



Rapid measurement of room temperature ionic liquid electrochemical gas sensor using transient double potential amperometry



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ABSTRACT

Intense study on gas sensors has been conducted to implement fast gas sensing with high sensitivity, reliability and long lifetime. This paper presents a rapid amperometric method for gas sensing based on a room temperature ionic liquid electrochemical gas sensor. To implement a miniaturized sensor with a fast response time, a three electrode system with gold interdigitated electrodes was fabricated by photolithography on a porous polytetrafluoroethylene substrate that greatly enhances gas diffusion. Furthermore, based on the reversible reaction of oxygen, a new transient double potential amperometry (DPA) was explored for electrochemical analysis to decrease the measurement time and reverse reaction by-products that could cause current drift. Parameters in transient DPA including oxidation potential, oxidation period, reduction period and sample point were investigated to study their influence on the performance of the sensor. Oxygen measurement could be accomplished in 4 s, and the sensor presented a sensitivity of $0.2863 \mu\text{A}/[\%O_2]$ and a linearity of 0.9943 when tested in air samples with different oxygen concentrations. Repeatability and long-term stability were also investigated, and the sensor was shown to exhibit good reliability. In comparison to conventional constant potential amperometry, transient DPA was shown to reduce relative standard deviation by 63.2%. With transient DPA, the sensitivity, linearity, repeatability, measurement time and current drift characteristics demonstrated by the presented gas sensor are promising for acute exposure applications.

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1. Introduction

Measurement of ambient gases is of great interests due to the critical role gases play in human health and safety. Individual exposures to gaseous hazards vary significantly in different locations and over time, requiring rapid assessment of gas concentrations for vulnerable individuals. For example, toxic gases such as NO_2 and SO_2 may lead to severe cardiac and respiratory symptoms even under low exposure [1,2], and other gases may directly cause fire and explosion hazards that seriously threaten human safety [3,4]. Oxygen is an especially important gas that is crucial for human survival, as oxygen deficiency could result in suffocation or death. Furthermore, although oxygen is nontoxic and inflammable, in high concentrations it contributes to fires and explosions acting as an oxidizer. Hence, a reliable gas sensing technology suitable for rapid monitoring of acute exposure is of great interest and necessitates investigation.

Several gas sensing approaches have matured to some level of use in practical industrial applications. Gas chromatography (GC) is a classical and very accurate method for gas separation and quantification. However, GC suffers from large instrument size, high equipment cost, long measurement time, and complicated operation requiring professionally trained personnel [5], making it unsuitable for field applications. Metal oxide semiconductor (MOS) sensors are widely used in commercial gas sensing instruments and exhibit high sensitivity, fast response time, low limit of detection (LOD) and the ability to detect many gas species [6,7]. Unfortunately, MOS-type gas sensors require heating to increase gas absorption and catalyze gas reaction [8,9], leading to high power consumption and complicated instrumentation that limits miniaturization and field deployment. They also suffer from long recovery periods that limit real-time operation in practical applications [10]. Optical gas sensors show high sensitivity, good stability and fast response time [11], but they exhibit undesirable size and cost characteristics [10]. In contrast to all of these approaches, electrochemical gas sensors exhibit excellent performance in terms of sensitivity, selectivity and LOD while also being suitable for miniaturization and low cost [12,13]. However, traditional liquid-electrolyte electrochemical gas sensors suffer from

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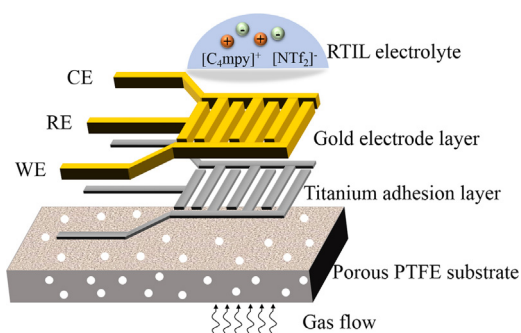


Fig. 1. Structural schematic of the microfabricated RTIL gas sensor.

inherent drawbacks in electrolyte evaporation and low gas permeation [14]. Solid electrolytes, such as Nafion membranes [15,16] and cermet membranes [17] have been investigated as a possible solution to improve gas permeation and electrolyte lifetime, yet they still suffer from limited lifetime [14].

Remarkable benefits can be achieved using room temperature ionic liquid (RTIL) as the electrolyte in electrochemical sensing, since RTILs possess negligible vapor pressure, high thermal stability and a large working potential window [18]. This has encouraged design of many RTIL-based gas sensors that exhibit good performance and demonstrate the value of RTIL. Nevertheless, due to RTIL's high viscosity, low gas permeation still remains a critical issue [12,14]. Two approaches that have been introduced to address this limitation are decreasing RTIL thickness and avoiding gas permeation through RTIL [19]. In our previous work, the second approach was adopted in a sensor structure with a porous polytetrafluoroethylene (PTFE) substrate through which gas could quickly diffuse [18,20]. This sensor utilized constant potential amperometry, which is a classic electroanalytical method for gas detection [12,21] that continuously applies a constant potential while recording the oxidation or reduction current for qualitative and quantitative gas analysis. Although this method can record real-time current changes over a long time period, reaction by-products could accumulate on the surface of the working electrode due to the high viscosity of RTIL [22], which would cause adverse effects on diffusion of target gas and current response. Furthermore, the measurement by constant potential amperometry is generally conducted after full decay of the charging current that requires long time to stabilize. R. Wang et al. [23] reported a potential-step chronoamperometry method for oxygen detection with reversible reactions, but the 40 s measurement time is still slow for real time health and safety applications. To meet the requirements of acute gas exposure applications, the sensor must respond rapidly to changes in gas concentration, and the measurement time must be on the order of seconds. To achieve this goal, this work introduces a new transient double potential amperometry (DPA) method for

rapid gas detection with RTIL-based gas sensors by utilizing transient currents and reversible reactions of oxygen.

In this paper, a microfabricated gold interdigitated gas sensor with RTIL electrolyte was used for oxygen detection in air. Porous PTFE was utilized as the sensor substrate to speed target gas diffusion to the sensing electrode, which was formed directly on the porous PTFE using photolithography and custom microfabrication procedures. Cyclic voltammetry and the new transient DPA method were used for sensor characterization and analysis of oxygen measurement performance. A comparison between constant potential amperometry and transient DPA was conducted to validate the superiority of transient DPA in oxygen sensing.

2. Experimental

2.1. Methods and apparatus

High purity nitrogen and compressed air (21% oxygen) were purchased from Airgas Inc. for background gas and oxygen targets, respectively. RTIL 1-butyl-1-methylpyrrolidinium bis-(trifluoromethylsulfonyl)-imide ($[C_4mpy][NTf_2]$) (IOLITEC, Inc.) was chosen as the electrolyte for oxygen sensing because it presented good performance in our previous work [18,24]. A CHI 760 (CH Instrument, USA) instrument was utilized for all electrochemical tests. A Gas Blender 103 (MCQ Instrument, Italy) was used for automatic gas mixing and flow control. In all tests, the gas flow rate was set to 200 standard cubic centimeter per minute (sccm). Cyclic voltammetry was employed to characterize reactions in RTIL. Transient DPA was implemented for oxygen measurement in air using the multi-potential steps method within the CHI software, as detailed in Section 2.3. Constant potential amperometry was employed for comparison with transient DPA.

2.2. Sensor design and implementation

Porous PTFE offers excellent thermal and chemical stability as well as good permeability as a gas sensing substrate [25,26]. These properties permit the microfabrication of a PTFE-based sensor with fast gas diffusion through PTFE substrate. Microporous PTFE (POREX, USA) with 4 μm pore size and 35% porosity was chosen for the sensor substrate. Gold was chosen as the sensing electrode because it is a readily available material for microfabrication and has shown good performance for oxygen sensing in many other studies [27–29]. To form a complete and miniaturized gas sensor, three electrodes including working electrode (WE), counter electrode (CE) and quasi-reference electrode (RE) were designed to be integrated on the same porous PTFE substrate using the interdigitated structure shown in Fig. 1. Beneath the gold electrode, a thin layer of titanium was included to enhance adhesion of electrodes to the porous PTFE substrate. Each WE/CE electrode finger was designed to be 1.8 mm \times 250 μm , and the gap between electrodes was set to 150 μm . Both WE and CE had three fingers to

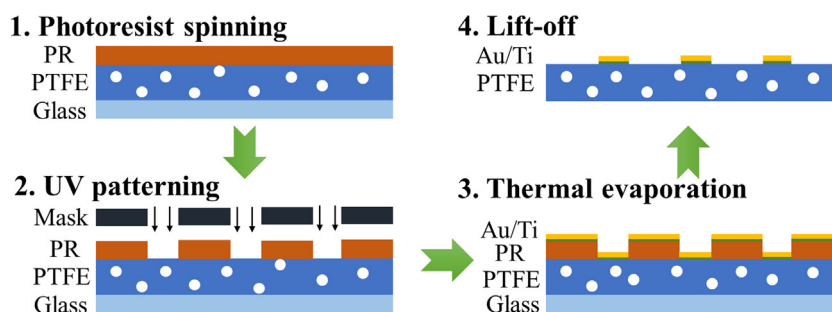


Fig. 2. Microfabrication process flow for formation of sensor electrodes on porous PTFE substrate.

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