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A simple laboratory procedure for packaging and testing part-fabricated gas microsensors

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Abstract. Simple schemes for gas microsensor packaging and testing are presented. The packaging procedures are developed for mostly hand operations without reliance on sophisticated equipment. Special flow-cell features and compact design enable easy configuration for testing in gas streams by flow-injection. Automation improves the efficiency of the sampling of gas microsensors and hence provides a higher compilation rate for the test data.

1. Introduction

Post-fabrication treatments of gas microsensor chips include wire bonding, encapsulation and packaging into a configuration for measurement in gas streams. Although the tasks are not difficult to complete, for researchers without access to a well-staffed and well-equipped microelectronics facility, they are nonetheless time-consuming. Great inconvenience through the years has motivated us to formulate a series of simple steps leading towards the complete testing of the gas microsensors from the fabricated chips within our own laboratory. They require only a few small tools and one person can carry out the whole procedure by himself. With some practice, we have been able to prepare packaged microsensors ready for measurements in gas streams within hours.

This paper presents a detailed account of these new procedures, following which we have recently processed and tested fabricated gas microsensors in quantity. The efficient data collection has enabled us to report the typical microsensor characteristics by employing statistics on a sufficiently large sample size.

2. Initial device bonding and packaging

The initial steps involve attachment of the microsensor chips to headers. Metal can headers with 8–12 pins were often used. The small headers were first held in place by plugging them into sockets already welded onto a small piece of the circuit board. The board served as a holder that permitted the adjustment of header positions and work angles quickly by hand. Observation required a stereomicroscope (Bausch & Lomb Stereozoom 6) to provide a relatively long working distance and large depth of field. The entire micromanipulation process that followed, including work on the highly reflective chip surface, was visualized by employing vertical illumination (a ring fluorescence lamp) on the microscope. As shown in figure 1, the initial bonding and packaging process was generally carried out in three steps.

(i) The microsensor chip was first held with tweezers in one hand and rubbed on its back with an insulating epoxy resin (Conap Easypoxy K-20). After its having been settled down onto a header, more of the epoxy resin was applied to the vertical sides to prevent edge conduction. The epoxy resin was transferred drop by drop through a hand-held microprobe tip (Universal Tool Kit, from Terra Universal, USA). The epoxy resin hardened in about 1 h at room temperature and was cured at about 80 °C for about 2 h. Obviously, 5 min epoxy resins will not work since there will not be enough time to complete the epoxy transfer. Attempts to quicken the epoxy resin hardening process by means of heating in an oven had invariably resulted in flow of the epoxy resin to unwanted areas on the chip surface. Once the epoxy resin had already hardened, curing the epoxy resin at a higher temperature did not pose any further problems.

(ii) The next step involved forming the interconnections between the bonding pads on the chip surface and the header pins. This was performed under the microscope by joining across drops of highly conductive Ag epoxy resin (Dotite FA-705A, Japan) into a beaded, connecting line, figure 1. The Ag epoxy resin drops were delivered through a hand-held microprobe tip as before. As noted from practice, the imprecise nature of working by hand inevitably introduced a minimum required working distance between the bonding pads on the chip surface. For bonding pads spaced less than about 0.5 mm apart, the chance of smearing the Ag epoxy resin across the adjacent pads increased. This had not become a problem, however, for our typical

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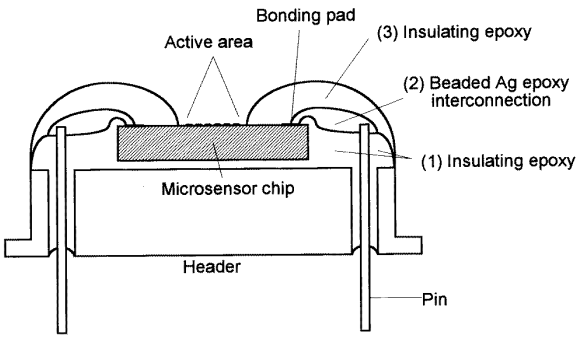

Figure 1. A scheme showing the three initial device bonding and packaging steps.

roughly 3 mm×3 mm microsensor chips with 8–12 bonding pads (two or three on each side). Following epoxy resin curing, the insulating and connectivity measurements (Keithley 617) were carried out on the device to test for short-circuits or broken connections. Other electrical characterization measurements were also performed at this stage to acquire the device base functions in control experiments.

(iii) In the last step, a layer of the insulating epoxy resin was applied to the device to cover up the Ag epoxy resin interconnections. Only the surface exposing the active area on the device was left open, figure 1. At this stage, the device was ready for further packaging to handle the gas flows.

3. Microsensor flow-through fixtures

Following initial bonding and packaging, the microsensors were further packaged with a fixture for the handling of gas flows. There were two flow-through fixture configurations for the microsensors, figure 2, for conducting specific gas sensing experiments. They are based on the components commonly found on chromatographic autosamplers and vacuum systems.

The first flow-through fixture was designed for situations in which we have come across the need to interchange among the microsensor samples and gas sources frequently at a test station during operations in the field. The configuration makes use of an inexpensive glass vial part for chromatography (Alltech 66002, USA), as illustrated in figure 2(*a*). The 'crimp top' opening on the glass vial was sectioned off and mounted by epoxy resin over the initially packaged microsensor body. By carrying out just such a quick step, the microsensors were equipped with a full flow-through capacity. The septum used for link-up to gas channels through syringe needles was a simple solution for loading the samples quickly. The syringe needles (Alltech 94855) with Luer type adapter can make easy connections to various gas manifolds. The internal volume of the flow-through cavity was about 0.15 ml, small enough to allow fast equilibration of gases for most applications.

This fixture configuration has since been adopted extensively on our practical microsensors for its compact

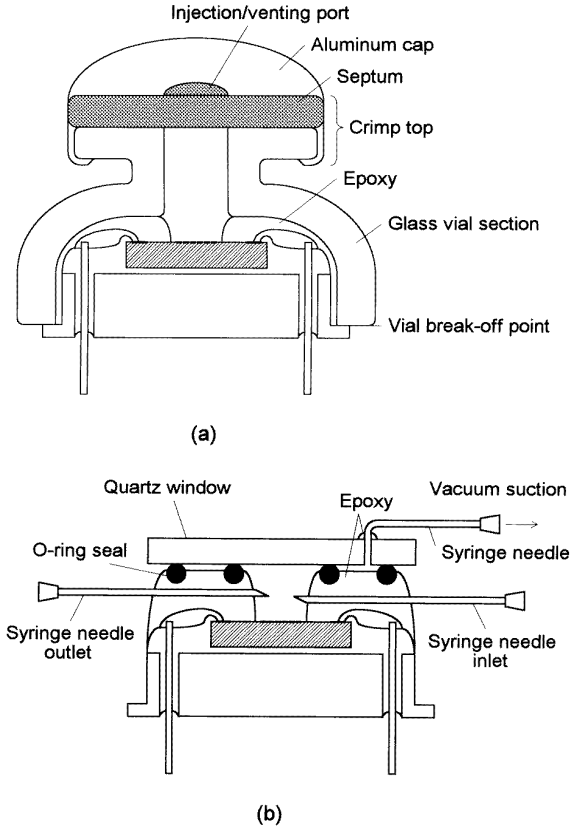

Figure 2. Schematic drawings of (*a*) the 'crimp top' flow-through fixture, which uses syringe needles for the injection and venting of gases and (*b*) the flow-through fixture with an optical monitoring capability by microscopy.

size and simplicity in use. Convenient to carry around, the use inside the laboratory was often extended to operations in the outside field. This fixture configuration also led to easy storage for our practical microsensors. Normally filled with an inert argon atmosphere when not in use, the microsensors were sealed from contaminants in the open air. Such a feature was particularly helpful when we had to carry the microsensors around from place to place in the field.

Next, a second flow-through fixture was designed for use in special situations. When making electrical measurements on the microsensors placed under gas flow, there is frequently a need to observe the changes simultaneously by optical means. In some cases, the ability to monitor optical properties is crucial. For example, we had previously created microsensors with electrochromic components (Chao 1993, Chao and Wrighton 1987). The solid-state CO_2 microsensors based on WO_3 (Chao 1993) can undergo a bleach/colour cycle when it is exposed to a switched N_2/CO_2 flow. The spectral change in the microsensor had been successfully monitored in recent months by employing a second flow-through fixture, as illustrated in figure 2(*b*).

The fixture comprised a flow compartment that was covered by a removable quartz window. Two Luer-type syringe needles affixed to the perimeter of the compartment

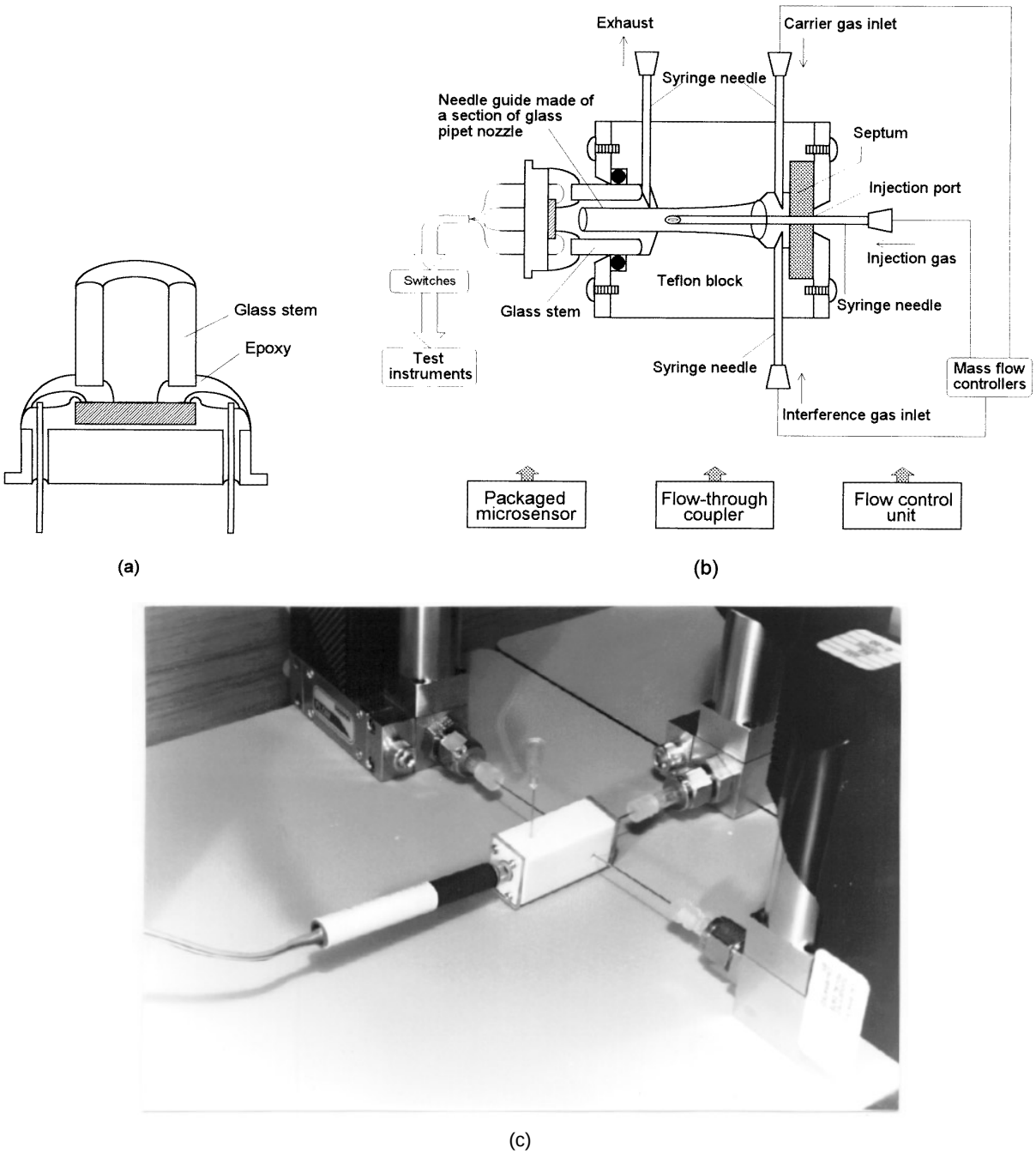

Figure 3. The apparatus used for automated testing: (*a*) the specially packaged microsensor with a glass stem for quick adaptation; (*b*) the apparatus and the arrangement for the connections between the flow-through coupler, the specially packaged microsensor and the flow control unit; and (*c*) a photograph of the apparatus. For low-level injections, an infusion pump was used at the injection port instead of the mass-flow controller.

functioned as the channels for gas flow. The two O-rings arranged concentrically with vacuum suction in between held the window in place. For facilitating microscope observation, the quartz plate (1 mm thick) was kept in close proximity to the surface of the microsensor chip. The distance in between was close to 3 mm that left us with a roughly 4 mm working distance for practical microscopy.

For the cylindrical flow-through cavity, which measured roughly 3 mm in diameter in figure 2(*b*), the internal volume was only about 0.02 ml. Comparable internal volumes can be found in many commercial flow-through detectors for chromatography. The vacuum suction around the flow cavity was also a safety measure against the accidental leakage of noxious gases during sensing experiments.

4. Automated microsensor testing

The apparatus for automated testing is functionally a programmable 'flow-injection' analyser for the microsensors.

The apparatus incorporated three functional parts, a specially packaged microsensor, a flow-through coupler and a flow control unit for gases. They are each described separately below.

(i) For the specially packaged microsensor, an extra glass stem was mounted following the initial bonding and packaging process, figure 3(*a*). The stem was a piece of glass tubing about 8 mm long with about 3 mm inner diameter. It served as a link to the flow-through coupler for channelling the gas flows. Its front surface had a fire-polished finish to prevent possible cutting into elastomers.

(ii) The construction of the flow-through coupler was quite expeditious and uncomplicated. The work involved drilling some holes through the small Teflon® block as illustrated in figure 3(*b*). A glass pipette nozzle was inserted through the centre bore to serve as a needle guide. An O-ring and a septum (made of Teflon®-lined silicone rubber with 8 mm diameter) were then packed into the appropriate holes for union with the microsensor and for forming the injection port for a syringe needle. The flow-through coupler design allowed the specially packaged microsensors to slip on and off quickly during sample changes. Gas channel openings on the coupler were added by pushing the syringe needles through slowly into the Teflon® block. Normally, three channels for the carrier, interference and exhaust gases were adequate, figure 3(*b*). More gas channels can be opened by pushing through additional needles when needed.

(iii) Electronic flow controls were employed for feeding in the test gases. Steady flows into the inlet and injection ports on the coupler were normally provided by programmable mass-flow controllers (Tylan General or Sierra Instruments, USA), figure 3(*c*). The proportions of the injection or interference gases blending into the carrier gas were adjusted by varying the appropriate flow ratios. This was done while keeping the total flow to the microsensor constant.

For experiments involving low-level injections, an infusion pump (Orion Research Sage 341B, USA) directly connected to the injection port was used. The pump advanced the plunger of a removable gas-tight type syringe (Hamilton Gastight® syringe) with high precision. By a combination of the appropriate syringe volume and the pump advancement rate, reproducible flows as low as 1.2 μl min^{-1} (with a 5 ml syringe) were generated routinely in the laboratory. An in-line microvalve (Mininert® Syringe Valve, Alltech 654051) was attached to the syringe to assist closure of the gas content within the syringe. The syringe was regularly filled from a glass bulb that had been previously evacuated and purged by the appropriate gas with a desired composition. For further reduction in the flow levels of the injection gas, a diluted gas mixture from an analysed commercial source was used.

The procedures for running the apparatus in figure 3 were uncomplicated. The only operating needing attendance was the manual loading of the microsensor samples onto the flow coupler. Other work including adjusting the flow control, triggering the measurement instruments and recording the electrical response was performed by running programs on a personal computer. Normally, the preparation before starting data collection occupied the operator for just a few minutes. The almost minimal time spent in between tests increased the speed for the microsensors sampled in quantity. The accelerated experimental process allowed more microsensors to be sampled at once for a sufficient sample size, which is essential in presenting the test results by employing statistics. The accelerated process also helped in implementing the experiments over one stretch without any discontinuation to avoid the variations in the day-to-day laboratory conditions.

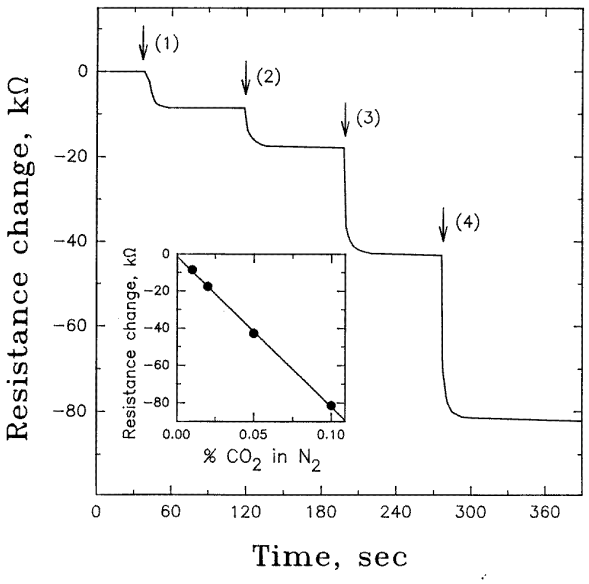

Figure 4. The measured resistance change in a WO_3-based microsensor as a function of the low CO_2 concentrations: (1) 0.01%, (2) 0.02%, (3) 0.05% and (4) 0.1%, in N_2 under a constant total flow of 11.5 ml min^{-1} at 1 atm, 295 K and 50% H_2O saturation. A simple two-terminal measurement with 300 mV bias was employed for the test. Other details of the microsensor and the method of the electrical measurement have been published elsewhere (Chao 1993).

Figure 4 shows the typical performance characteristics when a CO_2-sensitive, WO_3-based microsensor was placed under test by use of the flow injection apparatus in figure 3. Details for the construction of this WO_3-impregnated microsensor and its other CO_2-dependent electrical characteristics across closely spaced microelectrodes can be found in a previous publication (Chao 1993). The injection of CO_2 was performed by use of the infusion pump equipped with a 5 ml syringe. The injection rates were purposely set to produce steady-state low-level CO_2 flows with the ratio factors 1, 2, 5 and 10. High-purity grade N_2 with < 1 ppm residual CO_2 was used as the carrier gas. The steady flows of N_2 were generated by the mass-flow control unit.

Figure 4 plots the resistance change as a function of the low-level CO_2 concentrations to which a WO_3-based microsensor was exposed. The response to low CO_2 concentrations in the range about 0.01–0.1% in N_2 is reversible and reproducible. The resistance drop is

relatively linear as the concentration is increased. The sensitivity or the slope of the curve in the linear region is of the order of 840 kΩ per 1% CO_2 concentration change. Since each microsensor required only about 10 min to complete the sensing test, a statistically sound sample size can be reached quickly. Gathering results from 28 packaged samples, the mean sensitivity value and the standard deviation for the microsensor can be determined. The valuation yields a resistance drop of 836 kΩ per 1% CO_2 change in N_2 for the mean sensitivity value and a standard deviation of 31 kΩ per 1% CO_2 change in N_2.

5. Conclusion

For a small laboratory like ours that relies on a foundry for fabrication services, the packaging procedures outlined here have been very effective. They have reduced the work involved in the post-fabrication treatment of the received microsensor chips to a level manageable by a single person in the laboratory. The initial device bonding and packaging procedure is an expeditious way to process prototype gas microsensors. The procedure, however, is limited to microsensors having bonding pads spaced not less than about 0.5 mm apart due to the chances of smearing the Ag epoxy resin. Very-high-density-array-type microsensors are thus not applicable. The use of epoxy resins near gas microsensors can lead to erroneous readings and even poisoning of certain microsensors, particularly with some microsensors that run at elevated temperatures. The procedure is thus limited to gas microsensors compatibleto epoxy resins that operate at near room temperature.

A point worth emphasizing on the flow-through fixtures reported here is that, for microsensors, the designs are compact and well suited for making a quick link-up to gas sources at various test sites. They are particularly handy for performing field demonstrations. For example, we have employed the fixtures in figure 2(*a*) in successful demonstrations of microsensors for sensing CO_2 in the human breath and spinach plant photosynthesis cycles outside the laboratory.

As reported here, we have also trimmed down the time required for carrying out the sensing tests by automation. The specially designed flow-through coupler for fast sample loading and computer-based instrumentation and flow control have assisted us in accelerating a variety of microsensor tests under gas streams. By now, each experienced laboratory worker can routinely test microsensors at a rate of up to about 30 per day.

Acknowledgments

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References

Chao S 1993 Electrical characteristics of WO_3-based CO_2-sensitive solid-state microsensor *Japan. J. Appl. Phys. 2* **32** L1346–8

Chao S and Wrighton M S 1987 Characterization of a 'solid-state' polyaniline-based transistor: water vapor dependent characteristics of a device employing a poly(vinylalcohol)/phosphoric acid solid-state electrolyte *J. Am. Chem. Soc.* **109** 6627–31