Chemiresistor-type NO gas sensor based on nickel phthalocyanine thin films

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Abstract

The sensing characteristics of nickel phthalocyanine (NiPc) thin films for use in a chemiresistor-type nitric oxide gas sensor are discussed. The gas-sensing properties, including current transient, sensitivity, response time, and aging, are studied. A kinetic model proposed in the literature for sensing NO₂ with lead phthalocyanine (PbPc) thin films, in which adsorption involves displacement of surface adsorbed O₂ from a range of heterogeneous sites, can be used to explain our experimental results. It is inferred that exposure to NO concentrations lower than 50 ppm only involves the population of weak adsorption sites, while exposure to NO concentrations higher than 50 ppm may involve the population of stronger adsorption sites. Repeated step changes in NO concentrations of 50 ppm could result in a significant change in response currents within the first five cycles, presumably due to the presence of an irreversible adsorption. Further analysis of current responses reveals that the lower the concentration of NO, the larger the sensitivity. For a lower concentration range, between 5 and 50 ppm NO, the sensitivity lies between 0.41 and 0.42, while for a higher concentration range, between 50 and 500 ppm, the sensitivity decreases to about 0.17 to 0.19. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

Metal phthalocyanine thin films have been proposed for use as printing inks, colorants, photoconductors, solar cells, electrochromics, and gas sensors. As for the sensor applications, the principle is based on the change of conductivity upon adsorption of gas species at the surface [1,2]. Most of the metal phthalocyanines (MPcs) [3–17] are p-type semiconductors with good thermal and chemical stabilities, whereas copper tetra-4-(2,4-di-*tert*-amylphenoxy) phthalocyanine (tap CuPc) is a n-type semiconductor [18]. For p-type MPcs, their conductivity increases following adsorption of oxidizing molecules, because of the generation of hole carriers induced by the formation of charge transfer complexes at the surface of the phthalocyanines. It is well known that various gases in ambient environment can affect the semiconductivity of a number of metal phthalocyanines. A partial list of the metal phthalocyanines for gas-sensing is given in Table 1.

Even though the gas-sensing properties involving NO₂ and MPcs, as shown in Table 1, are well studied, few studies have been carried out on exploring the gas-sensing properties of NO using metal phthalocyanines. The aim of this

research is to investigate the gas-sensing property of vacuum-deposited NiPc films for NO detection. Liu et al. [19] derived an unsteady-state current increment–time ($\Delta I-t$) relation which takes into account the adsorption–desorption at the gas/thin film interface. Our experimental data for the current responses are consistent with this model. Various parameters, as defined in the model, have been extracted from the experimental results. The results showed that the lower the NO concentration, the larger the sensitivity. Moreover, the theoretical relationship between the film thickness and the response current, as predicted by the model, is verified experimentally. The effect on gas response characteristics will also be discussed.

2. Experimental

2.1. Preparation

Nickel phthalocyanine (NiPc) (Strem Chem. 94%) was purified twice by sublimation in a vacuum glass tube at 5.0×10^{-4} Torr and 500°C. Purified powders were vaporized and condensed onto Al₂O₃ substrates in a diffusion-pumped vacuum system operating at a base pressure of 1.8×10^{-5} Torr. Al₂O₃ substrates were screen-printed with gold interdigitated electrodes with 200 µm electrode

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Table 1
A partial list of metal phthalocyanines for gas-sensing

MPcs	Types	Applicable gases	References
CuPc	p-type	NO ₂ , NH ₃	[3]
NiPc	p-type	NO ₂	[4]
PbPc	p-type	NO ₂	[5–9]
		Cl ₂	[10]
		H ₂	[11]
(AlPcF) _n	p-type	NO ₂ , O ₂	[12–14]
Lu(Pc) ₂	p-type	NO ₂ , HCl, H ₂ S, SO ₂	[15]
TiOPc	p-type	NO ₂	[16]
FePc	p-type	NO ₂	[17]
tap CuPc	n-type	NH ₃	[18]

spacing. The evaporated NiPc films were deposited under vacuum as thin layers by controlling the evaporation rate and thickness. The deposition rates were kept between 0.1 and 5 nm/s, the substrate temperature was kept at 25°C. Typical film thickness for gas-sensing experiments was maintained at 100 nm with an oscillating quartz-crystal thin film monitor. NO gas diluted with ultra-high purity N₂ (99.9995%) was passed through the test chamber at flow rates of 100–500 ml/min, controlled by a mass flow controller (Sierra, Model 840). The dc conductivity of the NiPc sensing unit was measured using a power supply (Keithley, Model 236) with a source voltage of 10 V. X-ray diffraction experiments were performed on a Rigaku diffractometer (Model RTP-300RC) using monochromatized Cu K_α incident radiation.

2.2. Sensing chamber

The sensor was an alumina substrate (12 mm square, 1 mm thick) with an interdigitated electrode pattern over which a thin NiPc film was vacuum-deposited. The sensor was mounted on a base located at the bottom of the sensing glass chamber, as shown in Fig. 1.

Fig. 1. Chamber design in a sensing unit: (a) NiPc-coated Au-interdigitated sensing electrode; (b) thermal couple.

3. Elovich kinetics and equilibrium kinetics

Passard et al. [14] observed two different kinetics for the interaction of the oxidizing gases O₂ and NO₂ with (AlPcF)_n thin film. They proposed a doping mechanism to separate the physical process from the chemical process. For a constant heat of adsorption, independent of the surface coverage, the chemisorption kinetics [1] follow the Elovich equation:

$$\frac{d\theta}{dt} = a \exp(-b\theta) \quad (1)$$

It can be seen from Eq. (1) that the fractional surface coverage (θ), and hence the conductivity or the net sensing current, varies linearly with log(time) rather than with linear time. Archer et al. [8] used the Elovich equation to explain the kinetic data for the adsorbed NO₂ on PbPc. With adequate time allowed for the attainment of surface coverage equilibrium, Liu et al. [19] recently used a simple adsorption–desorption equilibrium model to interpret the response characteristics of NO₂–PbPc interaction. As our typical current response data for the NO–NiPc interaction does not indicate two dramatically different kinetic processes and that the Elovich kinetics is suitable only for a qualitative explanation, we will adopt the following adsorption–desorption equilibrium model and assume that the pseudo-equilibrium state can be achieved within the time interval of interest:

Let us assume that k_a and k_d represent the rate constants for adsorption and desorption, respectively. A total site balance gives

$$\frac{dN_s(t)}{dt} = k_a C_0 [N_0 - N_s(t)] - k_d N_s(t) \quad (3)$$

where C_0 is the bulk concentration of NO, N_s the surface concentration of the adsorbed NO, N_0 the total sites concentration available on the surface, and $[N_0 - N_s(t)]$ represents the surface concentration of the unoccupied sites. The surface coverage, θ , is defined as the ratio of the adsorbed sites concentration to that of the total sites concentration, i.e.

$$\theta = \frac{N_s}{N_0} \quad (4)$$

Eq. (3) becomes

$$\frac{d\theta}{dt} = k_a C_0 [1 - \theta] - k_d \theta \quad (5)$$

The equation can be solved with the initial condition of $\theta(0) = 0$. The surface coverage, θ , as a function of time, is written as

$$\theta = \frac{KC_0}{1 + KC_0} [1 - \exp(-(k_a C_0 + k_d)t)] \quad (6)$$

where $K = k_a/k_d$ is the equilibrium constant. For simplicity, we assumed that the net sensing current (ΔI) resulting from the adsorption–desorption process is proportional to the adsorbed gas concentration, N_s , or the surface coverage, θ , i.e.

$$\Delta I = k\theta(t) \quad (7)$$

where k is the proportionality constant. The net sensing current can be put into the following form:

$$\Delta I = \Delta I_{ss}[1 - \exp(-(k_a C_0 + k_d)t)] \quad (8)$$

where ΔI_{ss} is the steady-state current, and is defined as

$$\Delta I_{ss} = \frac{kKC_0}{1 + KC_0} = k\theta(\infty) \quad (9)$$

In gas sensor application, a power law of the form $\Delta I \propto C_0^S$ is observed [20]. The power S can be determined from the slope of the all-logarithm plot of ΔI versus C_0 , i.e.

$$S = \frac{d \ln(\Delta I)}{d \ln C_0} = \frac{d \log(\Delta I)}{d \log C_0} \quad (10)$$

The value of S stands for the relative sensitivity of the thin film.

4. Results and discussion

Previous workers have studied the effect of the NO_2 concentration on the current response of NiPc films, but the NO concentration effect on the sensing properties of NiPc films has not been discussed before. The present data, together with other related published results, now permit a fuller discussion of the sensing characteristics for the interaction between NO gas and NiPc thin films.

4.1. Morphology

It is well known that the morphology of the vacuum-deposited metal phthalocyanines thin film depends on its growth conditions. For NiPc films deposited on Al_2O_3 substrates at rates between 0.1 and 5 nm/s and substrate temperature of 25°C, XRD data in Fig. 2 show the same peak positions for all of the samples. There is only one small peak which is not due to the underlying Al_2O_3 substrate, but the characteristic peak at $2\theta = 6.3^\circ$ for the α -phase NiPc.

4.2. Response characteristics

Upon exposure to NO gas, the electrical conductivity of the NiPc film increases. Fig. 3 shows the response characteristics of NiPc films when exposed to 250 ppm NO in N_2 at 160°C. The inset in Fig. 3 is a replot of current versus $\log(\text{time})$ with data taken from Fig. 3. A straight line can be obtained with no sign of reaching equilibrium within 60 min. Note that the response of the NiPc film is rather slow; generally it takes more than 500 min to reach the

Fig. 2. XRD diagrams of NiPc film prepared under various evaporation rates set at 0.1, 0.3, 2.0, and 5.0 nm/s. Film thickness: 100 nm, substrate temperature: 25°C, substrate material: Al_2O_3 .

pseudo-steady-state. It can be found from Fig. 4 that, for NiPc films made with the same thickness, i.e. 100 nm, the faster the deposition rate, the sooner it will take to reach the pseudo-steady-state, and the smaller the response current.

4.3. Concentration effect

The response currents of a typical NiPc film exposed to different NO concentrations, following an increasing concentration order of 5, 10, 20, 40, 50, 100, 300, and 500 ppm, were recorded. Fig. 5 shows the consecutive current responses when NO concentrations were increased sequentially from 5 to 500 ppm. Both adsorption and desorption times were set at 20 min. At the end of each desorption, the surface still contained traces of NO adsorbed at some

Fig. 3. Response current of NiPc film exposed to 250 ppm NO in N_2 at 160°C with film thickness: 100 nm, substrate temperature: 25°C, evaporation rate: 1 nm/s, mass flow rate 200 ml/min. The inset is a replot of current vs. $\log(\text{time})$, showing the slow Elovich kinetics.

Fig. 4. Response currents of NiPc films deposited at various evaporation rates. The films were continuously exposed to 250 ppm NO in N₂ at 160°C for 8 h with film thickness: 100 nm, substrate temperature: 25°C, mass flow rate 200 ml/min.

adsorption sites, unless the desorption process was allowed to continue for a very long time. This is consistent with a kinetic model [8], proposed for NO₂ sensor using PbPc thin films, in which adsorption involves the displacement of surface adsorbed oxygen from a range of heterogeneous sites. Although ultra-high purity N₂ was used in this study, the purity level still allows 5 ppm of oxygen. Fig. 6 is a plot for the current increase versus NO concentration. The circles in Fig. 6 were obtained from the experimental data, while the curve is a model fit based on the adsorption–desorption kinetics with $k = 10.10 \mu\text{A}$ and $K = 0.042 \text{ ppm}^{-1}$. The sensitivity plot in Fig. 7 gives the sensitivity (S) values of 0.413 and 0.174 for NO concentrations in the ranges 5–50 ppm and 50–500 ppm, respectively. It is worthwhile to

Fig. 5. Response current of NiPc film sequentially exposed to 5, 10, 20, 40, 50, 100, 300, and 500 ppm NO in N₂ at 160°C. Film thickness: 100 nm, substrate temperature: 25°C, evaporation rate: 0.1 nm/s, mass flow rate 200 ml/min, adsorption time: 20 min, desorption time: 20 min without aerating N₂.

Fig. 6. Current increase of NiPc film sequentially exposed to 5, 10, 20, 40, 50, 100, 300, and 500 ppm NO in N₂ at 160°C. Operating conditions are the same as those in Fig. 5. The circles are obtained from the experimental data, while the curve is a model fit based on the adsorption–desorption kinetics.

mention that the reported S values are the pseudo-sensitivity data, as the circles in Fig. 6 represent the pseudo-equilibrium data.

In order to test the reversibility of the NO–NiPc interaction, the same NiPc film exposed to different NO concentrations, following the reverse concentration order in Fig. 5, were also recorded. Figs. 8–10 are the corresponding results for the concentration variation following the decreasing concentration order. In this sensing experiment, adsorption time was set at 20 min while complete desorption was ensured by allowing the current to return to the background value, as shown in Fig. 8. The fact that these desorption times become shorter as the NO concentration decreases is strong evidence that adsorption involves displacement of

Fig. 7. All-logarithm plot of current increment vs. NO concentration, showing the sensitivity of NiPc film on NO sensing. Operating conditions are the same as those in Fig. 5. The circles were obtained from the experimental data, while the straight lines are the best model fits.

Fig. 8. Response current of NiPc film sequentially exposed to 500, 300, 100, 50, 40, 20, 10, and 5 ppm NO in N_2 at 160°C. Film thickness: 100 nm, substrate temperature: 25°C, evaporation rate: 0.1 nm/s, mass flow rate 200 ml/min, adsorption time: 20 min, desorption time: desorbed completely without any gas.

surface adsorbed O_2 from a range of heterogeneous sites, as proposed by Archer et al. [8] and Wilson et al. [9]. Hence the sensing mechanism for NiPc involving NO and O_2 can be described as follows: exposure to low NO concentrations only involve the population of weak adsorption sites, from which O_2 is readily displaced, hence, the reversal is rapid; exposure to higher NO concentrations involves the population of stronger adsorption sites, which are depopulated more slowly on reversal. Fig. 9 is a plot of the current increase versus NO concentration. The circles in Fig. 9 were obtained from the experimental data, while the curve is a model fit based on the adsorption–desorption kinetics with $k = 10.87 \mu A$ and $K = 0.041 \text{ ppm}^{-1}$. The sensitivity plot in Fig. 10 gives the sensitivity (S) values of 0.186 and 0.423 for NO concentrations in the ranges 500–50 ppm and

Fig. 9. Current increase of NiPc film sequentially exposed to 500, 300, 100, 50, 40, 20, 10, and 5 ppm NO in N_2 at 160°C. Operating conditions are the same as those in Fig. 8. The circles are obtained from the experimental data, while the curve is a model fit based on the adsorption–desorption kinetics.

Fig. 10. All-logarithm plot of current increment vs. NO concentration, showing the sensitivity of NiPc film on NO sensing. Operating conditions are the same as those in Fig. 8. The circles were obtained from the experimental data, while the straight lines are the best model fits.

50–5 ppm, respectively. In fact, the sensitivity of the NO–NiPc interaction decreases when the NO concentration is increased from low to high with a transition occurring at about 50 ppm, as shown both in Figs. 7 and 10. Therefore, it is concluded that exposure to NO concentrations lower than 50 ppm only involves the population of weak adsorption sites, while exposure to NO concentrations higher than 50 ppm involves the population of stronger adsorption sites. Table 2 summarizes the values of the best fitted parameters, k and K , and the sensitivity S . The sensitivity depends on the concentration range of NO. For a higher concentration range, between 50 and 500 ppm NO, the sensitivity lies between 0.174 and 0.186, while for a lower concentration range, between 5 and 50 ppm, the sensitivity increases to about 0.413–0.423. Therefore, it is expected that the sensitivity would be improved when NiPc films were used for sensing low concentration NO.

4.4. Mass flow rate/cooling effect

The effect of the mass flow rate or the cooling effect on the current responses of NiPc film exposed to 5 ppm NO in N_2 is shown in Fig. 11. The preparation and operating parameters are: film thickness: 100 nm, substrate temperature: 25°C, operating temperature: 160°C, evaporation rate: 0.1 nm/s, adsorption time: 20 min, desorption time: complete desorption without any gas. No attempt was made to pre-heat the

Table 2
Effect of concentration range on the sensitivity

C_0 (ppm)	k (μA)	K (ppm^{-1})	S
5–50	10.10	0.042	0.413
50–500	10.10	0.042	0.174
500–50	10.87	0.041	0.186
50–5	10.87	0.041	0.423

Fig. 11. The effect of mass flow rate on the current response of NiPc film exposed to 5 ppm NO in N₂. Film thickness: 100 nm, substrate temperature: 25°C, operating temperature: 160°C, evaporation rate: 0.1 nm/s, adsorption time: 20 min, desorption time: complete desorption without any gas.

gas to the same temperature as the sensing chamber before admitting the gas to the chamber. The temperature in the chamber reached a steady-state value 20 min after the gas was allowed into the chamber. The steady-state temperatures recorded were 160, 159.5, 159, 158, and 157°C for curves (a)–(e), respectively. The results indicate that, under the same NO concentration, the larger the mass flow rate, the larger the cooling effect, and thus the smaller the response current. The cooling effect may be neglected in a realistic sensing as usually the mass flow rate is rather low.

4.5. Repeated sensing

Repeated sensing was carried in a 50 ppm NO with N₂. Fig. 12 shows the result from the repeated sensing current. During sensing, adsorption time was set at 20 min while

Fig. 12. Repeated response current of NiPc film exposed to 50 ppm NO in N₂ at 160°C for the first nine cycles. Film thickness: 100 nm, substrate temperature: 25°C, evaporation rate: 0.1 nm/s, mass flow rate 200 ml/min, adsorption time: 20 min, desorption time: desorbed completely without any gas.

complete desorption was ensured by allowing the current to return to the background value. It can be seen from Fig. 12 that the sensing becomes reasonably stable after five sensing cycles, which is acceptable in a realistic sensing environments.

5. Conclusions

This work reported on the sensing characteristics of nickel phthalocyanine (NiPc) thin films for use in a chemiresistor-type nitric oxide gas sensor. The main conclusions from this study are as follows.

1. A kinetic model proposed in the literature for the NO₂–PbPc interaction, in which adsorption involves displacement of surface adsorbed O₂ from a range of heterogeneous sites, can be used to explain our experimental results for the NO–NiPc interaction. We concluded that exposure to NO concentrations lower than 50 ppm only involves the population of weak adsorption sites, while exposure to NO concentrations higher than 50 ppm may involve the population of stronger adsorption sites. Our experimental data for the current response were qualitatively consistent with the model.
2. Quantitatively, we adopted the pseudo-steady-state current increment versus time (ΔI versus t) relationship based on the adsorption–desorption process at the gas/thin film interface to quantify the sensitivity and model parameters. The sensing results of NO with NiPcs showed that the lower the NO concentration, the larger the sensitivity. For a lower concentration range, between 5 and 50 ppm NO, the sensitivity lies between 0.41 and 0.42, while for a higher concentration range, between 50 and 500 ppm, the sensitivity decreases to about 0.17 to 0.19.
3. The experimental data revealed that repeated step changes in a NO concentration of 50 ppm could result in a significant change in response currents for the first five sensing cycles, presumably due to the presence of an irreversible adsorption. The sensing becomes relatively stable after five sensing cycles.
4. It is possible to fabricate chemiresistor-type sensors using MPc thin films with M being nickel for sensing NO gas in the concentration range below 50 ppm.

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