and durable to be used as stand-alone materials for commercial implementation. Thus, many of the chemical sensor systems developed over the next decade are likely to be more generic and also more versatile, employing an array format or some kind of separation process such as the "lab-on-a-chip" format. To meet the needs of such array-based systems, suitable transducer technologies, pattern-recognition methods, and signal-processing schemes are already available or in the advanced stages of development. The primary challenge, then, is to identify combinations of chemically sensitive interfacial materials and transducers that yield reproducible responses amenable to interpretation by pattern-recognition algorithms, without neglecting the fundamental understanding of interaction mechanisms and application-specific effects. This need will be largely application-driven, requiring excellent communication among chemists, engineers, software developers, and the end users of the sensors. An additional, crucial challenge for chemists and engineers comes in the form of developing suitable packaging and interconnection materials and methods. While these issues have often been ignored during the research and preliminary development phases, they are best considered from the outset if the product is to successfully reach and compete in the commercial marketplace. ■

Acknowledgments

In May of 1997, the three coauthors of this article and 26 additional scientists and engineers representing academia, U.S. government laboratories, industry, and several U.S. federal agencies met in Blue Mountain Lake, New York for a three-day workshop on chemical sensors sponsored by the National Science Foundation. A similarly diverse group met again in July of 1998 in Henniker, New Hampshire, to reconsider and update the report that resulted from the first workshop. (Reports from both workshops are available on the Web: http://www.chem.tamu.edu/walker/chemsensors.html). These workshops sought to determine the current state of the field of chemical sensors, to identify critical R&D objectives for the next five to ten years, and to define strategies for achieving those objectives. This brief article represents the collective wisdom of these individuals, whose names are given below. We gratefully acknowledge the participants of both workshops for participating in discussions, writing drafts of various sections of this article, and for critically reading and commenting on the final version. We especially acknowledge Dr. Petr Vanysek for critically reading the manuscript and offering substantial comments to improve it.

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