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ZnO Nanowires/Nanobelts Structured CO Gas Sensor

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Abstract: Zinc oxide nanowires/nanobelts thin films were prepared by thermal evaporation deposition of Zn on two different kinds of substrates, namely Cr-glass and Cr-<100> n-type Si. ZnO nanostructured based CO- gas sensing thin films were formed, and the morphologies of these films were investigated by scanning electron microscopy (SEM). The sensing response of the films toward CO gas at operating temperatures in the range of (150-200) ^{o}C were studied. The set-up designed to measure the gas sensing ability due to the change of the resistivity related to the gas addition, the electrical resistivity as well as the gas sensing properties of the ZnO nanowires/nanobelts films changed significantly with the operating temperatures. The sensitivity increased with increasing operating temperature, particularly above 175 ^{o}C where it increased by three folds. Typically ZnO nanowires/nanobelts thin film sensor had fast rise times (~4s) and (~24s) and good recovery times (~2s) and (~12s) for glass and silicon substrates, respectively.

Keywords: Nanowires ZnO; ZnO gas sensors; CO gas sensors; Nanobelts structures; Response time gas sensors.

Introduction

Zinc oxide is one of the transparent conducting materials, which has a direct wide band gap in the group II-IV semiconducting material and it has a large exciton binding energy of 60 meV[1]. ZnO is a promising functional material due to its high electrical and chemical stability, controllable resistivity, high visible transparency and non-toxicity[2]. The production of nanostructured thin films by means of physical vapor deposition (PVD) has been considered for many years. Low deposition rates can be achieved using an electron beam (e-beam) method, while high deposition rates can be achieved by thermal evaporation. Wide ranges of applications have been developed for nanostructured ZnO thin films as in solar cells [3], light emitting diodes (LED's) [4,5], touch screens [6], flat panel displays [7], harmonic generation for laser systems [8], gas and chemical sensing [9-12], or piezoelectric nano-generators [13].

Among the materials used for gas sensing purposes, ZnO has been highly considered because it has high sensitivity to toxic and combustible gases [14], and because it can be readily obtained in nanostructured form, as nanowires [15,16], nanosheets [17-19], nanoneedles [20], nanotubes [21], nanorods [22-24], or flower-like [25].
Several different methods have been used for synthesizing pure, doped and composite ZnO thin films as; magnetron sputtering [26-29], successive ionic layer adsorption and reaction (SILAR) [30], pulsed laser deposition (PLD) [31, 32], chemical vapor deposition (CVD) [33], chemical bath deposition (CBD) [34,35], physical vapor deposition (PVD) [36], spray pyrolysis [37], solvo-thermal method [38], hydro-thermal [39], sol-gel [40], cathodic vacuum arc deposition [41].

Most of gas sensors based on zinc oxide thin films have been fabricated for detecting toxic and hazardous gases such as H2, CO_2, CO, H_2S, and NO_2 [42-48]. Pure or doped ZnO or composites of ZnO with other materials have been used for detecting gases, in order to enhance the response and recovery towards both the oxidizing and the reducing analyte [48]. Furthermore, sensors based on ZnOnanobelts were found to be highly selective in their response to NO [13]. The mechanism of improving the response to H_2 based on annealing has been proposed and discussed [49]. CO sensing properties were investigated for the grown structures, and the response of comb-like structures was found to be 1.4 at 75 °C, while belts and mixtures of belts and rod like structures did not show any response [50]. The influence of Mn on trimethylamine (TMA) and ethanol sensing properties has been reported [51]. ZnOnanorod sensors with embedded Ag nanoparticles ZnOnanorod sensors have shown long-term stability and exhibited highly enhanced gas sensing performance in their response and selectivity for detecting ethanol vapor [24]. High sensitivity, fast recovery, and reliability have been achieved by Al doped ZnO prepared by RF magnetron sputtering [14]. The characterization of sensing properties for detecting NO_2 and CO gases by nanocomposite thin films such as CdO-ZnO, ZnO-SnO_2, and ZnO-TiO_2 have been studied [52, 53, 10].

The aim of this work is to produce a high-quality nanostructured ZnO thin films by thermal evaporation of zinc material on glass and silicon substrates, followed by annealing at an optimum temperature. Special attention paid to form needle-like and

sheet-like structures, as well as to examine these films concerning the CO gas sensing properties. Comparable the fast rise and recovery times (of a few seconds) of ZnO sensors have not previously been reported in literatures. In this work, the authors produced a fast, strong and repetitive response ZnO sensor for CO gas detection which can be fabricated at low coast.

Experimental

A. Synthesis of ZnO nano structures

Glass and n-type Si substrates were used. The substrates were cleaned by the following sequence; detergent and rinse under tap water; acetone, ethanol, distilled water; ultrasonic bath of acetone and ethanol for 15 min followed by the next cleaning step in distilled water; finally the substrates dried in the flow of hot air for 1 min, then the substrates were ready for deposition. Prior to the fabrication of ZnO needle-like films, chromium metal (Cr) was deposited by the electron beam (e-beam) method on the glass substrate as a buffer layer to improve the film adhesion. In the next step the Zn film was deposited on the Cr-glass substrates through thermal evaporation. The system (EDWARDS FL 400) was used for both methods. A molybdenum boat was used as the evaporation heating source, and the precursor was zinc granulate with a purity of 99.999%. The deposition parameters are listed in table (1):

Table 1: The Deposition Parameters

Deposition parameters	Data
Boat to substrate distance	12 cm
Weight of zinc in boat	0.2 g
Pressure through deposition	1 x 10^{-6} mbar
Deposition rate	0.5 Å/s

After the deposition process, the films were subjected to atmospheric thermal oxidation at 500 ^{o}C for 1 h, using a tube furnace. After the oxidation to achieve ZnO thin films, the finger Ag electrodes were deposited onto film surface by e-beam evaporation using a shadow mask as illustrated in Figure (1).

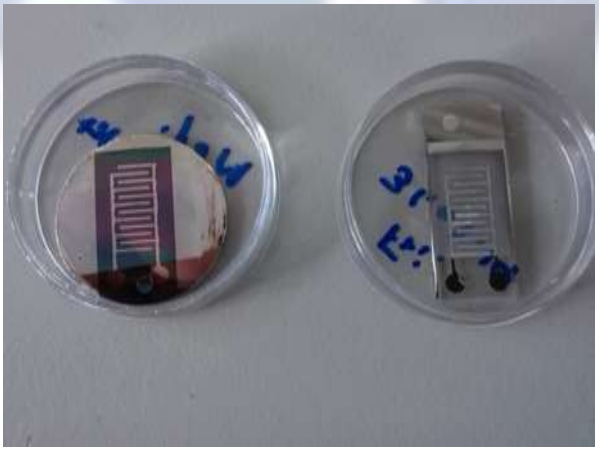

Figure 1.The finger electrodes.

B. Characterization and Measurements

The surface morphology of the films was studied by a (NOVA NANOSEM 230) scanning electron microscope (SEM) with field emission cathode. Gas sensing studies have been carried out using a stainless steel chamber with multiple inlets, outlets and feed throughs connected to the electrometer (KEITHLEY 4200-SCS). The chamber was evacuated by a rotary pump (EDWARDS), and solenoid valves served to switch the test gas on and off (Figure 2).

Figure 2.Sensing chamber.

Results and Discussions

A. SEM study

The analysis of the surface morphology of the prepared ZnO thin films was carried out by scanning electron microscopy (SEM). Micrographs of the ZnO thin films deposited on the glass substrate by thermal evaporation of Zn thin films and oxidized in the tube furnace at temperatures (550-650)^{o}C for (1:00) h show different types of nanostructures. Figure (3) shows the surface morphology of ZnO prepared and deposited on the glass substrate and oxidized at (550)^{o}C for 1 h. The figure clearly shows a dense distribution of nanowire/nanoneedles. Also nanosheets have been found.

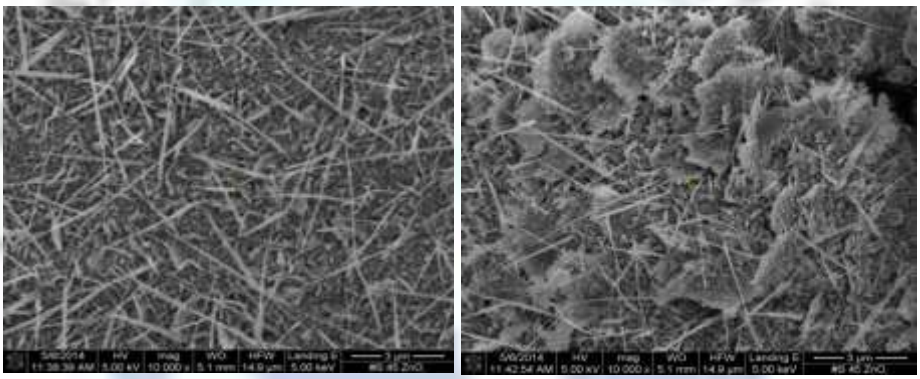

Figure 3.SEM images of ZnO oxidized at 550 ^{o}C; (a) needle-like and (b)sheet-like deposits on the glass substrate.

Nanostructures in the form of ultra-thin ribbons as shown in Figure (4) have grown on the Si substrate. Clearly, the structures that have formed are characterized by a high aspect ratio. Rising the oxidation temperature to 650^{o}C caused to form the rose-leaf like structures as shown in Figure (5). The particles have the morphology of ultrathin circular sheets (disks), with the diameter of the disks amounting to about a few micrometers. There was no significant change in crystallinity when the annealing temperature was increased from room temperature to 300 ^{o}C. As the temperature was increased to above 400^{o}C, the domain boundaries (or micro-cracks) became more distinct [26]. Oxidation in air provides higher deformation of the surface of the zinc thin film in presence of oxygen molecules. Furthermore, at high temperatures the nanostructures grow homogeneously on the surface and these nanostructures intersect each other in some locations to form a new type of structure as rose-sheet like structures at 650^{o}C (Figure 5), as also reported in [27].

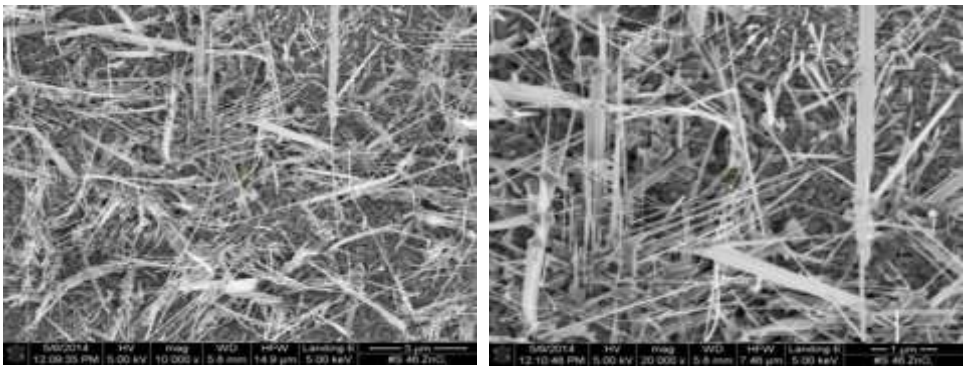

Figure 4. SEM images of ZnO oxidized at 550 ^{o}C; (a) needle-like and (b)sheet-like deposits on the silicon substrate.

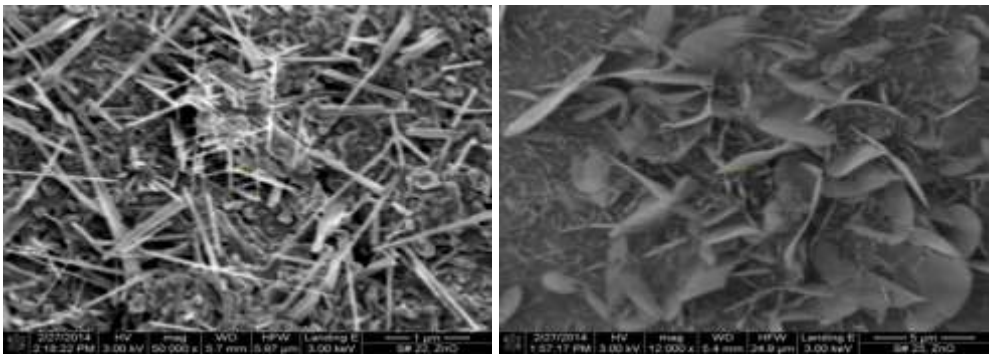

Figure 5. sheets-like structures.

B. Sensing characterization

Gas sensing properties of ZnO nanowires/nanobelts on both glass and silicon substrates were investigated. The sensing resistivity to CO gas was firstly investigated at room temperature and the results show no response for the film deposited on the glass substrate, whereas that on the silicon substrate indicated a strong response signal with short rise times as shown in Figure (6).

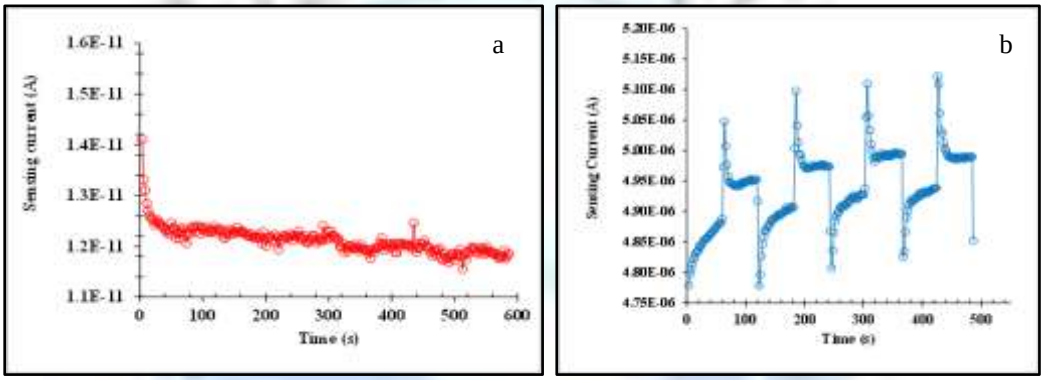

Figure 6. Room temperature response of the ZnO thin film nanostructures to CO gas at room temperature.

The films were deposited on: a) glass substrate, and b) silicon substrate.

The analysis of the sensing behavior of ZnO nanostructured thin films with respect to CO gas at various operating temperatures were carried out by measuring the electrical resistivity change with respect to the resistivity of the films in air. The change in the electrical resistivity of the films as a function of time during the exposure to a partial pressure of CO gas and venting was observed. For a n-type ZnO, a reduction interaction with the CO gas has occurred. The dynamic response of the ZnO nanowires deposited on the glass substrate in the temperature range of (150-200) ^{o}C to CO gas (with the gas flow switched on and off for 1 min. respectively) are shown in figure (7). The sensing current increased instantaneously to a maximum value, which was maintained at the maximum current upon exposure to CO and recovered completely to the initial value upon the removal of CO. The sensor shows high stability during repeated test cycles. The significant enhancement in the response of the ZnO nanowires to CO gas by increasing the temperature was attributed to the decrease of the contact resistance between the grains.

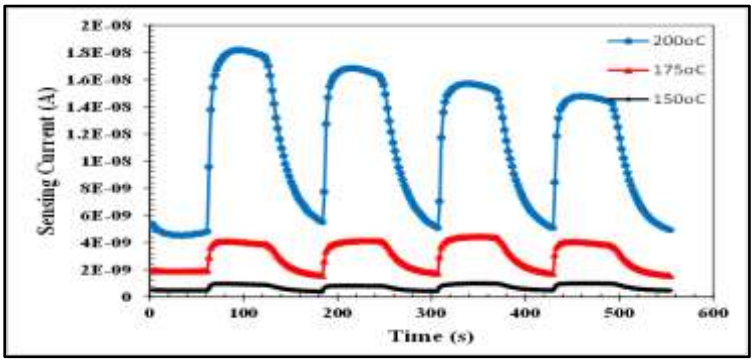

Figure 7. CO gas sensing by ZnO nanowires deposited on the glass substrate.

The sensitivity and typical response of ZnO nanowires and nanobelts to CO gas measured at different temperatures are shown in figures 8 and 9. Here, the sensitivity (S) is defined as:

$$S = \frac{I_g - I_a}{I_a} x100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Were I_g is the current after exposing to gas and I_a is the current in air.

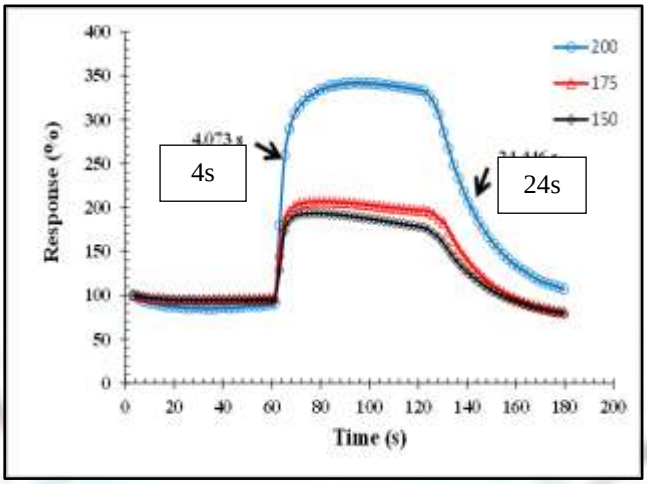

Figure 8. Typical response of ZnO nanostructure gas sensor to CO gas for different temperatures: 150, 175 and 200 ^{o}C.

Figure 8 shows the typical response of the ZnO nanowires and nanobelts to CO gas at three different temperatures 150, 175 and 200 ^{o}C, the fast rise times (~ 4 s) and recovery times (~ 24 s) indicate the strong response of the films to CO gas, which is a prerequisite for using the films as sensors.

The sensitivity of ZnO nanowires in the presence of reducing CO gas was defined as in relation (1), the influence of the operating temperature on the sensitivity of the films is plotted in Figure 9. It was clearly observed that the sensitivity of the films increased three times within the range of operating temperatures of (150-200) ^{o}C.

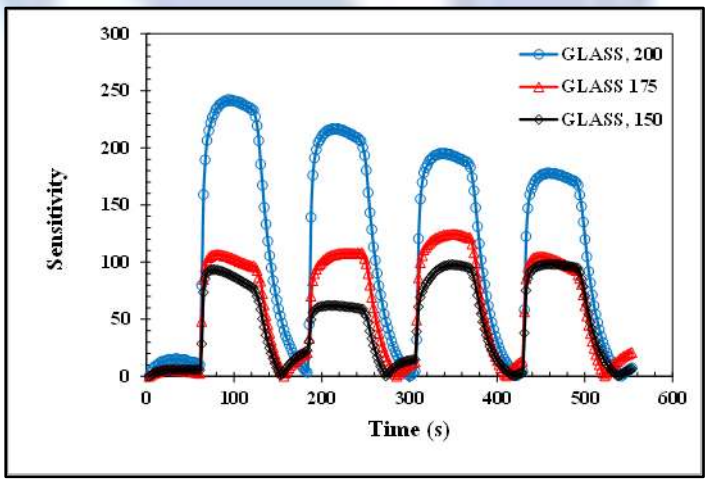

Figure 9. The sensitivity of ZnO nanowires towards CO detection deposited on the glass substrate for various operating temperatures (150, 175 and 200) ^{o}C.

Figure 10 shows the sensing current of the ZnO nanowires deposited on the <100> n-type silicon substrates at operating temperatures in the range of (150-200) ^{o}C.as indicated in Fig. 9, fast, highly response and excellent recovery have been recorded for this sensor. Also, the sensor has very good repetitive characteristics and quick release cycles could also be obtained.

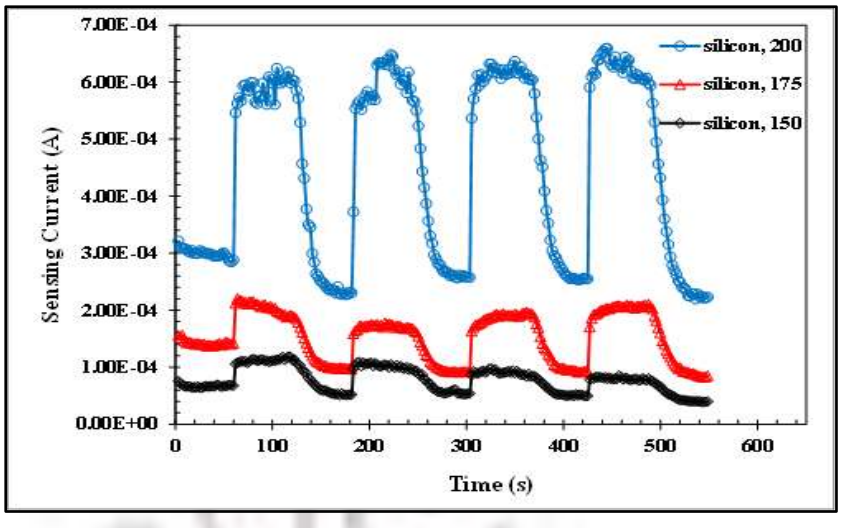

Figure 10. The sensing current of the ZnO thin film deposited on the silicon substrate.

The response time behavior as well as recovery time at different operating temperatures was determined from the I(t)-behavior. The ZnO nanowires deposited on the silicon substrate had a response of (~ 190%) when operated at 200°C. The rise time and recovery time were (~2s) and (~12s), respectively. Figure (11) shows the rise time and recovery time of the ZnO sensor deposited on the silicon substrate.

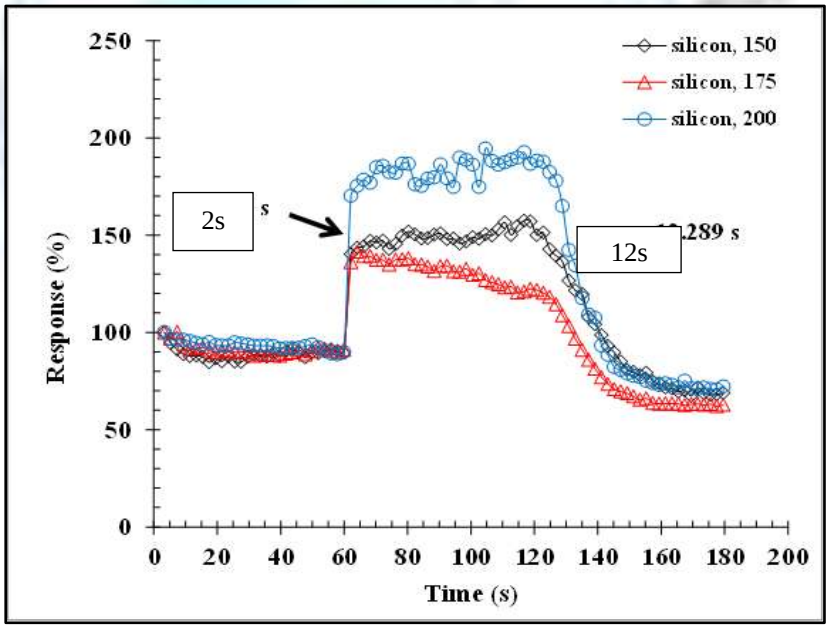

Figure 11. The response of the ZnO thin film deposited on the silicon substrate.

The reason for the fast rise time is the fast saturation of the change of the sensing current which can be attributed to the small surface area of the sensor (8 mm x 15 mm). However, the low recovery time can be attributed to the optimum operating temperature and the ability for desorption on the sensor surface area.

The sensitivity of the ZnO thin film deposited on the silicon substrate increases with the operating temperature as shown in Figure 12. Figure 12 also indicates a higher stability of the sensor at 200°C than at the other testing temperatures (175 and 150) °C.

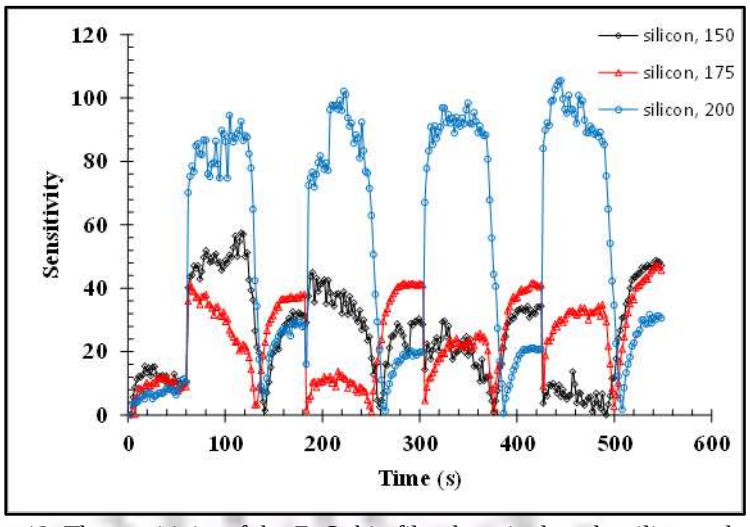

Figure 12. The sensitivity of the ZnO thin film deposited on the silicon substrate.

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Conclusion/Results

To conclude, ZnO thin films have been synthesized and deposited on both glass and silicon substrates. Nanowires, nanorods and nanosheets are grown by optimizing the deposition parameters of the PVD system. The present work discusses and compares the sensing characteristic of ZnO nanostructured films as CO sensors. Both kinds of deposited films are characterized concerning their microstructure and concerning their sensing behavior towards CO at different operating temperatures 150, 175 and 200 ^{o}C. The sensor response and recovery time of both deposited films on glass and silicon substrates is found to increase with the temperature. However, considering the response and recovery time characteristics, ZnO nanowire sensors deposited on the silicon substrate at an operating temperature of 200 ^{o}C shows the best sensing performance.

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