CHAPTER 3

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FABRICATION OF SENSOR DEVICE AND ELECTRICAL MEASUREMENTS

3.1 RESISTIVE DEVICE FABRICATION BASED ON:

3.1.1 Pure palladium nanofilm device with interdigitated electrodes

Three simple steps (Figure 3.1) prepared palladium based sensing device. Step 1 includes drawing parallel electrodes (silver paint) using a fine needle manually across the edges of palladium film (film is prepared in section 2.3). Step 2 contains drawing lines perpendicular to these parallel electrodes. More such lines are drawn carefully (along with drying so that they do not conjoin) to form a mesh like design which are called interdigitated electrodes (step 3) so as to cover almost all sections of the sensing film. The separation between any two parallel silver electrodes was less than 1 mm.

![Figure 3.1: Schematic of sensor device (pure Pd).](image)

The two opposite ends of interdigitated electrodes were then connected to the multimeter using thin copper wires to record the change in resistance of the film (Figure 3.2).

![Figure 3.2: Sensing device connected to digital multimeter.](image)
3.1.2 AZO:Pd nanofilm device with two parallel electrodes

The fabrication of AZO:Pd device includes the similar steps as discussed in Figure 3.1. The only difference is that in this case, two parallel electrodes were drawn on the surface of the prepared AZO:Pd films (as discussed in section 2.6) [Figure 3.3]. The total (AZO:Pd) device area was ~5mm×5mm, and the separation between the two parallel silver paste electrodes was ~2 mm. The two opposite ends of interdigitated electrodes were then coupled with the multimeter using thin copper wires to record the change in resistivity of the film (in a similar manner as shown in Figure 3.2). The arrangement of AZO grains and Pd NPs is shown as displayed by AFM images in section 2.7.2.

![Figure 3.3: Schematic of sensor device (AZO:Pd).](image)

3.2 IMPORTANCE OF SUBSTRATE

Substrate is a platform onto which a thin layer of nanomaterial is deposited. A large range of substrates exists like Teflon, glass, Quartz, alumina, plastics, silicon etc. Selection of substrate is in accordance with the application of deposited material. Thin films of thickness less than micrometers loses tendency to withstand the stresses due to temperature and pressure. Thus, a support is needed to withstand these stresses. The supporting material should be porous, hard (resistant to fracture), corrosion resistant, fast adhesion, isotropic, stability, free of contaminants and organic components, moisture free, porous or non-porous (application dependent), etc. Substrates are categorized as interfering substrate and non-interfering substrates. Substrates such as glass are non-interfering substrates, as it does not interfere with the electrical, optical or mechanical properties of deposited film. In case of interfering substrates, the orientation and crystallinity of substrate readily affects the
morphology and crystallinity of deposited film [226-228]. Sometimes it becomes important to use interfering substrates to modify the properties of the film for particular applications.

Many substrates are reported to prepare palladium thin films like glass [217,229-235], Al₂O₃ [214], Silicon [236-238], GaAs [239], alumina [240-241] etc. In this work, palladium nanoparticles are deposited on clean glass substrate (non-interfering substrate) to display overall effect of palladium in hydrogen sensing (no contribution from substrate). A simple low cost glass substrate could reduce the overall price of commercial sensor. The pure Pd nanoparticle based sensors have efficient performance at low temperatures (<60°C) [97,242]. To improve the response of palladium based sensor device at high temperatures also, palladium was decorated on the surface of AZO (Aluminium doped Zinc Oxide) (interfering conducting base). AZO surfaces are stable at high temperatures and their conducting properties could help in maintaining the sensor response of palladium film. Consequently, the Pd decorated AZO devices are useful for both low and relatively high temperature applications. Moreover, this temperature range of application can be further controlled by suitable choice of substrate and catalytic nanoparticle.

3.3 SENSOR SETUP AND ELECTRICAL MEASUREMENTS

A basic gas sensor setup is demonstrated in Figure 3.4. The testing chamber is connected to a gas inlet and gas outlet for the entry and exit of gases respectively. The gas cylinders that are bridged with mass flow controllers and mass flow meters provide the gases to flow in the testing chamber. Mass flow controller is a device that controls the flow of specific gas in a chamber at particular flow rates. Heater provides the wide range of temperature and pressure gauge maintains the desired pressure of gases inside the chamber. The sensing device is placed in this chamber and the electrical change in device is recorded on a sensitive multimeter.
In this study, the prepared sensing device (based on Pd NPs and AZO:Pd) was placed inside the sensing chamber. High purity gases (negligible moisture content (<2 ppm)) like hydrogen, methane, and air [hydrogen gas (H$_2$) 99.998 vol. %; methane gas (CH$_4$) 99.99 vol. %; and air (oxygen ~20 vol. % and nitrogen ~80 vol. %)] were sent into the sensing chamber using mass flow controllers and mass flow meters (Digiflow, USA). For recording the data, Agilent digital multimeter (Agilent U1252A) and Keithley Pico ammeter (Model 6487, M/S Keithley Instruments) were used. The main chamber is placed co-axially inside a resistively heated furnace with a 4 cm constant temperature zone. A copper-constantan thermocouple coupled with a precise temperature controller (±1°C accuracy) is used to monitor the temperature.

Basically, sensor response data is represented as the relative response of sensing device. The electrical sensor response can be plotted in many ways depending on the properties of material. For example, Current vs Voltage (I-V) at constant temperature, Voltage vs Temperature (V-T) at constant current, Resistance vs Time at constant voltage, Response% vs Time at constant gas concentrations etc.

In this study, the hydrogen gas response of prepared resistive sensor devices was determined by plotting device resistance (hydrogen gas loaded) versus exposure time graphs. Since the
electrical properties of palladium film changes when it is exposed to reducing or oxidizing gas [243]. Thus, the response study of palladium based films was carried out as resistance-time plots.

The sensor parameters were computed as follows:

1. **Sensitivity:** Sensitivity is the ability of the sensor of how fast the output (say for example, electrical quantity such as volts or resistance) changes in response to the change in the input parameter. Sensitivity of a resistive gas sensor is calculated by the following formula:

   \[
   \text{Sensitivity} = \frac{\text{change in resistance (gas loaded)}}{\text{resistance (no gas)}}
   \]

   Or

   \[
   \text{Response \%} = \frac{R_a - R_g}{R_e} \times 100, \quad \text{at constant voltage.}
   \]

   where \(R_a\) is the resistance of device in air (no test gas loaded) and \(R_g\) is the resistance of device when test gas is loaded (Figure 3.5)

2. **Response time:** Response time is the time taken by sensor to reach 90% of saturation value.

   From Figure 3.5, response time is given by \(t_2-t_1\)

3. **Recovery time:** Recovery time is time taken by sensor to reach 90% of its baseline value.

   From Figure 3.5, recovery time is given by \(|t_3-t_4|\)

![Figure 3.5: General response pattern](image)

For a commercial sensor, the response time and recovery time should be very low (<1 second)
4. **Selectivity:** Selectivity means that the sensor is unresponsive to the gases other than test gas. If a contribution to the sensor signal comes from another gas, the sensor reading will provide a false measurement.

5. **Reproducibility:** It is the ability of sensor to produce same results after repeated use at constant gas concentration. For a reproducible sensor, response pattern should be same as shown in Figure 3.6.

![Figure 3.6: Reproducible sensor response.](image)

6. **Long-term stability:** Stability is a degree to which sensor results remains constant with time. Long-term stability is very important as sensor performance should not vary within minutes, hours or days. Generally, it happens that the material used to fabricate sensor degrades with time that changes its mechanical, electrical or chemical properties. Sometimes environmental conditions and storage conditions (improper isolation from environment) could also degrade the sensor device.