CHAPTER – 6

Response of ITO Thin Film Sensors to

1 Carbon Tetrachloride (Ccl$_4$)

2 Hydrogen Peroxide (H$_2$O$_2$)
<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>CONTENTS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>6.1</td>
</tr>
<tr>
<td></td>
<td>CARBON TETRACHLORIDE</td>
</tr>
<tr>
<td></td>
<td>6.1.1</td>
</tr>
<tr>
<td></td>
<td>INTRODUCTION</td>
</tr>
<tr>
<td></td>
<td>6.1.1 (i) Need and Importance of Detection of CCL₄</td>
</tr>
<tr>
<td></td>
<td>(a) Applications</td>
</tr>
<tr>
<td></td>
<td>(b) Hazardous Effects</td>
</tr>
<tr>
<td></td>
<td>(c) Exposure Limits</td>
</tr>
<tr>
<td></td>
<td>6.1.1 (ii) Detection</td>
</tr>
<tr>
<td></td>
<td>(a) Physical, Chemical Properties</td>
</tr>
<tr>
<td></td>
<td>(b) Review of Literature on the Detection of CCL₄ reported by Researchers</td>
</tr>
<tr>
<td></td>
<td>(c) Performance Criteria required</td>
</tr>
<tr>
<td></td>
<td>6.1.2</td>
</tr>
<tr>
<td></td>
<td>EXPERIMENTAL METHODOLOGY</td>
</tr>
<tr>
<td></td>
<td>6.1.2 (i) Fabrication of the ITO thin film Sensor</td>
</tr>
<tr>
<td></td>
<td>6.1.2 (ii) Detection and Measurements</td>
</tr>
<tr>
<td></td>
<td>6.1.3</td>
</tr>
<tr>
<td></td>
<td>RESULTS AND DISCUSSION</td>
</tr>
<tr>
<td></td>
<td>6.1.3 (i) Variation of electrical resistance of ITO thin film sensor with temperature in the presence of carbon tetrachloride vapours</td>
</tr>
</tbody>
</table>
### Chapter 6

| 6.1.3 (ii) | Response of ITO sensors having different thickness of film with concentration of carbon tetrachloride vapours |
| 6.1.3 (iii) | The effect of catalytic layer on the ITO thin film surface to detection of carbon tetrachloride vapours |
| 6.1.3 (iv) | Response time of carbon tetrachloride sensor |

### 6.2 HYDROGEN PEROXIDE

#### 6.2.1 INTRODUCTION

- **6.2.1 (i) Need and Importance of Detection of H$_2$O$_2$**
  - (a) Applications
  - (b) Hazardous Effects
  - (c) Exposure Limits

- **6.2.1 (ii) Detection**
  - (a) Physical, Chemical Properties
  - (b) Review of Literature on the Detection of H$_2$O$_2$ reported by Researchers
  - (c) Performance Criteria to be met

### 6.2.2 EXPERIMENTAL METHODOLOGY

- **6.2.2 (i) Fabrication of ITO sensor and Measurements**

### 6.2.3 RESULTS AND DISCUSSION

- **6.2.3 (i) Variation of electrical resistance of ITO thin film sensor with temperature in the presence of**
| **6.2.3 (ii)** | Response of ITO sensors having different thickness of film with concentration of carbon tetrachloride vapours |
| **6.2.3 (iii)** | The effect of catalytic layer on the ITO thin film surface to detection of carbon tetrachloride vapours. |
| **6.2.3 (iv)** | Response time of carbon tetrachloride sensor. |

**References:**
- Carbon Tetrachloride
- Hydrogen Peroxide
6.1 CARBON TETRACHLORIDE

6.1.1. INTRODUCTION

6.1.1 (i) Need and importance of detection of carbon tetrachloride

Carbon Tetrachloride is an important solvent having wide range of applications; but at the same time, it is highly toxic. Hence a portable sensor to detect CCl₄ is a need of the day.

Carbon tetrachloride, also known by many other names, the most notable being *Carbon Tet* in the cleaning industry. *Halon* or *Freon* is the organic compound with the formula CCl₄. Both carbon tetrachloride and tetra chloromethane are acceptable names under IUPAC nomenclature. It is a colourless liquid with a "sweet" smell that can be detected at low levels [1].

Carbon tetrachloride has practically no flammability at lower temperatures. Under high temperatures in air, it forms poisonous phosgene [2].

(a) Applications or Uses of CCl₄ [2]:
Used in Fire Extinguishers; in Spot Removers, Cleaning Fluid, and Insecticide.

As an Agricultural Fumigant and as a solvent in the production of Semiconductors, in the processing of Fats, Oils and Rubber and in Laboratory Applications (Lewis, 1993; Kauppinen et al., 1998).

Applying Paints to Automobiles & other Metal Surfaces

Degreasing

Extraction of Oils or other Food products

Textile Cleaning

Wool Processing

Preparation of Inks, Paints & Varnishes

Production of Pharmaceuticals

Cleaning of Electronics, Semiconductors & Precision Instruments

Cleaning of Aircraft & other Military hardware

Minor uses in Veterinary & Medical Applications.

(b) Hazardous/Harmful effects [1]
Carbon Tetrachloride can affect when inhaled and by passing through the skin.

- Enters the body through the respiratory organs and the skin, inducing a narcotic effect on the central nervous system, a slightly irritating effect on the skin, and a toxic effect on the liver, kidneys, and other organs.
- Carbon Tetrachloride can be irritated and burn the skin and eyes with possible loss of vision.
- Acute poisoning is accompanied by headache, vertigo, weakness, nausea, and vomiting.
- Can damage the liver and kidneys.

(c) **Exposure Limits:**

- Occupational Safety and Health (OSHA) The legal airborne permissible exposure limit (PEL) is 10 ppm averaged over an 8-hour workshift; 25 ppm, not to be exceeded during any 15-minute work period; and 200 ppm as a 5-minute maximum peak in any 4-hour work period.
- National Institute for Occupational Safety and Health (NIOSH): the recommended airborne exposure limit (REL)
is 2 ppm, which should not be exceeded for any 60-minute period.

American Conference Government Industrial Hygienists (ACGIH): The threshold limit value (TLV) is 5 ppm averaged over an 8-hour work shift and 10 ppm as a STEL (Short-Term Exposure Limit) [3-4].

6.1.1 (ii) DETECTION

(a) Physical, Chemical Properties

In the carbon tetrachloride molecule, four chlorine atoms are positioned symmetrically as corners in a tetrahedral configuration joined to a central carbon atom by single covalent bonds because of CCl$_4$ is non-polar & symmetrical geometry [9] shown in figure 6.1.1 (i)

Fig. 6.1.1. (i) Structure of Carbon Tetrachloride [1]
Table 6.1.1 (ii) shows the Chemical and Physical Properties of CCl₄.

Table 6.1.1 (ii) Chemical and Physical Properties of CCl₄ [9]

<table>
<thead>
<tr>
<th>CHEMICAL &amp; PHYSICAL PROPERTIES OF CARBON TETRA CHLORIDE</th>
</tr>
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<tbody>
<tr>
<td>Molecular Formula</td>
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<td>Molar Mass</td>
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<td>Melting Point</td>
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<td>Solubility in Water</td>
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<td>Solubility</td>
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<tr>
<td>Explosive Limits</td>
</tr>
</tbody>
</table>
(b) Review of literature on the detection of CCl₄ reported by researchers

Various scientists have reported the detection of CCl₄. Most of them have used thick film sensors based on Tin Oxide material [5]. Patel et al [6], i.e. our research group developed ITO based sensor and this work is a further extension. There are reports of sophisticated SAW devices for the sensitive, down well and in off-gas stream detection of VOCs, in particular CCl₄ at the Hanford site. However these are not cheap and the fabrication technology is not friendly for the mass production [7-9]. Van der Avert et al [10] have reported a laboratory test on VOCs, with a larger portion of the tests was performed with CCl₄ as the only VOC present in the sludge. Adil Meziane et al [11] discussed the transitions of carbon tetrachloride in confined geometry because CCl₄ is an effective solvent for polymer swelling and also presents this solid state phase transition. Thomas K.G. Mohr et al [12] have reported determining the right mix of additives that will successfully stabilize a chlorinated solvent for a particular application was the subject
of intensive research by industrial chemists working for the major solvent producers.

Wonyong Choi et al [13] suggested that the initial electron-transfer process may involve both one- and two-electron transfer reactions. The one- and two-electron steps result in the formation of trichloromethyl radical and dichlorocarbene as intermediates respectively. Both pathways result in the release of chloride ions (dissociative electron capture). Shukla et al [14] published data of sensor arrays fabricated and characterized, and demonstrated excellent gas/odour identification of our gas sensor system to identify individual gas/odour.

(c) Performance criteria required

The present work proposes the development of a thin film ITO sensor for the detection of carbon tetrachloride with faster response enhanced sensitivity and selectivity. It is a proven fact that the ITO thin films show better stability.

6.1.2 Experimental Methodology

6.1.2 (i) Fabrication of ITO Carbon Tetrachloride sensor
The response of ITO thin film semiconductor sensors to different concentration of CCl₄ gas was observed. The experimental set up shown in fig. 6.1.2 (i) Sensitivity and selectivity thin film CCl₄ sensor is demand of the day.

Fig. 6.1.2 (i) Measurement chamber
6.1.3 Results and Discussion

6.1.3 (i) Variation of electrical resistance of ITO sensor with change in operating temperature upon exposure to Carbon Tetrachloride (CCl₄) vapours

Figure 6.1.3 (ii) shows the variation in the electrical resistance of ITO semiconductor thin film gas sensor R_gas (R_g) with operating temperature in the presence of Carbon Tetrachloride vapours. The CCl₄ concentration was kept 167 ppm.

![Graph showing variation of electrical resistance of ITO sensor with temperature](image)

Fig. 6.1.3 (i) Variation of electrical resistance of ITO sensor with temperature
The background resistance of the sensor $R_a$ in the absence of the test gas was found to be almost constant with temperature within the range of ±4%. It was also observed that the film showed excellent stability against temperature and time variations and hence it is a better substitute to tin oxide based gas sensors shown in figure 6.1.3 (iii).
Fig. 6.1.3 (iii) Variation of electrical resistance with temperature for ITO sample when exposed to 167 ppm of CCL\textsubscript{4}.

Fig. 6.1.3 (iii) shows the variation of the resistance of ITO semiconductor thin film gas sensor $R_a$ with operating temperature.
Fig. 6.1.3 (iv) The working mechanism of carbon tetrachloride semiconductor gas sensors:

Fig. 6.1.3 (iv) indicates the sensing mechanism of ITO semiconductor thin film carbon tetrachloride gas sensor. It shows the conductivity of oxide semiconductor materials changes according as the gas concentration changes. This is caused by adsorption/desorption of oxygen (O₂) and reaction
between oxide surface oxygen ($O_2$) and gases. These reactions cause a dynamic change of electric potential of ITO polycrystalline and results in the decrease of the rest of the semiconductor sensor resistance under the presence of reducing gases of $CCl_4$.

6.1.3 (v) The Output Response of the ITO Thin Film Semiconductor sensor to $CCl_4$ with Operating Temperature

![Graph showing the variation in the relative change in resistance of ITO sensor with operating temperature when exposed to 83 ppm of $CCl_4$.](image)

**Fig.6.1.3 (v)** Variation in the relative change in resistance of ITO sensor with operating temperature when exposed to 83 ppm of $CCl_4$.

Figure 6.1.2 (v) shows the variation in the percentage relative change in resistance of sensors $(Ra-Rg)/Rg$ to $CCl_4$.
vapours with operating temperature. The response shows peak at 530 K, the optimum temperature.

6.1.3 (vi) The output response of the ITO thin film semiconductor sensors having different thickness to varied concentration of CCl₄

Fig. 6.1.3 (vi) shows the output response of the ITO thin film semiconductor sensors having two different thicknesses to varied concentration of CCL₄. The concentration was varied in the range from 167 and 1000 ppm. As shown in the figure 6.1.3 (vi) the sensor shows almost a linear response with change in concentration. Also the sensors with lower thickness show more sensitivity than the sensors with higher thickness. The reason for this is in Polycrystalline thin films and at sufficiently High Temperatures, a peripherial diffusion of gases at grain boundaries can generate inhomogeneous depletion layers. As discussed in previous chapter that thinner the film, more deep is the depletion layer and hence more scope to adsorb more number of gas species on the surface. The conduction channel can disappear by the chemisorption in the presence of the atmospheric oxygen. A subsequent
oxidation of the reducing gas with the chemisorbed oxygen species $O_2^-$ and $O^-$ can form again the conduction channel and so the resistance decreases in the presence of the reducing gas [5].

![Graph showing relative change in resistance](image)

**Fig. 6.1.3 (vi) Variation in the relative change in resistance $(Ra-Rg)/Rg$ of the sensor with different concentration**

In order to enhance the output response and the sensitivity of semiconductor thin film sensor, the films were coated with promoter layer. Thin films of Cu having thickness of about 10 nm were deposited at room temperature as a promoting layer on the film. Cu causes change in the
electrical properties of the semiconductor thin film enhancing the resistances. As the temperature increases and reaches around 543 K, the sudden decreases in the resistance are observed. The oxidation process of carbon tetrachloride is much enhanced and faster at 543 K on the surface of an ITO thin film following the redox reactions

![Graph showing variation in relative change in resistance with temperature for Cu/ITO films when exposed to 83 ppm and 1000 ppm of CCl₄.]

**Fig.6.1.3 (vii)** Variation in the relative change in resistance with temperature for Cu/ITO films when exposed to 83 ppm and 1000 ppm of CCl₄.
6.1.3 (ix) Response time of carbon tetrachloride ITO

![Graph showing response time of carbon tetrachloride ITO](image)

**Figure 6.1.3 (viii) Response Time of the ITO thin film to carbon tetrachloride vapours**

Figure 6.1.2 (viii) shows the measurement of the output voltage of the ITO sensor with Cu catalytic layer with respect to time. The rise time i.e. the time required for the output to go from 10% to 90% of its maximum value was found out to be 37 sec where as the fall time was found out to be 10 sec. It can be shown that the sensor needs less time to recover back to its original value.
6.2 HYDROGEN PEROXIDE

6.2.1. INTRODUCTION

6.2.1 (i) Need and importance of detection of Hydrogen Peroxide

Hydrogen peroxide is a colourless liquid with a slightly sharp odour, which is unstable and flammable under heating, friction or when contaminated [1]. Detection of hidden explosives is increasingly attracting more interest because of the global increase in terrorism. Highly selective and sensitive sensing techniques are required in order to detect trace explosives hidden on a person’s body or in baggage to provide security at airports, air travel and access control for sensitive infrastructures [1].

(a) Applications

The semiconductor gas/vapour sensors have found applications in environmental pollution, to improve the diagnostics for patient care in medical, bio-medical, and defence or space applications, to provide small, low power, fast, and sensitive tools for process and quality control in
industrial applications, and to implement or improve
detection of warfare threats and security by detecting the
explosives [2-3]. Hydrogen Peroxide is a solvent used in
many chemical processes however, recently, it is under
test to check for its use in explosives.

(a) Hazardous and Harmful effects [4]

Hydrogen peroxide is an aggressive oxidizer and will
corrode many materials, including human skin. In the
presence of a reducing agent, high concentrations
of H₂O₂ will react violently. Toxic via ingestion, inhalation,
skin or eye contact, harmful and corrosive, ingestion may
cause irritation of the gastrointestinal tract with vomiting
which may contain blood, gastrointestinal bloating,
tiredness and coma inhalation of hydrogen peroxide may
cause irritation of the nose, throat and respiratory tract. In
severe cases an accumulation of fluid in the lungs may
occur skin contact can cause bleaching of skin, and at
higher concentrations redness and swelling, blistering and
burns exposure to the eyes may result in pain, running of
the eyes, conjunctivitis, and sensitivity to light [1].
(c) Exposure Limits

The American Conference of Governmental Industrial Hygienists (ACGIH) states a TLV–time weighted average (TWA) of 1 ppm (1.4 mg/m$^3$) (ACGIH, 2008). This value is based mainly on results from mice inhalation studies suggesting that 1 ppm would be sufficient for protection against irritation Gagnaire et al [5]. The German Research Foundation has proposed a maximum allowable concentration (MAK) value of 0.5 ppm (0.74 mg m$^{-3}$) (DFG, 2008) and in UK, the value suggested by the Health and Safety Executive was 1 ppm for a TWA 8-h exposure and 2 ppm as a short-term exposure level (STEL) (HSE, 2005) Cr/IT. The database for the values cited above is, however, rather meager and some data indicate that these threshold values do not provide optimal protection for the workers. Rough skin on the hands and decolorized hairs were observed in workers exposed to H$_2$O$_2$ concentrations 1 ppm (Suenaka et al., 1984).
6.2.1 (ii) DETECTION

(a) Physical, Chemical Properties

![Structure of Hydrogen peroxide](image)

Fig. 6.2.1. (i) Structure of Hydrogen peroxide [4]

Fig. 6.2.1 (i) shows the structure and Table 6.1.1 (ii) shows the Chemical and Physical Properties of H₂O₂.

Table 6.2.1 (ii) Chemical and Physical Properties of H₂O₂.

<table>
<thead>
<tr>
<th>Properties</th>
<th></th>
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</thead>
<tbody>
<tr>
<td>Molecular formula</td>
<td>H₂O₂</td>
</tr>
<tr>
<td>Appearance</td>
<td>Very light blue colour; colourless in solution</td>
</tr>
<tr>
<td>Door</td>
<td>slightly sharp</td>
</tr>
<tr>
<td>Density</td>
<td>1.135 g/cm³ (20 °C, 30-percent)</td>
</tr>
<tr>
<td></td>
<td>1.450 g/cm³ (20 °C, pure)</td>
</tr>
<tr>
<td>Melting point</td>
<td>−0.43 °C; 31.23 °F; 272.72 K</td>
</tr>
<tr>
<td>Boiling point</td>
<td>150.2 °C; 302.4 °F; 423.3 K</td>
</tr>
<tr>
<td>Solubility in water</td>
<td>Miscible</td>
</tr>
</tbody>
</table>
(b) Review of literature on the detection of hydrogen peroxide reported by researchers

Recently, Yang et al. reported various synthetic methods for preparing efficient $\text{WO}_3$ sensing elements for high temperature potentiometric $\text{NO}_x$ sensors. Materials and methods include deposition on Yttria-stabilized zirconia (YSZ) attached to two Pt and Pd wires (Sensor A), $\text{WO}_3$ mixed with a-terpineol (Sensor B), a hydrogen peroxide/$\text{WO}_3$ solution (sensor C), and $\text{WO}_3$ deposition on YSZ followed by UV radiation and ozone treatment (Sensor 4). The experimental results showed that the Pt electrode (Sensor A) had the lowest $\text{NO}_x$ signal compared to the other devices containing $\text{WO}_3$ whereas, the $\text{WO}_3$/YSZ sensing electrode fabricated by the UV-ozone treatment method (sensor 4) had better mechanical stability, higher sensitivity, and better response/recovery.

<table>
<thead>
<tr>
<th>Solubility</th>
<th>soluble in ether, alcohol insoluble in petroleum ether</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity</td>
<td>1.245 cP (20 °C)</td>
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</table>
times than devices fabricated from commercial WO$_3$ powder [8].

Sofian M. Kanan et al, reviewed on some papers published in the last fifty years and focused on semiconductor metal oxide (SMO) based sensors for selective and sensitive detection of various environmental pollutants [9]. Advances in fabrication methods have enabled the production of low-cost sensors with improved sensitivity and reliability compared to those formed using previous methods [10].

(C) Performance criteria to be met

Hydrogen Peroxide is an oxidizing agent for organic and inorganic chemical processing as well as semiconductor applications, bleach for textiles and pulp, and a treatment for municipal and industrial waste defence applications.

6.2.2 Experimental Methodology

6.2.2 (i) Fabrication of ITO hydrogen peroxide sensor

The sensor was fabricated in the same manner as described in Chapter 3.
Fig. 6.2.2 Measurement chamber

The relative changes in resistance of sensors to different concentrations (ppm) of vapours were observed. Hydrogen peroxide ($\text{H}_2\text{O}_2$) in liquid form was injected inside the chamber using a calibrated syringe/micro-pipette, where in, it was allowed to evaporate naturally and come in contact with semiconductor sensors kept at elevated temperature. After every measurement, the glass chamber was flushed with fresh air. Measurements of resistance were carried out by using Standard $3^{1/2}$ digit Digital
Multimeter as the resistances of the films were stable and in the measuring range. The resistance was also checked with Keithley Electrometer.

6.2.3 Results and Discussion

6.2.3 (i) Variation of electrical resistance of ITO semiconductor thin film gas sensor with change in operating temperature upon exposure to hydrogen peroxide vapours.

Fig. 6.2.3(i) shows the variation in the electrical resistance of ITO sensor with temperature, in the absence and the presence of the hydrogen peroxide vapours. The concentration of hydrogen peroxide vapours was 333 ppm. As can be seen from the figure, the sensor resistance in the absence of the test vapour remains almost constant with temperature. This shows the stability of the ITO films. Upon injection of the hydrogen peroxide vapours, the resistance of the sensor decreases. It is minimum at 338 K.
As it is known for the oxide semiconductors, the test vapours interact with the surface oxide ions created due to the adsorbed oxygen from the atmosphere on the surface. This results in a change in charge carrier concentration of the material. This change in charge carrier concentration serves to alter the conductivity (or resistivity,) of the material.

In case of an n-type semiconductor, as the majority charge carriers are electrons, upon interaction with a reducing gas, an increase in the conductivity occurs. Conversely, an oxidising
gas serves to deplete the sensing layer of charge carrying electrons, resulting in a decrease in conductivity.

In case of p-type semiconductor it is reverse. A summary of the response is provided in Table 1.

Table 6.2.3 (i) shows the sign of relative change in resistance for oxidizing and reducing gases [13].

Table 6.2.3 (i) Sign of relative change in resistance for oxidizing and reducing gases

<table>
<thead>
<tr>
<th>Classification</th>
<th>Reducing gases/vapours</th>
<th>Oxidising gases/vapours</th>
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<tbody>
<tr>
<td>n-type</td>
<td>Resistance decrease ↓</td>
<td>Resistance increase ↑</td>
</tr>
<tr>
<td>p-type</td>
<td>Resistance increase ↑</td>
<td>Resistance decrease ↓</td>
</tr>
</tbody>
</table>

As the ITO film reflects the characteristics of an n-type semiconductor, the decrease in the sensor resistance in the presence of the vapours shows that the hydrogen peroxide vapours get oxidized by the surface oxygen ion on the surface of ITO film.

\[
O_2 + e^- \rightarrow 2O^- \quad (1)
\]

\[
H_2O_2 + O^- \rightarrow H_2O + O_2 + e^- \quad (2)
\]
Fig. 6.2.3 (ii) The relative change in resistance of ITO sensor with temperature in the presence of hydrogen peroxide (333 ppm)

As can be seen from the equation 2, the oxidation of hydrogen peroxide generates water and oxygen and liberates electron. The generated oxygen again gets adsorbed on the surface and get converted to oxide ion. Thus it has a cumulative effect.

Fig. 6.2.3 (ii) shows the relative change in resistance \( \frac{(R_{air} - R_{gas})}{R_{gas}} \times 100 \% \) of ITO sensor with operating temperature. As can be seen at the operating temperature of 338 K, the redox reaction on the surface of the sensor is maximum. Hence it can
be said that for ITO sensor, the optimum temperature for detection of hydrogen peroxide is 338 K. The decrease in the relative change in resistance after 338 K is due to lowering of the redox activity for higher temperatures.

Fig. 6.2.3 (iii) shows the relative change in resistance of ITO film with temperature for different concentrations of hydrogen peroxide vapours ranging from 166 ppm to 1000 ppm. As can be seen, the relative change in resistance is maximum at 338 K for all the tested concentrations. This confirms 338 K to be the operating temperature of ITO sensor for hydrogen peroxide vapours.

![Graph showing relative change in resistance of ITO sensor with temperature for different concentrations of hydrogen peroxide](image_url)

**Fig. 6.2.3 (iii) The relative change in resistance of ITO sensor with temperature for different concentrations of hydrogen peroxide**
6.2.3 (ii) Variation in relative change in resistance of ITO sensor with different concentrations of hydrogen peroxide

Fig. 6.2.3 (iv) The relative change in resistance of ITO sensor with different concentrations of hydrogen peroxide

Fig. 6.2.3 (iv) shows the variation in the relative change in resistance of ITO sensors with different concentrations of hydrogen peroxide. As can be seen from the figure, the ITO sensors operated at 338 K show almost linear response with concentration in the range 167 to 1000 ppm.
6.2.3 (iii) Effect of Cu promoting layer on the surface of ITO to the response of hydrogen peroxide

Fig. 6.2.3 (v) Relative change in resistance of the Cu/ITO with operating temperature for different concentrations of H$_2$O$_2$

Fig. 6.2.3 (v) shows the relative change in the resistance of the Cu/ITO film with operating temperature in the presence of H$_2$O$_2$ vapours. As can be seen from the figure, for the H$_2$O$_2$ concentrations varying from 160 - 1000 ppm, the relative change in resistance of the film decreases up to 333K and
thereafter increases. This reflects that there is an oxidation as well as reduction of hydrogen peroxide vapours on the surface of Cu/ITO.

6.2.3.(iv) Effect of Ag promoting layer on the surface of ITO to the response of hydrogen peroxide

![Diagram](image)

**Fig. 6.2.3 (vi)** Relative change in resistance of Ag/ITO sensor with operating temperature for different concentrations of H$_2$O$_2$.

Fig. 6.2.3 (vi) shows the relative change in resistance of the Ag/ITO sensor with temperature in the presence of hydrogen peroxide vapours. The nature of response is almost similar to
that of the Cu/ITO as in Fig. 6.2.3(v). However, for sensor with Ag/ITO, the oxidation of hydrogen peroxide after 333 K is more prominent than with Cu/ITO. This is because the Ag metal has got more electron affinity than Cu. The working mechanism with Ag layer on ITO is same as that of Cu/ITO. Ag in presence of the atmospheric oxygen gets converted to Ag₂O, and reduces back to Ag upon the exposure of H₂O₂.
References: Carbon tetrachloride


4. The Great Soviet Encyclopaedia, 3rd Edition (1970-1979). 2010, the Gale Group, Inc. All rights reserved


11. Adil Meziane, Jean-Pierre E. Grolier, Mohamed Baba and Jean-Marie Nedelec, Crystallization of carbon tetrachloride in confined geometries, Dr Jean-Marie Nedelec TransChiMiC, Laboratoire des Matériaux Inorganiques (CNRS UMR 6002), Université Blaise Pascal, 24 Avenue des Landais, 63177 Aubière, FRANCE.


**References : Hydrogen Peroxide**


