

# Development Of Co Gas Sensing Based SnO<sub>2</sub> Thin Film

I DewaPutuHermida<sup>\*</sup>, Yuyu R. Tayubi<sup>\*\*</sup>, Rani Nopriyanti<sup>\*\*</sup>)

**Abstract--** This research, have done design and fabrication CO gas sensing based on SnO<sub>2</sub> thin film by *sol gel method*. For component of gas sensing such as electrode was made of gold by sputtering and heater was made of PdAg by screen printing. So that, sensor is expected to yields high sensitivity. Tests on sensors is conducted to determine the effect of temperature on the resistance of the sensor and the sensitivity of the sensor with a given flow rate of 10 ppm CO gas. The effect that occur in the sensor gas that when the temperature that given to sensor is enlarged then the sensitivity will increase as well, but when the temperature is given to sensor through its operational temperature sensor, the sensitivity of sensor value will decrease. It also conducted tests using SEM and EDS to determine the morphology and composition of the constituent sensor. Thus shown that the coating formed sensor has 21.21% C atoms, 22.43% O atoms, 14.98% Si atoms, 0.34% Cr atoms, 1.16% Ag atoms, 1.78% Sn atoms, and 38.11% Au atoms. Operational temperature owned by this sensor is 95° C with the highest sensitivity value of this sensor is 16.59.

**Index Term--** SnO<sub>2</sub>, CO gas sensing, sensitivity, sputtering.

## 1. INTRODUCTION

Carbon monoxide (CO) is a gas that is flammable, poisonous, colorless, and tasteless. Because of it is not colored and tasteless carbon monoxide is difficult to detect when without using detection technology [1]. Therefore, carbon monoxide is often dubbed the "silent killer", and needed a technology that can detect the presence of CO gas.

Science and technology of the semiconductor gas sensor was growing rapidly. Device semiconductors gas sensor are generally known as a metal oxide gas sensor because it is made of metal oxides such as TiO<sub>2</sub>, ZnO, CeO<sub>2</sub> and SnO<sub>2</sub> [2].

Among all the metal oxide, SnO<sub>2</sub> is one material that is good enough to be used as the active layer of CO gas sensors [3]. Nevertheless, SnO<sub>2</sub>-based gas sensor to date has not made the high sensitivity and selectivity. [4]. According to Pires, one of the factors that can improve the quality of a sensor that is optimizing the quality of the electrode. Electrodes used in a sensor must have high conductivity values. In order for the resulting electrode has a good quality, the electrodes can be fabricated using thin-film technology and made using materials that have good electrical conductivity [5].

Along with the development of microelectronics technology or nanotechnology at this time, electrode fabrication technology that is applied to metal oxide-based gas sensors can be done using the method sputtering. Using Sputtering technology of thin film growth becomes simpler and operational costs become cheaper. In addition, by using the method of Sputtering adhesives between the coating and the substrate surface becomes stronger, coating thickness easily observed and controlled [6].

The purpose of this research is to determine the characterization of SnO<sub>2</sub>-based CO gas sensors has been done in terms of the development of electrode components.

## 2. EXPERIMENTAL

### 2.1. Design of Gas Sensor

#### ✓ Parts of Sensor

Gas sensor is composed of electrodes that coated by a layer. And the layer is sensitive to the presence of CO gas sensors are printed on a substrate of silicon (Si). Heater sensor printed on the substrate surface alumina (Al<sub>2</sub>O<sub>3</sub>). Here's a specifications of gas sensor CO:

- Dimensions : ≤10 mmx25 mm
- Researching Power of Heater : 1.8 Watt
- Operating temperature : 25-250° C
- Measurement Range : 0 ~ 10ppm
- Voltage Sensor : 3 volt dc
- Heater Voltage : 3 volt dc

#### ✓ Designing of Eelectrode

The electrodes are commonly used in gas sensor components are generally an interdigital electrode structure (interdigital electrodes), which are usually made of materials such as Au or Pt noble metal. The use of the structure was based on the consideration to minimize space but maximize the sensing area. To calculate the value of the electrode resistance can be seen in the equation and the picture below:

$$R_{el} = \left( \frac{l_1}{w} + \frac{l_2}{w} + 0,56 \right) R_s \quad (1)$$

Where

$R_s$  is the value of sheet resistance ( $\Omega$  /sq).

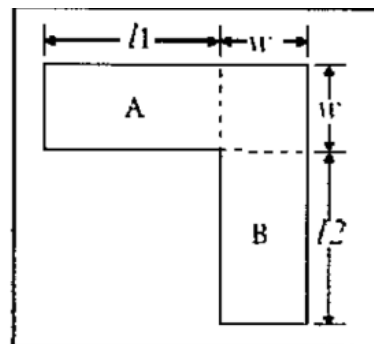


Fig. 1. Image Calculation of electrode resistance value.

The design layout of electrodes that used can be seen in the following fig.:

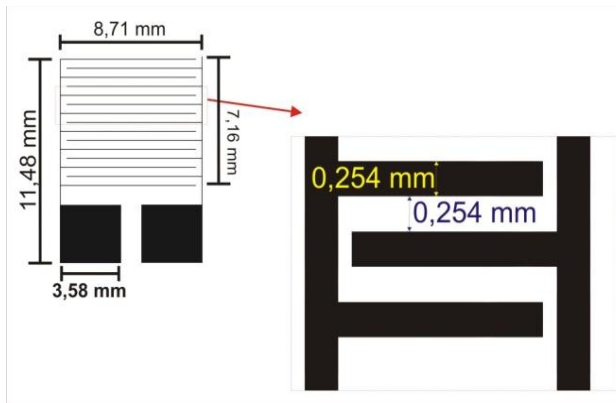


Fig. 2. Design of Electrode

✓ **Design of Heater**

One of the factors that determine the success of thin film gas sensors that are temperature factors that determine the success of thin film gas sensors that is temperature. Appropriate temperature distribution will affect the level of selectivity and sensitivity of this sensor element. To determine the characteristics of the heater, the parameters that must be considered are : the desired temperature, the power needed, and the area that you want to heat the area, as well as the character of the material heater itself (TCR, dissipation maximum current that can pass through, etc. ). Thus the characteristics of the sensor heater is designed for:

- Operating Temperature : 300°C
- Average power : 1.8 Watts
- Flow : 0.612 A

Based on the calculations that have been carried out, the heater has a length of line conductivity sensor of 94.107 mm and a track width of 0.529 mm. Then the resulting layout design heating gas sensor is as shown below:

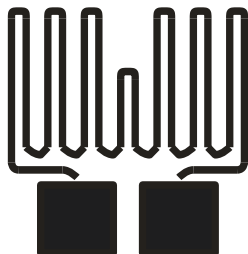


Fig. 3. Heater of Gas Sensor Layout Design

3. SENSOR FABRICATION PROCESSES

SnO<sub>2</sub> gas sensor fabrication process is divided into three phases; there are the electrodes fabrication, heater fabrication, and the active layer fabrication. Here is the exposure steps research that has been conducted by the author in several stages of research as follows:

3.1. Electrodes fabrication

The electrodes used in gas sensors, fabricated using sputtering method. Broadly speaking electrode fabrication steps illustrated in the diagram below:

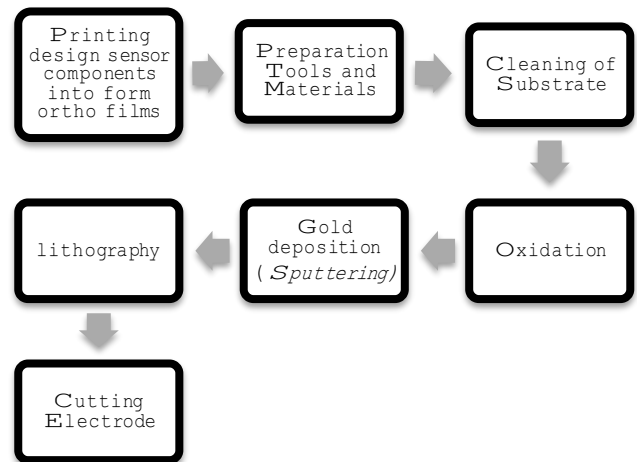


Fig. 4. Electrode Fabrication Process

The first steps in the fabrication of electrodes is to do the washing substrate using acetone, 5% HF solution, DI Water and H<sub>2</sub>O<sub>2</sub> solution: HCL:H<sub>2</sub>O. After that was done the process of oxidation. This research used dry oxidation process at a temperature of 1100°C for 130 minutes. Then later, the growth of Au thin films on silicon substrates using by DC Sputtering method and the pressure within the chamber to 5.9 x10<sup>-5</sup> Torr and argon gas flowed at a pressure of about 4mTorr ± 0.1 mTorr. Before coated with gold, the substrate was coated with chromium first. Chromium coating was done for 2 minutes, which is then immediately followed by a coat of gold for 10 minutes.

Then to form the electrode pattern on a silicon substrate performed lithography process consists of several stages.



Fig. 5. Lithography process.

In the prebake process substrates in an oven at a temperature of 85°C for 3 minutes in the oven. Immediately after completion oven substrate, the substrate is coated resistors ma-P215 positive resist using a spin coater. Then do the soft bake process carried out in an oven with a temperature of 150°C for 15 minutes.

Then do the process of exposure for 2 minutes in the sun and continued with the process of development by using MF319 developer solution until the pattern emerges and then wash with DI Water. Then, the substrate reheat in the oven for 5 minutes with a temperature of 85°C. Further etching process using a solution of I<sub>2</sub>:KI:H<sub>2</sub>O with a ratio of 1:4:40 until golden patterns emerge, then wash with DI Water and dry. And then remove a layer of the remaining positive resist using acetone.

And the last thing to do in making the cutting was electrode substrate. The process of cutting the substrates used in this study was done manually using diamond eyes.

### 3.2. Making Sensor Heater

Heaters were made using thick-film technology and screen printing methods. In the making, to produce heater must pass through several processes including washing the substrate, making the screen, thick film growth, drying and burning. The substrate used in the fabrication of heater was alumina substrate. Before use the substrate, it must be washed first by soaking in a glass beaker containing water dye, then put in the Ultrasonic cleaner for 5 minutes.

Furthermore, the first step that must be done in this process was the selection screen. In this study, used 325 mesh screen density and size 20 x 20 cm. After determining the screen to be used, the next thing to do is clean the screen by using Ulano 5.

Then after the screen is completely dry, CDF3 film paper that has been cut into a size of 10 x 10 cm is placed in the middle on the front surface of the screen. CDF 3 placed over the screen with the emulsion at the top, and a little duct tape glue on one side of CDF 3 that were not easily moved. Then on the back surface of the screen was coated Ulano 133, just behind the CDF layer 3. Remove the masking tape that attached to one side of CDF 3, then dry it with a hair dryer for 15 minutes.

After Ulano 133 completely dry, a layer of plastic / mylar on CDF 3 removed carefully. Furthermore, ortho-film paste on the upper right that has been released CDF 3 plastic coating. Then the screen was placed in the middle of the field of radiation on the radiation machine, the process was called by photography.

And the last one was on the screen that has not been covered by the CDF 3 coated again with a flattened Ulano 133 using rakel and dried using a hair dryer. After the pattern was formed on the screen, then performed the process of growing a thick film on alumina substrate using screen printing and pastes used the pasta PdAg (Dupont 7484) to the value of the sheet resistance 15-30 mΩ/□. Furthermore heater dried using an oven for 15 minutes at 195°C temperature used. After heater becoming dry, the heater should be firing in a high temperature furnace. The length of time firing for about 30 minutes.

After the heater is formed, then cut to the size of the alumina substrate heater is formed. Cutting substrate performed using diamond eyes.

### 3.3. Synthesis Procedures

First, we should prepare materials that will be us is powder SnCl, isopropanol, propanol and water. Comparison of the composition of the materials

to be used are powder SnO<sub>2</sub>: Isopropanol: Propanol: Water is 5: 7: 7: 6.



Fig. 6. SnCl powder

And then, puree SnO<sub>2</sub> powder using a mortar and Pestle for ± 1 hour without stopping. This is done so that the particle size of SnO<sub>2</sub> become nanoscale. Combine SnO<sub>2</sub> powder that has been mashed with 2/3 parts of propanol and 1/3 water, then stir for 1 hour while heated at a temperature of 80°C.

Burn the solution that has been made in the oven with a temperature of 300°C. After this stage, the solution will turn into powder. Combine remaining water, isopropanol and residual of propanol and powder. And then stir for 1 hour without heating, until resulting paste clear and homogeneous. Of the whole process can be generated with sol-gel, then will be used to make a sensitive layer.



Fig. 7. SnO<sub>2</sub> paste

Furthermore, to form a sensitive layer, SnO<sub>2</sub> paste which was formed dripped right over the electrode which then leveled using a squeegee. In order to coat the pasta in the electrode except for the contact, then before coated the SnO<sub>2</sub> contact parts should be covered by using solatip first. After the paste dries then the tape is opened.

## 4. RESULTS AND DISCUSSION

### 4.1. Micro Structure and Structure Analysis by SEM-EDS

To determine the composition of the sensitive layer formed tested using EDS (Energy Dispersive Spectroscopy) with the type JEOL JSM 6360 LA. Materials used are sensing material SnO<sub>2</sub> testing performed by EDS as follows:

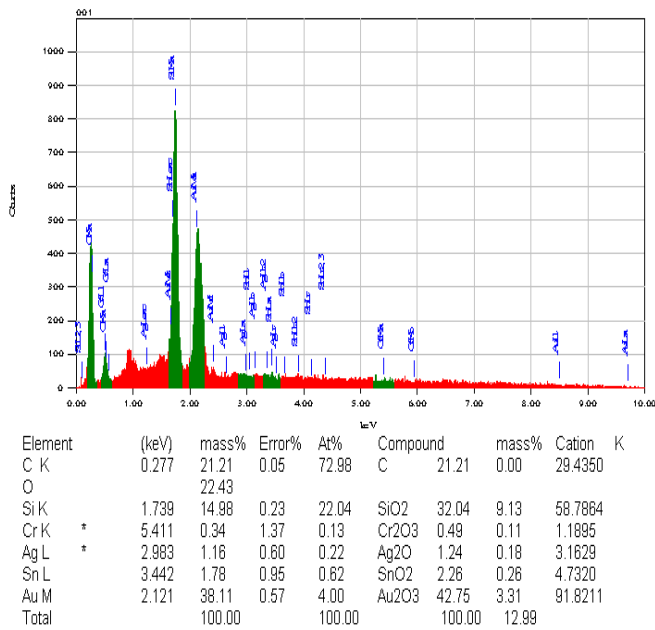


Fig. 8. EDS result gas sensor

Based on the fig. 12, it was observed that the elements contained in the SnO<sub>2</sub>-based CO gas sensors have the atomic composition of 21.21% C, 22.43% O atom, 14.98% Si, 0.34% Cr atoms, 1.16% atom Ag, 1.78% Sn atoms, and 38.11% Au atoms. The number of C atoms in the sensor was obtained when the combustion process and testing using CO gas sensor, while the O atoms derived from the absorption of free air.

While for the microstructure of the sensitive layer on a silicon substrate to deposition characterized using SEM. The results of the measurements in the form of SEM photographs shown in Fig. 8.

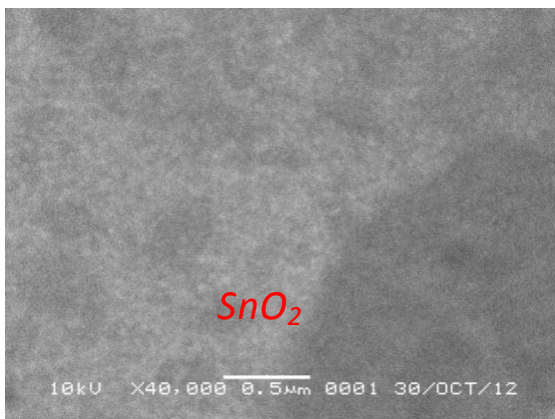


Fig. 9. Micro structure of SnO<sub>2</sub> Thin Films with a magnification of 40000X

From the SEM results above shows that the grain size of the SnO<sub>2</sub> thin film has reached the order of nano, but it can also be seen that the number of pores formed in the sensitive layer. This means, SnO<sub>2</sub> thin layer formed qualify as gas sensors. Because, the gas sensor formed pores that capture of CO gas, which will then lower the resistance value and increases sensor sensitivity.

Based on the fig. 11 above, could be seen the thickness of each material coated on a silicon substrate. Fig. 11 shows the micro structure of SnO<sub>2</sub> layer with a thickness of 0.118 μm, Au (gold) with a thickness of 0.379 μm, Cr (Chromium) with 0.508 μm thickness, SiO<sub>2</sub> (Silicon Dioxide) with 18.288 μm thick, while the Si (silicon) 39.03 μm.

4.2. Testing of Sensor

Test electrical characteristics initially conducted under 0 ppm CO gas in other words without the granting of CO gas. Testing was done by changing the value of current supplied to the heater, which then change the value of a given temperature gas sensors. Along with the change in the value of the test temperature, then the value will change the resistance.

Then, the test continued with the characteristics of the gas flow rate of 10 ppm CO gas. From the test results obtained can be seen in Fig. 11.

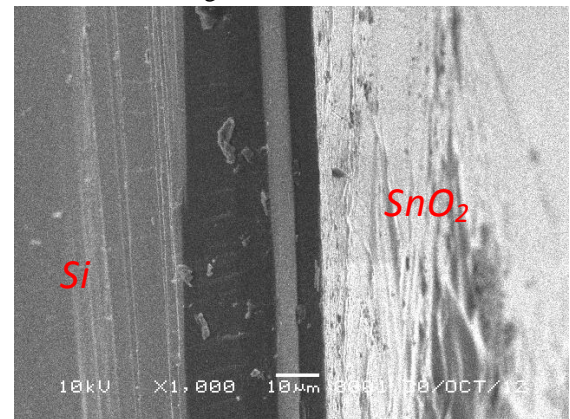


Fig. 10. Transverse cross section of SnO<sub>2</sub>-Based CO Gas Sensor

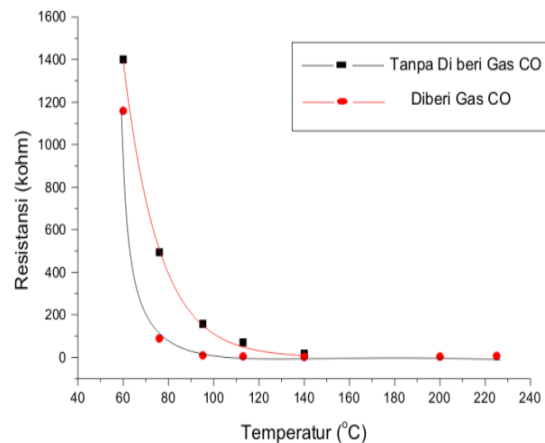


Fig. 11. Chart Changes of Resistance Against Temperature Operations Sensor When Given and Without Gas CO

Based on the graph above, it can be seen that the gas sensor resistance decreases exponentially with increasing operating temperature is given, either when given only when given gas CO or CO gas. This is in accordance with the following equation :

$$R = R_o \exp \left( \frac{E_a}{KT} \right)$$

Description:

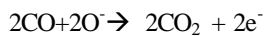
- R is the resistance at temperature measurement

- $R_0$  is the resistance at the initial temperature measurement
- $E_a$  is the activation energy.

But the decline in the value of the resistance that occurs when no CO gas were not as decrease the resistance value when given gas. According to Andrew et. al. in 1999, it was because the normal air condition and heated sensor, there will be a process adsorbs oxygen from the air around to the surface gas sensors through grain boundaries. The oxygen has been adsorbed on the surface of the gas sensor will attract electrons from the conduction band of an n-type semiconductor gas sensor to the surface, so that the oxygen - oxygen is ionized into ions - oxygen ions  $O^-$ .

Thus, it resulted in the potential barrier height (barrier between grains) will be high and hinder the movement of the electrons flowing through the grain boundaries. Then decrease the resistance value is not as big on testing for impairment testing resistance at the same time but fed CO gas.

When compared to the results of the resistance obtained when no CO gas with CO gas is given, can be seen at a temperature of 60-140°C impairment resistance occurs when CO gas were greater than without CO gas. This is due, when the CO gas flow into the heating chamber sensors remain to be done. In this situation, the reaction between CO gas with the gas sensor surface is:



However, given the testing conditions with gas sensor irregularities. Deviations can be seen when the temperature reaches 200°C and 225°C, the temperature increased resistivity values. According to Khalil et al In 2009, it was caused by the amount of carbon monoxide adsorbed gas molecules and accumulation of gas molecules occurs akibanya surface. Thus inhibiting the subsequent adsorption of carbon monoxide gas molecules.

From the test results can also be obtained by the value of the sensor sensitivity. From the data obtained a graph as shown in Fig. 12.

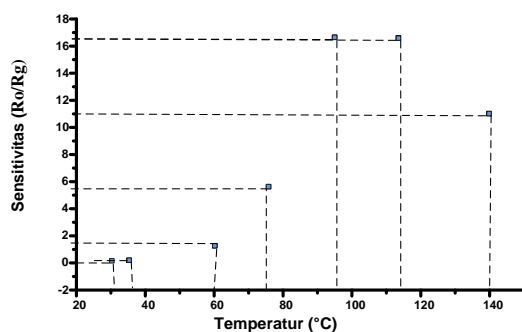


Fig. 12. Chart Of Change Sensitivity Sensor Against Operating Temperature

At temperatures of 60°C to 95°C sensitivity value seen a sharp increase in sensitivity due to the increasing value of the temperature. However, when the temperature is above 95°C decrease with the addition of temperature sensitivity.

## 5. CONCLUSION

From the results of testing that has been done, it can be concluded as follows :

1. By using thin-film technology electrodes, gas sensors able to detect the gas at a concentration of 10 ppm with a high value of sensitivity.
2. Gas sensor is able to react to CO gas, a change in the sensor resistance value down when given gas CO.
3. Changes in sensor sensitivity can be affected by temperature resulting heater.
4. The results of the EDS analysis of the elemental composition of the CO gas sensor has a composition of 21.21% C atom, atom O 22.43%, 14.98% Si, 0.34% Cr atom, atom 1.16% Ag, 1.78% Sn atoms, and 38.11% Au atom.
5. On further research, where to get a more sensitive sensor added doping Au/Pt on the surface of the sensor

## REFERENCE

- [1] Manjula P.S., Arunkumar, Sunkara V.M. (2010). *Au/SnO<sub>2</sub> an Excellent Material For Room Temperature Carbon Monoxide Sensing*. <http://elsevier.com/locate/snb>
- [2] Kuang Q., Lao C.S., Li Z., Xie Z., Zheng L., and Wang Z. (2008). *Enhancing The Photon and Gas Sensing Properties of a Single SnO<sub>2</sub> Nanowire Based Nanodevice by Nanoparticle Surface Functionalization*. Journal Physics Chemistry C, Vol. 112, No. 30, 2008.
- [3] Batzill M., Ulrike D. (2005). *The Surface and Material Science of Thin Oxide*. [http://www.surface.tulane.edu/pdf/surface science of T in Oxide.pdf](http://www.surface.tulane.edu/pdf/surface%20science%20of%20T%20in%20Oxide.pdf)
- [4] Hiskia, Hermida I.D.P., (2006). "Development of Carbon Monoxide Gas Sensor (CO) Based on SnO<sub>2</sub> Material". <http://elib.pdii.lipi.go.id/katalog/index.php/searchkatalog/downloadDataById/7039/7039.pdf>
- [5] Pires J.M.A. (2003). *Thin Film Gas Sensor*. PhD Dissertation for Universitas Minho, Portugal.
- [6] Yunas J., Lia M. (2001). *Sputtering System Applications for Thin Layer Deposition*. <http://elib.pdii.lipi.go.id/katalog/index.php/searchkatalog/downloadDataById/2676/2677.pdf>

## I Dewa Putu Hermida

Research Center for Electronic & Telecommunications – LIPI  
 Jl. Cisu No.21/154 D, Komplek LIPI Sangkuriang,  
 Phone: +62 22 250 4660, Fax: +62 22 250 4659; Bandung - 40135,  
 Indonesia  
 email:  
[idedwaputu@gmail.com](mailto:idedwaputu@gmail.com), [putu@ppet.lipi.go.id](mailto:putu@ppet.lipi.go.id)

## Yuyu R. Tayubi, Rani Nopriyanti

Department of Physics Indonesia University of Education  
 Jl. Dr. Setiabudhi No. 229 Bandung 40154 Jawa Barat Indonesia  
 Telp/Fax.(022)2001108, 2013163 Pes. 4632-4635