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Properties of SnO₂ based gas-sensing thin films prepared by ink-jet printing

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ABSTRACT

SnO₂ precursor inks with appropriate viscosity and surface tension were prepared using the sol-gel technique with anhydrous ethanol as the primary solvent. The inks were printed on both alumina ceramic and silicon substrates using an ink printing apparatus produced by modifying a commercial printer. SnO₂ based gas-sensing films with different thicknesses and additives were formed, and the morphologies and electrical properties of these films were investigated by scanning electron microscopy (SEM), X-ray diffraction (XRD) and an instrument designed to measure the gas sensing abilities of the films. A linear relationship was observed between the natural logarithm of the electrical resistance of the SnO₂ films and the reciprocal of the films' absolute working temperature in the range between 20 °C and 265 °C. The electrical and gas-sensing properties of the films changed significantly with the thickness of the films. The film thickness can be easily adjusted by modifying the number of printing events. The SnO₂ films selectivity for various gases could be modified by utilizing different additives. This work also showed that ink-jet printing was a convenient and low-cost method to prepare gas-sensing films with controlled film thickness and additive level.

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1. Introduction

As an important metal oxide semiconductor, SnO₂ has been widely used as gas-sensing material for many years [1]. These sensors have increased in importance and play an essential role in the fields of industrial processing, environmental protection and medical treatment. However, many problems need to be solved to use these sensors in other applications, and significant research is being conducted in these fields to further improve the selectivity, sensitivity and stability of the sensors. Active research areas include introducing different additives [2], fabricating multilayer films [3], producing electronic nose by network recognition techniques [4] and utilizing new techniques to prepare sensing films [5]. In recent years, increasing attention has been focused on SnO₂ based thin-film sensors composed of nanoparticles and nanostructures because of these film's excellent properties, such as small size, high sensitivity, good stability, fast response and recovery speed. Until recently, many traditional film deposition techniques have been used to prepare gas sensing films including screen printing, both physical and chemical vapour deposition (PVD and CVP), and sol-gel based dip and spin coating [6]. However, these methods do not fulfil all the necessary requirements for high quality sensors. This is a significant problem when depositing films with complex compositions or when integrating several different gassensing films onto one chip to make an electronic nose. Compared with traditional deposition methods, ink-jet printing is a promising technique with the potential to deliver precise quantities of materials while simultaneously forming predesigned patterns [7].

The ink-jet printing process is a contactless technique of printing, and it is widely used for desktop publishing. Recently, because of its many unique advantages, such as the potential for vacuumless processing, high throughput combinatorial chemistry, low temperature processing, low cost, and low materials waste, inkjet printing has received much attention in material and device preparation [5,8–11]. The use of ink-jet printing for the synthesis of different functional films had been studied for a variety of applications such as thin-film transistors (TFT) [12–14], ultra-thin film electrodes [15,16], optical chemical sensors [17,18], antennas for radio frequency identification (RFID) [19], and organic films for organic light-emitting diode (OLED) [20]. However, to the best our knowledge, there are only a few reports concerning the preparation of SnO₂ based gas sensing thin films using ink jet printing.

In this paper, an ink jet printing technique was used to prepare SnO_2 based gas-sensing films. The materials to be deposited were dissolved or dispersed in anhydrous ethanol and then used as precursor inks. When inks with appropriate surface tension and viscosity were injected into the cartridge, they could be printed directly onto substrates by the ink-jet printing apparatus. As more than four kinds of inks can be printed simultaneously with a common colour ink-jet printer, films with different chemical

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Fig. 1. This diagram illustrates the preparation of the metal-oxide gas sensing inks and the thin films.

ingredients could be deposited with controlled patterns. SnO₂ based thin films were prepared using this technique, and the structural, electronic and sensing properties of these films were studied.

2. Experimental

2.1. Preparation of precursor inks and SnO₂ based thin films

All reagents used were analytical grade reagents. SnO₂ based precursor inks were prepared as shown as Fig. 1. Both SnCl₂·2H₂O (0.05 mol) and oxalic acid (0.01 mol) were dissolved into 100 mL of anhydrous alcohol. This mixture was stirred for 1 h, resulting in a slightly turbid solution. The sealed solution was then heated at 65 °C in a water bath for 72 h until a yellow clear colloidal solution was obtained. This solution was marked as ink A and was used as the 0.5 mol/L SnO₂ precursor ink. Various additive inks, such as Au, Ag, Pd, Co, Ni, Cu, Sb and Zr were also prepared as shown as Fig. 1. For example, the 0.01 mol/L Au additive precursor ink was synthesised by adding 5×10^{-4} mol of AuCl₃·HCl·4H₂O into 50 mL of anhydrous alcohol. This mixture then underwent ultrasonic dispersion for 15 min. Using the above method, 0.01 mol/L Ag, Pd, Co, Ni, Cu, Sb and Zr were prepared.

The ink jet printing technique used in this paper was similar to the technique used in our previous work [21]. The pure SnO_2 precursor ink and the different additive inks were loaded into different cartridges and then printed onto a monocrystalline silicon wafer or an alumina substrate coated with interdigital gold electrodes using our modified ink jet printer. If ink was printed onto a substrate more than once, the sample was allowed to dry in air for 3 min in-between printings. When printing was complete, the samples were kept in air at room temperature. After 24 h, the samples formed gelled films that were then sintered at 550 °C for 1 h.

2.2. Characterisation of SnO₂ based thin films

Structural characterisation of the SnO₂ thin films was performed using X-ray diffractometry (XRD) using a Riaku D/max 2400 diffractometry with Cu K α , λ = 0.1542 nm. The surface morphology of the samples was examined by a scanning electron microscope (SEM, JSM-6301F). The films resistance and response to various gases were measured by the apparatus described in a previous paper [21]. The temperature of this apparatus can be set between room temperature and 500 °C, and the resistance of the films was recorded every 0.3 s. At a certain temperature and humidity, sensor response (*S*) was defined as $S = R_a/R_g$, where R_a was the sample resistance in air, and R_g was the sample resistance when the detected gas was injected into the measuring apparatus.

3. Results and discussion

3.1. Microstructure

Fig. 2 shows the XRD patterns of the SnO_2 thin films that were formed by printing ink A different times onto the monocrystalline silicon substrates and subsequently annealing the samples at 550 °C for 1 h. The diffraction peaks of the SnO_2 films were



Fig. 2. XRD patterns of SnO₂ thin films prepared by varying the number of printing events.

very weak because the films were thin. However, peaks can be observed clearly at 26.5°, 33.7° and 51.8°, which were attributed to the diffraction from the (110), (101) and (211) crystalline plane of the rutile structure of SnO₂. As shown in Fig. 2, the diffraction intensity of SnO₂ increased significantly when the number of printing steps was increased. Using the Scherrer formula, the grain size of the SnO₂ film was estimated to be approximately 10 nm. There is a peak which is attributed to the diffraction from the (111) crystalline plane of the Si substrate (PDF #895012) is visible nearly at 28.4° when the film printed once or twice in Fig. 2. And this peak is absent when the SnO₂ film printed more than twice, as shown as curve c and curve d. Fig. 3 shows the TEM image of the sample obtained by annealing ink A at a temperature of 550 °C for 1 h. The grain size of sample in the TEM images was approximately 10 nm, which was in agreement with the result obtained from the XRD pattern. After the gelation and sintering process, the SnO₂ sol films printed on the substrate were well crystallised.

After annealing at 550 °C, the SnO₂ thin films prepared by printing ink A 8 times on either a monocrystalline silicon wafer or an alumina substrate were cross-sectioned and observed by SEM as shown in Fig. 4(a) and (b). Fig. 4(a) shows that the thickness of the SnO₂ films printed 8 times on a Si wafer was approximately 7 µm. Large cracks can be observed on the surface of the film. When printed on an alumina substrate, the inks penetrated into the surface of the substrate because of the material's porous structure. Fig. 4(b) shows that the thickness of the interface of the SnO₂ layer and Al_2O_3 substrate was approximately 200 µm, while the pure SnO₂ layer had a thickness of approximately 6 µm. The SnO₂ film deposited on the alumina substrate was also cracked in multiple locations. Generally, cracks would appear when the films were prepared by printing an ink multiple times and then directly heated the sample above 500 °C. To reduce cracking, films were annealed at 300 °C for 15 min each time the ink was printed. During this procedure, the subsequently printed inks would fill the cracks in the film formed during the annealing process, significantly reducing



Fig. 3. TEM image and selected-area diffraction (SAED) pattern of SnO_2 nanoparticles obtained by annealing ink A at 550 $^\circ C$ for 1 h.

the number of cracks in the final film. After printing, the samples were annealed at $550 \,^{\circ}$ C for 1 h. SEM images of the SnO₂ films printed twice and three times on alumina substrates are shown in Fig. 5(a) and (b), respectively. Double layers were clearly observed for the SnO₂ films prepared by two printing events. There were cracks on the bottom layer while the top layer was quiet smooth. Uniform SnO₂ films with fewer cracks were obtained by increasing the number of times that an ink was printed. As shown in Fig. 5(b), there were only a few cracks on the surface of the SnO₂ films that were prepared by printing three times.



Fig. 4. SEM micrographs of the cross-section of SnO₂ thin films printed 8 times either on (a) a monocrystalline silicon substrate or (b) an alumina substrate.



Fig. 5. SEM micrographs of SnO₂ films on an alumina substrate obtained by printing either (a) two or (b) three times.



Fig. 6. Resistance-temperature curves of SnO₂ films printed for a various number of times.

3.2. Electrical properties of pure SnO₂ thin films

The thickness of the SnO_2 films could be controlled by changing the number of times an ink was printed, and the relationship between the film's electrical properties and the film's thickness was studied by a sensor response measuring system [21].

Fig. 6 illustrates the change in the temperature dependant resistance of the SnO₂ films that results from varying the number of printing times from two to five. Increasing the number of printing times results in an increase in the film's thickness and a decrease in the film's resistance. At a temperature of 375 °C, the resistance of a film that was printed twice was approximately 120 k Ω . The resistance decreased to 10 k Ω when the film was printed three times. When the films were printed 4 or 5 times, their resistances were lowered to 5.9 k Ω and 2.2 k Ω , respectively. The most dramatic resistance decrease occurred when the number of printing times was increased from two to three. The high resistance of the films printed twice may be due to the discontinuity of the film, as shown in Fig. 5(a). When the film was printed more than two times, it became more continuous, as shown as Fig. 5(b), and therefore, its resistance was reduced significantly.

When the films were prepared by printing ink A more than twice, the relationship between resistance and temperature between 50 °C and 425 °C was typical for a metal oxide semiconductor. As shown in Fig. 6, for the SnO₂ films printed 3 and 4 times,

there were three regions in the plot of resistance versus temperature, with two inflection points at 160 °C and 240 °C. For the films printed 5 times, the two inflection points were observed at 120 °C and 320 °C. When the temperature was below the first inflection point, the resistance decreased with temperature. When the temperature was between the two inflection points, the resistance increased with the temperature. When the temperature was higher than the second inflection point, the resistance again decreased with temperature.

3.3. Stability of SnO₂ thin films

Before performing sensor response measurements, samples were treated to an ageing protocol to increase the sample's stability. A SnO₂ film was prepared by sintering a film formed by printing ink A five times at 500 °C for 1 h. Then, the film was placed into a drying oven at 60 °C for 2 days. The sample's electrical property was measured and is shown in Fig. 7(a). The resistance of the film was measured as the temperature was varied from room temperature to 770 K over a 24 h (1 day) period. This measurement was repeated one time each day. And the initial experimental relative humidity was about 15% at the temperature of 20 °C. The samples were stabilised at the each testing temperature for half an hour before the measurement was performed. Fig. 7(a) shows that a large resistance increase occurred between day 1 and day 2 of the measurement. Fig. 7(a) also shows that the resistance of the films increased gradually with ageing time. Five days later, the resistance of the film became stable when it was measured at the working temperature of the gas sensors in the range of 570-770 K.

According to Morrison [22], the relationship between the resistance and the absolute temperature can be expressed as:

$$R = R_0 \exp\left(\frac{eV_s}{kT}\right) \tag{1}$$

where eV_s is the barrier energy between the grains and R_0 represents the bulk intergranular resistance and geometrical effects. R_0 can be considered relatively insensitive to the temperature when compared with the exponential term, and as a first approximation, this constant can be considered independent of temperature [23]. The other symbols have their usual meaning. Logarithm of Eq. (1) is taken, and it becomes

$$\ln R = \frac{eV_s/k}{T} + \ln R_0 \tag{2}$$

From Fig. 7(a), the relationship between the natural logarithm of resistance and the reciprocal of the temperature in Kelvin was



Fig. 7. The resistance-temperature curves of SnO₂ films with several different measuring times. (b) The resistance and temperature characteristics of SnO₂ films with different measuring times.

114 **Table 1**

The grain-boundary h	parriers of SnO ₂ th	hin films were calculated by	the Morrison Grain model at low temperature
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	Day 2	Day 3	Day 4	Day 5	Day 7	Day 15	Day 16
Inflection point (°C)	263	269	266	264	273	276	277
eV_s/k (K)	11.862	17.143	19.763	20.656	22.003	22.018	22.245
eV_s (10 ⁻²³ I)	16.381	23.675	27.293	28.526	30.386	30.407	30.720

obtained as shown in Fig. 7(b). There was one inflection point in this curve. When the measured temperature was below this inflection point, the natural logarithm of resistance increased linearly with reciprocal temperature, which allowed for the calculation of the slope and eV_s using Eq. (2) as shown in Table 1. The grain barrier energy (eV_s) increased significantly from day 1 to day 2 and then grew gradually with an increase in ageing time. The daily high temperature measuring process would result in new micro cracks appeared or old cracks extended, but at the same time its grain and old crack would be further stability. The thin film ageing process is actually an equilibrium conversion process of the film internal microstructure. That is the various defects decrease slowly and becomes gradually uniform distribution. Table 1 indicated that the internal microstructure of the film became more stable after 7 days.

As the measurement was performed in air with the fixed relative humidity of about 15% when the measuring temperature was $20Y^{\circ}C$, the variation in the grain barrier energy was primarily influenced by the density of oxygen ions absorbed on the surface. When the samples were measured at temperatures below the inflection point, chemisorbed oxygen on the SnO₂ film surface was mainly in the form of O₂⁻¹. This form of oxygen was relatively stable at these temperatures [23,24], and therefore, eV_s would remain constant. When the temperature was above the inflection point, O_2^{-1} began to convert to O^{-1} and O^{-2} by capturing electrons from the surface of the SnO₂ film [25]. The concentration of O^{2-} is an important role to detect the reduce gas. There would be better response for reducing gas when the concentration of O^{2-} increased. Thus, the working temperature of the SnO₂ gas sensor should be above this inflection point to obtain better response for reducing gas.

3.4. Responses of SnO₂ based thin films

3.4.1. The influence of printing times on the response of SnO_2 films

The thickness of the films was an important factor that influenced the sensor response. By utilizing the ink-jet printing method, the thickness of the films could be easily changed by varying the number of printing events. As shown in Fig. 8, the response to ethanol vapour at 375 °C was significantly increased by increasing the number of times the sample was printed. When the ink A was printed twice, the response to 100 ppm ethanol was 2.2. When the films were printed 3 or 4 times, the resulting film's response was 3.3 and 4.7, respectively. The response of the film increased to 6 when the ink was printed 5 times. These results could be explained by the porous structure of the SnO₂ thin films [26]. As mentioned above, the SnO₂ films prepared by the ink jet printing method were porous with a crystallite size of approximately 10 nm. Therefore, the chemisorbed oxygen not only occurred on the films surface, but it had also diffused into the films. Increasing the film's thickness can increase the amount of chemically active chemisorbed oxygen ions, which results in an increase of the films response to ethanol.

When NO₂ was injected into the measuring apparatus, the resistance of the SnO₂ films increased. The sensor response (*S*) to NO₂ was defined as $S = R_g/R_a$. Fig. 9 shows the response of SnO₂ thin films printed various times to NO₂ gas at 375 °C. The response of the film to NO₂ was significantly decreased by increasing the number of printing events. The response of the SnO₂ thin film printed twice to



Fig. 8. Response of pure SnO_2 thin films with various printing times to ethanol vapour at 375 $^\circ\text{C}.$

50 ppm of NO₂ was 20. When the films were prepared by printing ink A 3, 4 and 5 times, the response was decreased to 17.6, 14.8 and 13.7, respectively. The above results indicated that the sensor response was largely dependent on the film's thickness.

3.4.2. The influence of measuring temperature on the response of SnO_2 films

The response curves of SnO_2 films synthesised by 3 printing events to 50 ppm ethanol vapour at different temperatures are shown in Fig. 10. When the ethanol vapour was introduced, the resistance of the films decreased rapidly and reached a stable value after a short period of time. The response and recovery time was shorter at higher temperatures. When the temperature was above 350 °C, the film had a high response speed, and the 80% response



Fig. 9. Response of pure SnO₂ thin films with various printing times to NO₂ at 375 °C.



Fig. 10. Response curves of a SnO_2 film printed 3 times to 50 ppm ethanol vapour at different temperatures.

time was less than 1 s. The recovery was relatively slow, and 1 min was needed for the film's resistance to return to the original values.

Using the data shown in Fig. 10, the responses of the SnO₂ film at different temperatures were calculated, and the results are shown in Fig. 11. Initially, the responses of the SnO₂ film to 50 ppm of ethanol increased with measuring temperature and reached a maximum value of 7.2 at approximately 425 °C. The response decreased when the measuring temperature was above 425 °C. Many studies have shown that an optimal operation temperature (T_m) exists where the highest response can be obtained [27]. As shown in Fig. 10, the optimal operation temperature of the sample to ethanol vapour was approximately 425 °C. However, as shown in Fig. 12, the optimal operation temperature of the sample to 50 ppm H₂S was approximately 179 °C.

3.4.3. The influence of additives on the response of SnO_2 based films

When utilizing the ink-jet printing technique, three methods could potentially be used to prepare metal-oxide based gas-sensing films: printing the metal-oxide based gas-sensing materials ink, printing additive precursor ink onto the surface of the pure metaloxide gas-sensing film, and the simultaneous printing of the additive ink and gas-sensing materials ink. In this work, the second



Fig. 12. Response of a SnO_2 film printed 3 times to 50 ppm H_2S at different temperatures.

method was used to prepare SnO₂ based gas-sensing films. Fig. 13 showed the responses of one pure SnO₂ film and eight SnO₂ based films mixed with 1 mol% of several different additive materials to H₂ at 350 °C. When the films were prepared by mixing 1 mol% of a noble metal, such as Ag, Au and Pd, the response of the film to H₂ increased. However, the film's response to H₂ was decreased when the films was mixed with 1 mol% of Sb or Ni. The initial response of the SnO₂ film to 1475 ppm H₂ was 8.7 at 350 °C. The response increased to 20.1 when the film was mixed with 1 mol% Pd, and the response decreased to 3.3 when the film was mixed with 1 mol% Sb.

As shown in Fig. 14, the response of a SnO_2 film formed by 4 printing events to ethanol vapour increased when the film was mixed with Ag, Co, Cu or Zr. The sample mixed with 1 mol% of Cu had the highest response when the concentration of ethanol vapour was over 500 ppm. The initial response of the pure SnO_2 film to 1475 ppm ethanol vapour was 8.1 at 350 °C. The response increased to 13.2 when the film was mixed with 1 mol% of Cu, and the response decreased to 3.1 when the film was mixed with 1 mol% Ni. The additive's effect on the films response to ethanol was significantly less when the film was mixed with 1 mol% Au or Pd and the ethanol concentration was below 500 ppm.



Fig. 11. Response of a SnO_2 film printed 3 times to 50 ppm ethanol vapour at different temperatures.



Fig. 13. The response of SnO₂ based thin films printed four times to H₂ at 350 °C.



Fig. 14. The response of d SnO_2 based thin films printed four times to ethanol vapour at 350 $^\circ\text{C}.$

4. Conclusions

SnO₂ based gas-sensing films were successfully prepared using an ink-jet printing technique. An XRD analysis of the SnO₂ films annealed above 550 °C indicated the presence of a rutile phase. SEM images showed that the thickness of the SnO₂ films was less than 7 µm when the number of printing events was less than 8. SEM images also indicated that the films had a porous structure, which is a useful morphology for gas sensing. The electrical and gas sensing properties of the films changed significantly when the thickness of the films was varied. The film's thickness can be varied by altering the number of printing events. The SnO₂ films resistance decreased and its response to ethanol increased when the number of printing events was increased up until 5. Various elements, such as Au, Ag, Pd, Co, Ni, Cu, Sb and Zr, were deposited onto the SnO₂ films with a concentration of 1 mol% by printing inks containing these elements onto the SnO₂ film. Pd doping significantly improved the films response to H₂. However, Pd doping had a limited effect on the films response to ethanol vapours with a concentration below 500 ppm. Cu doping increased the films response to ethanol and decreased its response to H₂. Ni and Sb doping decreased the films response to both ethanol and H₂. These results indicate that the ink-jet printing technique is promising for the preparation of gas sensing material films, particularly when a number of films with different compositions are to be integrated on one substrate.

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Biography

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