AIP | Review of Scientific Instruments

Versatile computer-controlled system for characterization of gas sensing materials

M. Zhao, J. X. Huang, M. H. Wong, Y. M. Tang, and C. W. Ong

Citation: Rev. Sci. Instrum. **82**, 105001 (2011); doi: 10.1063/1.3648132

View online: http://dx.doi.org/10.1063/1.3648132

View Table of Contents: http://rsi.aip.org/resource/1/RSINAK/v82/i10

Published by the American Institute of Physics.

Related Articles

Enhanced H2 sensitivity at room temperature of ZnO nanowires functionalized by Pd nanoparticles
J. Appl. Phys. 110, 084312 (2011)

Expanded beam deflection method for simultaneous measurement of displacement and vibrations of multiple microcantilevers
Rev. Sci. Instrum. 82, 105112 (2011)

High quality factor graphene varactors for wireless sensing applications
Appl. Phys. Lett. 99, 163105 (2011)

The extended surface forces apparatus. IV. Precision static pressure control
Rev. Sci. Instrum. 82, 103902 (2011)

Effect of temperature on CO detection sensitivity of ZnO nanorod-gated AlGaN/GaN high electron mobility transistors
Appl. Phys. Lett. 99, 142107 (2011)

Additional information on Rev. Sci. Instrum.

Journal Homepage: http://rsi.aip.org

Journal Information: http://rsi.aip.org/about/about_the_journal

Top downloads: http://rsi.aip.org/features/most_downloaded

Information for Authors: http://rsi.aip.org/authors

ADVERTISEMENT

AIPAdvances

Explore AIP's new open-access journal

- Article-level metrics now available
- Join the conversation! Rate & comment on articles

Submit Now

Downloaded 25 Oct 2011 to 158.132.183.20. Redistribution subject to AIP license or copyright; see http://rsi.aip.org/about/rights_and_permissions

REVIEW OF SCIENTIFIC INSTRUMENTS **82**, 105001 (2011)

Versatile computer-controlled system for characterization of gas sensing materials

M. Zhao,[1,2] J. X. Huang,[1] M. H. Wong,[1] Y. M. Tang,[1] and C. W. Ong[1,a)]
[1]*Department of Applied Physics and Materials Research Center, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong, China*
[2]*Department of Electronic Science and Technology, Changzhou University, Changzhou, 213164, China*

(Received 11 August 2011; accepted 15 September 2011; published online 20 October 2011)

Design of a system used for characterizing gas sensing materials is described. It is distinctive of being able to measure electrical and optical responses of a sample simultaneously, control a number of measurement parameters, perform fast exchange of gaseous environment, and be fully controlled automatically. These features make the system to be versatile in determining most concerned performance indexes of a gas sensing material (e.g., sensitivity, stability, selectivity, response/recovery times, etc.) as functions of various combinations of measurement conditions (e.g., gas concentrations, temperature, total pressure, content of interferants, photo assist, relative humidity, soaking time in a fixed gas concentration, and number of switching cycles in a dynamic test, etc.). Rationales of the designs associated with general gas sensing mechanics are discussed. © *2011 American Institute of Physics*. [doi:10.1063/1.3648132]

I. INTRODUCTION

Research and development of advanced gas sensors are very active nowadays for the need of monitoring leakage or presence of hazardous gaseous contaminants in air, such as nitrogen dioxide,[1] sulphur dioxide,[2] ozone,[3] carbon dioxide,[4] volatile organic compounds,[5] carbon monoxide,[6] hydrogen,[7] methane,[8] etc. Practical experience accumulated so far indicates that quite a number of data are required to specify the performance of a gas sensing material, including detection range of gas concentration; sensitivity; response/recovery times; stability (in static and cyclic conditions); selectivity; ambient pressure, temperature and relative humidity (RH) dependences; photo-assist dependence, batch-to-batch reproducibility and power consumption, etc. We found that reports in this area had different emphases and different groups of the parameters were selected to be investigated.[9–12] In addition to different technical considerations, the designs of the setups used for characterizing gas sensing properties of materials are quite diversified.[12–15] This scenario leads to the difficulty in establishing general standards for characterizing gas sensing materials. We were thereby motivated to report the design of a characterization system used in our recent studies on gas sensing materials, which was made to be fully automatic and capable of covering the measurements of all the abovementioned parameters. The configuration was conceived by referring to general working principle of a chemical gas sensor, and may be further explored for setting up general procedural guidelines in characterizing a gas sensing material. The structure of the system and the underlining considerations are represented in detail in this article.

II. CONFIGURATION OF THE SYSTEM

Fig. 1 is a block diagram of the proposed system. The pipeline contains mass flow controllers (MFCs) and valve assemblies (V). In more detail, each of MFC1 and MFC2 depicted in the figure is a combination of two mass flow controllers (MKS) with working ranges of 0 ∼ 20 and 0 ∼ 200 sccm, respectively. This structure broadens the overall range of flow rate of the assembly. Each valve assembly, denoted as V1 ∼ V14 in the figure, contains a bellowed sealed pneumatic valve (Swagelok, Model SS-4BK-1C), which is activated by a 3-way solenoid valve (SMC, Model VT307–5G-01). This is a typical configuration widely adopted for handling hazardous gases. Other than the measurement chamber, there is a mixing chamber and a sealed water chamber. A gas sample is generated by mixing the detected gas, interferant(s), and balancing gas with targeted compositions in the mixing chamber. Moisture can be introduced into the gas sample by passing through the water chamber. For pressure measurement, P1 is a capacitive absolute pressure sensor with a working range of 0 ∼ 10 Torr (MKS, Model 122A-12225). P2 to P4 are MEMS-type pressure sensors with a lower resolution in a broader working range of 0 ∼ 2 atmospheric pressure (Honeywell, Model ASDX030A24R). This combination allows determination of pressure in a broad range.

Fig. 2(a) shows the details of the measurement chamber with a 3D drawing. It is a stainless steel cross of six KF-40 ports. The top flange contains all the electrical feedthroughs to be described in the next paragraph. Three glass viewports are set at three ports for optical measurements. Incident light beam, either a He-Ne laser beam or broad-spectrum light from a tungsten- (W-) halogen lamp, enters the chamber via one of the viewports and shines on the surface of a sample material with an incident angle of 45°. The reflected and transmitting beams from the sample material come out via other viewports, respectively, and are received by two silicon-based photodiodes (Hamamatsu, Model S1337-66BR) or spectrometers

---

[a)] Author to whom correspondence should be addressed. Electronic mail: apacwong@inet.polyu.edu.hk. Tel: (852) 2766 5689. Fax: (852) 2333 7629.

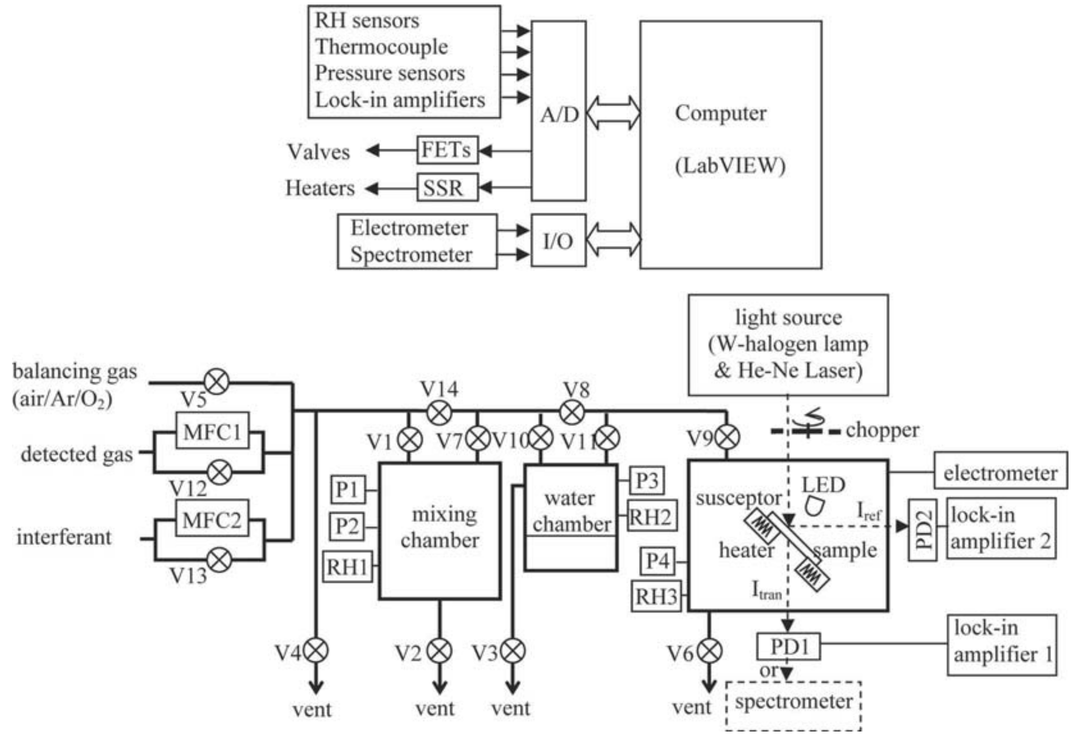

FIG. 1. Block diagram of the reported measurement system. V: valve; MFC: mass flow controller; P: pressure sensor; RH: relative humidity sensor; PD: photodiode; LED: light emitting diode; A/D: analog digital converter; FET: field effect transistor; SSR: solid state relay; I_{ref}: reflected light beam; I_{tran}: transmitting light beam.

(Newport, Model OSM2-400DUV). The variations of their intensities according to the changes in measurement conditions are detected to reflect the gas induced optical response of the sample material investigated. Other two ports have a 1/4″ gas inlet and an electromagnetic (EM) valve connecting to a 2-stage mechanical pump for venting, respectively.

Fig. 2(b) shows the details of the top flange. A hermitically sealed BNC socket is located at the center position for measuring the resistive response of a sample material. Other conducting leads are connected to two cartridge heaters (Hawco, Model HS25-2), a K-type thermocouple, a pressure sensor (Honeywell Model ASDX030A24R), an RH sensor (Sencera, 805H5V5, detection range of 0 ∼ 100%), and a light emitting diode (LED). The sample material is mounted at a copper susceptor, which is in good thermal contact with the cartridge heaters.

With this configuration, the system is ready to detect resistive and optical responses of a sample material simultaneously. The former is recorded with a Keithley Model 617 electrometer. For the latter, the incident light beam is modulated at 170 Hz with a mechanical chopper. A sample material to be investigated should be fabricated as a film on a transparent glass substrate. The reflected and transmitting lights from the sample material shining on the photodiodes generate ac electrical signals, which are collected by two lock-in amplifiers (Stanford SR830). Random noise and background interference are basically eliminated.

The system is fully automated with the aid of a computer program compiled in LabVIEW 2010. Condition of a test is set up by utilizing the values of pressure, temperature, and RH in the chamber. The pressure values in the mixing, water, and measurement chambers are detected by Sensor P1 to P4; temperature of the sample material is detected by a thermocouple adhered to the sample holder; and RH values are determined by Sensor RH1 to RH3. All these signals are sent to the computer through an analogue digital (A/D) module (NI, Model USB-6281) as shown Fig. 1. The computer program compares the pressure signals with set points. The resulting digital control signals are sent out by the A/D module to de/energize valves in a programmed sequence for (i) evacuating/filling up the chambers with gas sample; or (ii) feeding detected gas, interferant(s), and balancing gas into the mixing chamber in a defined ratio to form a mixture of gas sample (see more details in Sec. III). The program also receives the temperature of the sample material and calculates the ramp rate and output efficiency with respect to the set point. It generates control signal to operate a MOSFET-solid state relay (SSR) and hence regulates the power output applied to the heater. The temperature of the sample material is thus fairly controlled. The computer program also records RH values as one of the measurement parameters. In addition, the program receives data from the electrometer showing the resistive changes; and those from lock-in amplifiers or a spectrometer showing the optical responses of the sample material.

III. BASIC OPERATION AND CONTROL

We describe basic operation procedure of our system as follows.

Step 1: Valves V1, V2, and V3 are opened to pump down the mixing and water chambers with a rotary

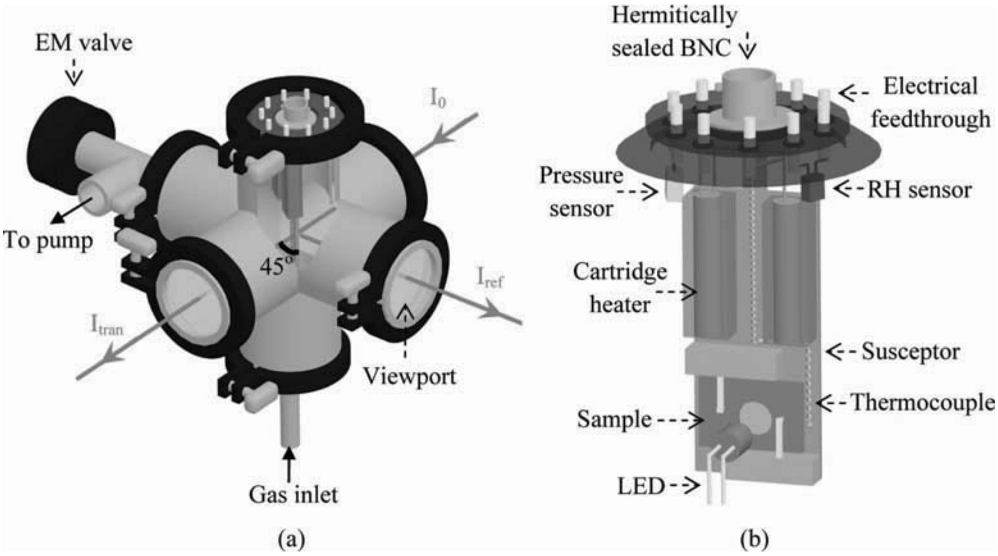

FIG. 2. (Color online) (a) Measurement chamber, (b) top flange and the attached components.

pump. They are then closed after reaching a pressure ≈ 0.15 Torr. Meanwhile, all other valves and MFCs are closed.

Step 2: MFC1 is activated to feed the detected gas into the pipeline. The flow is initially unstable, so that V4 is opened for about 10 s to wait for the flow to be steady. It is then closed and at the same time V1 is opened to release the detected gas into the mixing chamber. Until the pressure detected by P1/P2 reaches the set point, MFC1 and V1 are closed to stop the gas flow. The number of moles of the detected gas in the chamber is derived either from the duration of the flow or the pressure detected. A required amount of certain interferant can also be introduced with similar process through another channel controlled by MFC2.

Step 3: A balancing gas, e.g., air, pure oxygen, or argon, is fed into the mixing chamber by opening V5 and V1. The valves are closed after reaching the targeted pressure. The molar ratio of the constituents in the mixture, namely, gas sample in the test, is fixed.

Step 4: The measurement chamber is then pumped down by opening V6. Until the pressure drops to ≈ 0.15 Torr, valves V7, V8 and V9 are opened to release the gas sample into the measurement chamber and react with the sample material. The valves are finally closed at equilibrium.

Step 5: This step is applied if the influence of RH on the gas sensing properties of a material is investigated. Valves V7 and V10 are opened for the gas sample to enter the water chamber and carry some moisture. The level of RH is detected. The measurement chamber is pumped down by opening V6. Until reaching ≈ 0.15 Torr, V6 is closed, and V11 and V9 are opened to release the gas sample to react with the sample material. Valves V7, V10, V11, and V9 are closed at equilibrium.

Step 6: The resistive and/or optical responses of the sample material are recorded.

This basic testing process can be modified and recombined with great flexibility to meet the purposes of various studies. Two representative application examples are given in Sec. IV to illustrate the versatility of our system.

IV. PRACTICAL EXAMPLES

A. Quasi-static test: H_2 sensing properties of palladium/magnesium-nickel film

In this example, we demonstrate how the system is used to derive the quasi-static relationship between the gas sensing response of a material and surrounding gas concentration determined at a fixed temperature. The results lead to valuable insights into degradation mechanisms of the gas sensing properties of the material.

The experiment was done for measuring the resistance of a palladium coated magnesium-nickel (Pd/Mg-Ni) film as a function of surrounding hydrogen (H_2) partial pressure (P_{H2}). A cylinder containing 4% H_2_96% argon (Ar) was used as the gas source. After pre-pumping, the gas was fed into the system in small steps for P_{H2} to scan over a range of 17–3000 Pa. Each step was held for 30 min before the next admission. As such, P_{H2} was increased slowly, and the sample was considered to be in equilibrium with the gaseous environment throughout the test. Moreover, Step 5 was skipped to avoid incorporation of water vapour, and the sample was kept at 60 °C.

Curve 1 as shown in Fig. 3 is the result obtained, which is referred to as the resistance-pressure isotherm (RPI) of the film. One finds that the film resistance increases in different manners in three regions of P_{H2}. In the region of $P_{H2} < 1100$ Pa, the film resistance rises with a relatively slow rate as P_{H2} increases. According to literature, the film is suggested to be in an α-phase. In this state, H atoms are able to enter or leave the atomic network interstitially, without causing substantial irreversible composition and/or structural change. As such, the H-induced resistive response of the film is rather stable

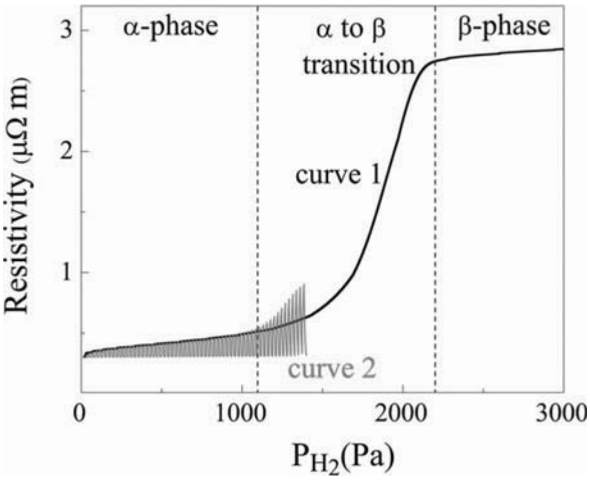

FIG. 3. (Color online) Quasi-static RPI (Curve 1) and cyclic resistive response (Curve 2) of a Pd/Mg-Ni film reacting with H_2 obtained in a dry environment at 60 °C.

and reproducible. This viewpoint is further supported by the results obtained in a cyclic test, as shown by Curve 2 in the figure. The data were collected when exposing the film to air and 4% H_2–96% Ar gas alternatively, with P_{H2} increased progressively in successive cycles. Each exposure lasted for 5 min, while pumping was performed before filling the chamber with another gas. One notices that in this region, the baseline of the film resistance measured in both the static and cyclic modes are consistent and do not shift. Moreover, the saturated resistive responses at any P_{H2} measured in the two modes are also about the same. This suggests that migration of H atoms into and out of the film does not alter the structure and hence the gas sensing response of the alloy.

In the region of P_{H2} in between 1100 and 2200 Pa, the film resistance rises much steeper, which is believed to originate from the occurrence of an α- to β-phase transition. From literature, it is pointed out that the transition involves a substantial volume change. As a consequence, the cyclic test is accompanied with reciprocating volumetric expansion-contraction cycles, which aggravate irreversible deformation of the film structure, and the deviation between Curve 1 and 2 observed in this region is explained. In the region of $P_{H2} > 2200$ Pa, the change of film resistance becomes slow again. It is suggested that only β-phase is present in this condition.

One concludes from these observations that a Pd/Mg-Ni film could be used to make high-sensitivity H_2 leakage alarm, because in a low-H_2 environment the reproducibility and durability of the sensor are greatly ensured. Contrarily, the use of a Pd/Mg-Ni film in detecting $P_{H2} > 1100$ Pa is not suitable, as α-to-β transition is involved to jeopardize the reliability of the H_2 sensing properties of the material.

At last, we further propose that the enthalpy of a phase transition (like the α-to-β one in this example) could be derived if one observes the temperature dependence of the representative P_{H2} values extracted from the transition regions of the plots. Analyses of this type are standard for chemists when analyzing a chemical reaction, which are normally done with a furnace of well control in gas flow and temperature.

B. Cyclic test: H_2 sensing stability of Pd functionalized tungsten oxide film

In this example, we demonstrate how to use the system to carry out a cyclic test aiming at investigating the dynamic sensing stability of a material with increasing number of switching cycles. Studies of this kind are of practical meaning, because in many reports involving this topic, data presented are just from small numbers of switching cycles, such that the genuine reproducibility of the sensing response is not fairly explored. However, the information is important if a sensor is required to be exposed repeatedly to a gas, such that the stability of sensitivity is a dynamic gaseous environment is greatly concerned.

The experiment was performed to measure the variations of the optical transmittance and reflectance, and resistance of a Pd functionalized tungsten oxide (WO_3) film at 90% RH and 80 °C. The sample was exposed to a 4% H_2–96% Ar gas and air alternatively over a substantially large number of cycles. Each exposure process was performed at 1 atmospheric pressure and lasted for 10 min, while pumping was performed before feeding another gas.

Fig. 4 shows the result of the measurements. A total of 200 switching cycles were conducted. Data show initial instability of the responses of the three quantities in the first few cycles. Thereafter, the variations of the optical transmittance and resistance lie steadily in respective bands. The optical reflectance is rather noisy because of the low reflection level of the film surface. The magnified line shapes of the last cycles of respective quantities are further illustrated on the right side of figure.

V. DISCUSSIONS

A. Flexibility and versatility

Our system is distinctive of being able to measure electrical and optical gas sensing responses of a sample concurrently, and hence is different from many other systems designed for measuring just one of them.[12–23] This feature helps find any difference of time dependences of electrical response and optical transmittance response, because the former is more sensitive to surface reaction and the latter is more dependent on the bulk reaction throughout the light path. The time dependences of optical reflectance due to the reflected light from the gas-film interface or that from the film-substrate interface may have some difference if diffusion of detected gas molecules/atoms needs some time to diffuse inside the material.

Our system is also able to control many measurement parameters independently in broad ranges, which include concentration of detected gas component, temperature, RH, content of interferant, type of balancing gas, total pressure, and intensity/wavelength of assist photo excitation. Hence, it is highly versatile in deriving a variety of parametric

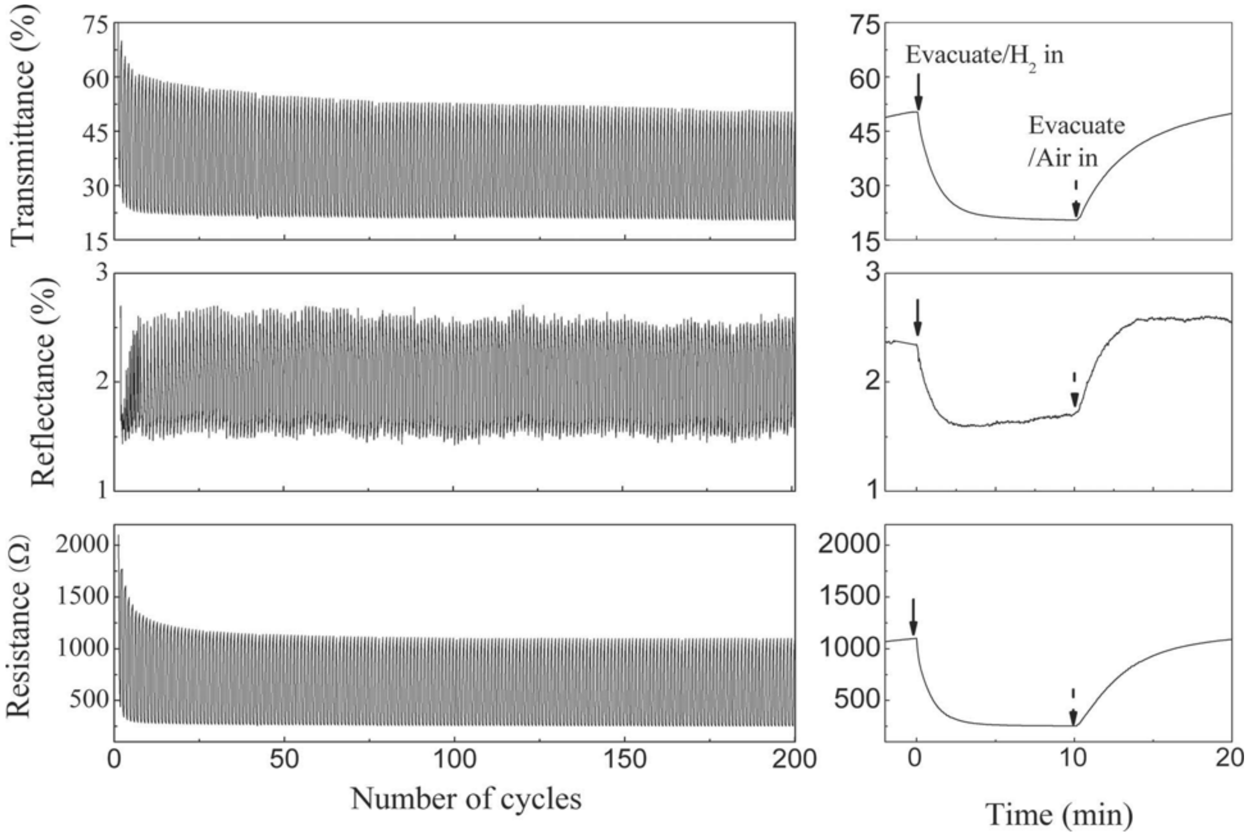

FIG. 4. Resistive and optical H_2 sensing responses of a Pd/WO_3 film measured at 80 °C and 90% RH, showing the sensing stability of the material.

dependences of gas sensing responses through adopting appropriate combination of measurement conditions.

B. Control of gas composition

The gas mixing technique adopted in our system facilitates adjusting gas composition over a broad range. This feature is illustrated by considering an example of using 4% H_2 in a balancing gas (air, or Ar or N_2) in a study of H_2 sensing. To get the lowest H_2 concentration with our system, one applies the 20-sccm MFC to release the gas into the chamber. The lowest acceptable setting with reliable accuracy is normally 2% of the full span of an MFC, i.e., 0.4 sccm of the mixture or 0.016 sccm of net H_2 flow in this case. The flow may last for a reasonable duration, say 10 s, to release a total of standard 0.00267 cm^3 of H_2 into the chamber. More balancing gas is then added through another pipeline until the total pressure reaches 1 atmospheric pressure. The H_2 concentration is further diluted. The volume of the mixing zone is 2350 cm^3. The H_2 concentration attained with this procedure is $\approx$ 1 ppm. A dynamic range of H_2 concentration available for the test is 1–40 000 ppm. The upper bound is achieved by passing the undiluted gas into the system. It should be noted that the lower bound of H_2 concentration can be further reduced if a more dilute H_2-containg gas source; or a shorter duration of gas admission; or an MFC of a smaller full span is employed.

Another advantage of the system is low consumption of gases compared with continuous flow technique employed by some other groups.[12–14, 17–22] In continuous flow method, admixture containing the detected gas is kept to flow throughout a test. If a net H_2 flow of 0.016 sccm is applied as in the previous example, a flow of 16 000 sccm of balancing gas is required to dilute H_2 to 1 ppm. With a measurement condition under 1 atmospheric pressure, a typical cylinder containing 6.8 m^3 would exhaust in about 7 hours, rendering the test to be rather uneconomical. Contrarily, in our system, the gas mixture is confined in a closed system in most of the time of a test and at equilibrium with the sample. Consumption of the gas is mainly due to pumping when the concentration of the detected gas or the gaseous environment is needed to be changed. However, it must be noted that this approach is valid only when the total amount of the detected gas in the measurement zone is large enough such that the drop of the concentration due to the reaction with the sample becomes negligible. Referring to the application example of Mg_2Ni film for H_2 detection described in Sec. IV, the internal volume of the 6-way cross is 350 cm^3. The molar number of H_2 (in 96% air) at standard condition enclosed inside is 6×10^{-6}. If the density, surface area, and thickness of an Mg-Ni film sample are 2706 kg m^{-3}, 0.5 cm^2, and 30 nm, the total molar number of the alloy molecules is 5×10^{-8}. If each alloy molecule consumes four H atoms after fully hydrogenated, only 10^{-7} moles of H_2 in the gas phase are used, and the H_2 partial pressure is not substantially affected.

One more advantage of our system is that the concentrations of detected gas, balancing gas, and interferant(s) in the mixture can be determined according to the readings of the pressure gauges. Since the gaseous components are added in sequence, successive increments of pressure detected in the process represent the respective partial pressures of corresponding constituents, from which their concentrations can be derived. This approach could be more reliable and accurate than referring to the relative flowing rates of gas components, since it avoids error due to instability of MFCs and counting of flow durations.

C. Accuracy in response time measurement

Accurate determination of response time of a gas sensor requires the rate of changing gaseous environment to be much faster than the rate of reaction between the gas and sensing material. In continuous flow technique without pumping,[14,20–22] the switching of gaseous environment relies on diffusion mechanism and hence is usually smooth. In our system, evacuation of the measurement chamber is forced by a vacuum pump, and the ensuing charging step is conducted by opening a valve for the gas to rush into the measurement zone via a free expansion-like process. As such, the gaseous environment can be altered rapidly, in many cases faster than the gas-material reaction rate. The accuracy of response time measurement is thereby limited by the time constant of the electronics used for collecting the signal. In our system, the resolution of resistive response time measurement is 0.33 s, corresponding to the fastest acquisition rate of a Keithley Model 617 electrometer.

VI. CONCLUSIONS

In conclusion, we reported the design of a system used for characterizing gas sensing properties of materials. First, the design allows electrical and optical response of a material in a gas to be determined concurrently. Second, it is able to control a number of measurement parameters; including gas concentrations, temperature, total pressure, content of interferants, intensity of photo illumination, and relative humidity to be varied independently in broad ranges. As such, many performance indexes of a gas sensor of most concern, including sensitivity, stability, selectivity, response/recovery times, etc., can be evaluated and correlated with various combinations of measurement conditions. Third, full computerization/automation not only saves manpower in operation, but also facilitates the performance of long lasting and/or multicycle experiments. One important use is to explore the stability of gas sensing response according to needs of specific applications. Adoption of fast gas exchange mechanism allows the response rate of a gas sensor to be determined more accurately. All these features make the system to be highly flexible, versatile, and distinctive to many other setups used for studies in this area.

ACKNOWLEDGMENTS

This work is financially supported by Research Grants Council of the Hong Kong Administrative Region (Project No. PolyU 5016/08P, account code: B-Q10N), a PolyU internal grant (account code: 1-ZV94), and an Innovative Technology Fund (Project No. ITS/558/09, account code: ZP2U).

[1] C. Balazsi, K. Sedlackova, E. Llobet, and R. Ionescu, Sens. Actuators B **133**, 151 (2008).
[2] F. N. Dultsev and L. L. Sveshnikova, Sens. Actuators B **120**, 434 (2007).
[3] Y. Park, K. Y. Dong, J. Lee, J. Choi, G. N. Bae, and B. K. Ju, Sens. Actuators B **140**, 407 (2009).
[4] R. N. Dansby-Sparks, J. Jin, S. J. Mechery, U. Sampathkumaran, T. W. Owen, B. D. Yu, K. Goswami, K. L. Hong, J. Grant, and Z. L. Xue, Anal. Chem. **82**, 593 (2010).
[5] X. Wang and K. M. Liew, J. Phys. Chem. C **115**, 10388 (2011).
[6] N. Du, H. Zhang, X. Y. Ma, and D. Yang, Chem. Commun. **46**, 6182 (2008).
[7] V. R. Khalap, T. Sheps, A. A. Kane, and P. G. Collins, Nano Lett. **10**, 896 (2010).
[8] T. Waitz, T. Wagner, T. Sauerwald, C. D. Kohl, and M. Tiemann, Adv. Funct. Mater. **19**, 653 (2009).
[9] M. H. Yaacob, M. Breedon, K. Kalantar-Zadeh, and W. Wlodarski, Sens. Actuators B **137**, 115 (2009).
[10] C. W. Ong and Y. M. Tang, J. Mater. Res. **24**, 1919 (2009).
[11] Y. M. Tang and C. W. Ong, Int. J. Hydrogen Energy **36**, 10188 (2011).
[12] T. Kida, T. Kuroiwa, M. Yuasa, K. Shimanoe, and N. Yamazoe, Sens. Actuators B **134**, 928 (2008).
[13] S. Shukla, S. Patil, S. C. Kuiry, Z. Rahman, T. Du, L. Ludwig, C. Parish, and S. Seal, Sens. Actuators B **96**, 343 (2003).
[14] W. P. Jakubik, Thin Solid Films **517**, 6188 (2009).
[15] A. Z. Adamyan, Z. N. Adamyan, V. M. Aroutiounian, A. H. Arakelyan, K. J. Touryan, and J. A. Turner, Int. J. Hydrogen Energy **32**, 4101 (2007).
[16] P. X. Feng, H. X. Zhang, X. Y. Peng, M. Sajjad, and J. Chu, Rev. Sci. Instrum. **82**, 043303 (2011).
[17] R. K. Sharma, M. C. Bhatnagar, and G. L. Sharma, Rev. Sci. Instrum. **71**, 1500 (2000).
[18] L. Boon-Brett, J. Bousek, P. Castello, O. Salyk, F. Harskamp, L. Aldea, and F. Tinaut, Int. J. Hydrogen Energy **33**, 7648 (2008).
[19] C. Salinga, H. Weis, and M. Wuttig, Thin Solid Films **414**, 275 (2002).
[20] N. Matsuyama, S. Okazaki, H. Nakagawa, H. Sone, and K. Fukuda, Thin Solid Films **517**, 4650 (2009).
[21] M. Slaman, B. Dam, H. Schreuders, and R. Griessen, Sens. Actuators B **123**, 538 (2007).
[22] K. Kalli, A. Othonos, and C. Christofides, Rev. Sci. Instrum. **68**, 3544 (1997).
[23] H. X. Zhang, K. Liu, D. G. Jia, T. H. Xu, T. G. Liu, G. D. Peng, W. C. Jing, and Y. M. Zhang, Rev. Sci. Instrum. **82**, 023104 (2011).