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Kelvin Probe Studies on Sensor Response Characteristics of H_2 with PT Metal at PPM Level Concentrations under Non-Zero Humidity Conditions

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Abstract
In the present study, Kelvin Probe (KP) technique is used to measure the shift in metal work function (WF) of a thin Pt film, deposited on thermally oxidized silicon substrate, on exposure to diluted hydrogen gas. The WF shift is measured at different H2 concentrations in synthetic air and its sensing behavior is studied for MOSFET configuration. A fixed flow of 100 sccm of the test gas is maintained for all the measurements. The 20% of diluted gas is flown through a water bubbler to obtain the required humidity in the test gas. The sample temperature is maintained at 30oC in the test chamber. The response characteristics i.e. changes in WF and its variations with respect to H2 concentration is evaluated by using a suitably modified Hewlett Packard Visual Engineering Environment (HP VEE program), a graphical programming language. The response characteristics are recorded for ascending and descending orders of hydrogen concentration with a time cycle of 20 min 'on' state and 20 min 'off' state for each concentration of diluted hydrogen. An empirical relation is derived from the observed shift in WF with respect to H2 concentration values. It is observed in the present investigation that the response is almost linear for low concentration values and the WF shift is a reversible phenomenon to a large extent in this range of concentration. The evaluation of response time (90) suggests saturation in WF shift of the film at about 500 ppm of H2 concentration.

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1. INTRODUCTION

Hydrogen gas (H_2) sensing is a subject of extensive research for long, and continues to draw lot of interest for its many scientific and technical applications because of its use as a process gas. H_2 has been extensively used as a reducing agent in chemical processes and as a carrier gas in many semiconductor fabrication steps. It also has been considered as a clean energy resource as a fuel gas, instead of petroleum and/or and projected as a futuristic source of energy. H_2 is a major component in hypergolic propellant fuels and is used for space rocket engines for the required thrust. Because of this, in the rocket propulsion industry, H_2 propellant leaks pose significant operational problems in handling. H_2 is lighter than air and permeates easily through most of the materials where it is stored. Therefore, it leaks easily from its feeding lines as well as through its containers. The identification of exact locations of its leaking points is one of the major problems

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in its industrial applications. H_2 is a combustible gas and explodes above the lower explosion limit (LEL) of 40,000 ppm in the presence of oxygen. It has wide combustion range (4-75%) with small ignition energy of 0.02 mJ. Its burning fires emit large amounts of ultraviolet and infrared radiations without any clear visible flame for its identification. Therefore, a highly reliable device, capable of sensing very low concentration of H_2 with small humidity dependence finds many applications. The high selectivity and long-term stability without any parameter drifts are other essential requirements of such a device.

A solid-state gas sensor is generally defined as a miniature device, which upon exposure to a gaseous species changes one or more of its physical / electrical properties, in a way that can be measured and quantified directly or indirectly [1] the changed parameter and the exposed gaseous species. The changes are generally in the form of gain in total elemental mass (mass sensors), changes in electrical conductivity (conductivity sensors) or variation in the dielectric properties (capacitive sensors) [2]. Accordingly, the sensors are named as mass, conductive / resistive and capacitive sensors respectively based on the parameter variations. Gas sensors based on the work function change of its sensing element forms a new class of sensors, termed as Field Effect Transistor (FET) type sensors and are active devices.

Based on change in properties of sensing element a variety of solid-state gas sensors have been developed for detection of H_2. However, each sensor has different characteristics and offers different level of performance and quality based on the material and exposed gaseous species. Most of these characteristics are reversible in nature and it is expected that properly fabricated solid-state sensors show very long life expectancy with high reliability parameters. Though in the literature many papers have been published on sensing properties of H_2 by Pt-film [3-6] but the objective of the present work is for a specific application. In the present investigation the aim is to examine sensing characteristics of Pt thin film on SiO_2 to H_2 species, at very low concentrations and at 20% relative humidity (RH) condition, which is very close to the realistic environment. This is directly suitable for to develop a device for leak detection in most generic environmental conditions. The work function based investigation is employed here to determine its suitability as a sensing element in three terminal devices such as field effect type MOSFET-sensor, where a sensing film on SiO_2 has a direct relevance.

2. H_2 GAS SENSORS

Several types of sensor structures such as diodes [7], Schottky diodes [5,6,8,9], switching devices [10], SAW systems [11,12], MIS structures [3,13], quartz-crystal micro-balance systems [14], and fiber-optic based sensors [15,16] have been developed and demonstrated for the detection of H_2. Metal and metal alloys [17,18] and different metal oxides [19,20] with and without surface promoters [21], have been used as sensing elements in these reported devices. In the recent past MOS [22,23] and FETs [4,24], ISFET transistors [25], MEMS-based cantilevers [26] have emerged as new class of gas sensors. Each one of these devices has its own advantages depending on its application. Many scientific groups are working actively for the development of integrated H_2 sensor, based on different working mechanisms in which platinum is primarily a sensing component of the sensor structures [3-7]. However, the main emphasis of the researchers is to develop a three terminal device that has a number of advantages over the conventional two terminal resistive or capacitive sensors [27]. Also a three terminal sensor is more compatible for integration with the signal processing circuit on an integrated microchip. The compatibility of a sensor structure with silicon processing technology is necessary to realize an intelligent integrated on-chip sensor. Therefore, world wide the research endeavors are mainly concentrated for the realization of FET type sensor for H_2 gas detection.

H_2 gas has a large diffusion coefficient, 0.61 cm^2/s, in normal atmospheric air ambient. To the best of our knowledge, the large diffusivity of H_2 was utilized in first H_2 sensor based on Palladium (Pd) film [28]. Subsequently Pt and Pt-Pd combinations were investigated to improve sensor sensitivity [4]. Further, the alloys of Pd-Ni or Pd-Ag alloy combinations have also been used to

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suppress phase transition [26]. In a Pd-based H_2 sensor, the surface of a Pd film catalyzes for the breakdown of molecular hydrogen (H_2) species into simple atomic hydrogen, which readily diffuses into bulk of the metal film. In case of sensors, based on Pd-MOS structures, diffused hydrogen atoms are adsorbed at the metal-insulator (SiO_2) interface. These atoms polarize at the interface and give rise to a thin dipole layer, which in turn changes the work function of the Pd metal. Change in metal work function results shift in flatband voltage of MOS capacitor. The magnitude of this shift is a function of number of atoms adsorbed at the metal-SiO_2 interface [23]. Thus the observed shift corresponds to concentration of H_2 exposed onto the metal surface. This basic principle is applied to the development of FET-Gas sensors [13,23,24,27]. In this the sensing element is incorporated in 'gate' electrode of the FET structure. It is either directly put on the gate region or as an extended gate region, as in the case of CCFET [29]. The present work therefore, aims to examine suitability of thin Pt film for FET type sensor capable of sensing low concentration H_2 in environment humidity condition.

3. KELVIN PROBE AND WORK FUNCTION MEASUREMENTS

The work function (WF) of metal is defined as an energy (in eV) that is required to raise an electron from the Fermilevel to vacuum i.e. to a free electron energy level. Kelvin probe (KP) is a well-established technique for the measurement of WF in most of the materials, with high degree of precision. Work function of a material is sensitive to surface conditions and therefore, KP is an excellent tool to measure variation in work function when a gaseous species is adsorbed on the sensing surface. Further, KP is a non-contact, non-destructive technique and mainly employs a vibrating capacitor device to measure the WF difference or for non-metals and non-conducting surfaces the surface potential between a conducting specimen and a vibrating tip with a known frequency. The KP is extremely useful to monitor the WF variations in processes where the reference material acts as an inert one to compare. For this reason, it is very useful for the characterization of gas sensing materials for sensor applications [30,31]. In the present work, we used gold tip (as it is inert to H_2) as vibrating tip and Pt film on SiO_2 substrate as specimen in the KP chamber for measurements.

For any gas sensor applications, it is also very much important for a sensing material to react accurately to the quick changes in gas concentration values surrounding the device. However, for optimization of any H_2 sensor to respond quickly to sensing requirements is not free from its problems. Many H_2 sensors exhibit slower response times means the surface interactions are slow with the hydrogen species. In the present case the response time (τ_{90}) is typically defined as the time it takes for a sensor to reach a 90% of full-scale reading after being exposed to a full scale uniform concentration of a given gas. In certain cases τ_{63} or τ_{80} are also used. In the present study we have adopted τ_{90} criteria for the evaluation of this sensor time constant [32].

4. EXPERIMENTAL

Fig.-1 shows the computer controlled experimental set for sensor response measurements. As seen in the figure, flow of gases into test chamber is controlled through mass flow controllers (MFCs), which in turn receive commands from a PC as per instructions set in the input program file. A water bubbler through which a part of the synthetic air is diverted prior to test chamber is seen in the picture. The design of the KP chamber is optimized such that the total volume in which sample is placed is minimum value and is about 10 cc or less. This is essential for quick clearance of test gas for subsequent measurement and thus to avoid influence of residual gas for subsequent test cycle. The Kelvin probe controller and substrate heating arrangement, shown in the figure, are standard components of a KP set-up.

The present gas sensing properties were studied by using HP VEE (Hewlett Packard Visual Engineering Environment), a graphical programming language, which runs on a set of instructions. The HP VEE object operates by accepting the data as an input for the sequential execution. Thus the submitted data is processed

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sequentially and the resulting information data is returned as an output from the system. The general flow of execution is through a program is called 'propagation' and it mainly runs depending upon the way the objects are connected. The program will not be executed until all of its data inputs are properly activated with the data.

A platinum metal film of 1000 Å thickness was sputter deposited on to a thermally oxidized silicon wafer for the present studies. The sample was kept inside a test chamber of the KP as indicated in the Fig.-1. We have used 100 sccm of test gas (synthetic air plus H_2) flow as the total flow for all these measurements. This total flow is always kept constant and is maintained for all the exposure cycles. 20% of the total flow of synthetic air is bubbled through the water bubbler (kept at room temperature, 23^{o}C) to maintain 20% relative humidity (RH) condition in the sensor test chamber. The temperature of the sample (Pt on oxidized Si) was maintained at 30^{o}C during the entire experiment with the help of a temperature controller connected to a built-in heater (placed beneath the sample) inside the chamber.

Initially, pure synthetic air is flown in the chamber to create a clean atmosphere desired for the precision measurements. This step is always recommended to drive out any unintentional gaseous species already adsorbed on the Pt film surface. Further, this process also stabilizes the surface prior to it is actually exposure to test gas for sensing measurement. The test gas i.e. synthetic air diluted H_2 gas in the present case, at different dilutions is allowed to flow for the measurements. 1000 ppm H_2 (calibrated dilute H_2 in synthetic air, as supplied by vendor) was mixed with pure synthetic air to obtain different desired levels of concentration of H_2 for these measurements.

The input data to the HP VEE program was provided through a format free input file indicating the sequence of events. The events were executed step by step accordingly. At each point of the event the total data were recorded in steps at every second time interval. A graphics visual chart indicates the complete data and the process continues till the last statement of the input file is executed. The complete data is stored in a format free ASCII output file for off-line processing. The data thus obtained was analyzed subsequently for further studies by properly separating the data generated. The WF shift was recorded as a function of H_2 gas concentration, with time intervals of 20 min 'on' and 20 min 'off' cycle, with and without the diluted H_2 gas in the main stream. This is followed for each concentration value i.e. for each concentration the test gas was flown in the chamber for 20 minutes followed by 20 minutes flush by synthetic air prior to next cycle of test gas flow. The H_2 concentration values used are recorded as: 90.09 ppm, 119.76 ppm, 200 ppm, 500 ppm, 1000 ppm, 500 ppm, 200 ppm, 119.76 ppm, and 90.09 ppm in this order to obtain step response as discussed in the next section.

5. RESULTS AND DISCUSSION

A typical response of H_2 sensing of Pt film on SiO_2 is shown in Fig.-2. It is apparent form the result that the change in WF increases with increase in H_2 concentration in the test gas in the ascending cycle. In the descending cycle, the value of WF shift for the same concentration is almost similar. This evinces that reaction of H_2 on Pt surface is reversible i.e. the signal recovers to the original level on removal of H_2 gas. A slight drift in base level seen in the response for subsequent exposure step is attributed to the reminiscent gas in the chamber of the previous test cycle. This is an experimental limitation and can be minimized by reducing further the volume of sample cavity used to test the Pt film.

Fig.-3 shows the response for the 1000 ppm H_2 concentrations on an extended scale to examine the response time characteristics. The open square points are the recordings made at a time interval of one second. This represents a pictorial view of sensing and recovery times of the film at 1000 ppm of H_2.

The WF shifts recorded in Fig.-2, are plotted on a semi-log scale in Fig.-4. It is observed that the shift in WF is continuously raising and showing more or less an exponential trend. These measured data of WF shifts with H_2 concentrations were mathematically fitted in the form of an empirical relation

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$$\Delta WF = 1.3857 \times 10^{-2} \ln [H_2 (ppm)] + 0.14973 \quad \ldots \quad (1)$$

Extrapolation of this mathematical relationship indicates that at 20.3 ppm of H_2 concentration the shift in WF is nearly zero. We are at the opinion that this is the lowest limit measurable by using this technique.

Trinchi *et al* [8] have reported that the response time of the Ga_2O_3 based MRISiC for H_2 gas sensors exhibited a value of 230 s at 210 ^oC and lower values at higher temperature operations. Tournier and Pijolat [33] also reported that the response of H_2 depends on the presence of oxygen concentrations in the sensor environment. The sensitivity measured in pure N_2 as carrier gas is about 10 times as high as in pure air for SnO_2 based sensors. Qiu *et al* [34] have measured the response time of H_2 sensors, based on SiGe, operated around 100^{o}C have shown a value of 50 s at 3% concentration (30000 ppm) levels using synthetic air as the carrier gas. In the case of Pt/TiO_2 Schottky barriers, Varghese *et al* [35] reported several minutes for the devices working at different temperatures, particularly in the range of, 180 ^oC to 375 ^oC and for 1000 ppm of H_2 concentration values.

In Fig.-5, we have presented the response time τ_{90} values vs recorded H_2 concentration. Here the response time τ_{90} is defined as the time a signal response takes to reach 90% of its maximum value (with reference to the base line) for a particular concentration value of a test gas. This is estimated form the Fig.-2 and plotted in Fig.-5 w.r.t H_2 concentration. It is evident from this figure that the response time decreases with increase in H_2 concentration. The fall in response time is faster in low concentration range (90 ppm to 200 ppm) and then decreases slowly to saturate at 500 ppm. This may be explained on the basis understanding adsorption of H_2 on to the Pt surface. For low concentration of H_2, probably the presence already adsorbed atomic or molecular species on the film surface are likely to reduce sites for adsorption of H_2 molecules. Therefore, the H_2 molecules adsorbed on the available sites diffuse laterally first and subsequently interact with film material to cause shift in its work function. Therefore, shift in WF is slow for low concentration H_2 in the test gas. At higher concentrations, there is an abundance of H_2 molecules so the presence of H_2 molecules (in excess to the available sites) in sample vicinity, interact with the already adsorbed species before lateral movement occurs thereby the change in WF is faster. However, this is just a plausible explain we conjectured and one needs to study exact interaction and diffusion processes of the species to quantify the concentration depended response time.

6. CONCLUSIONS

In the present study a computer controlled Kelvin Probe set-up is used to measure the WF difference of thin platinum films, deposited on thermally oxidized Si substrates on its exposure to different H_2 gas concentration values. Shift in the WF is measured at different H_2 concentrations in synthetic air by maintaining the sample at a fixed temperature of 30^{o}C and at 20% RH. The exposure events of different concentration values of H_2 were executed step by step for time interval of one second through an input formatted file. The data was recorded at each event in ascending and also in descending H_2 concentration values in a cycle of 20 min 'on' state and 20 min 'off' state. Data were post analyzed for the shift in WF with respect to the H_2 concentrations. An empirical relationship was derived from the observed shift in WF. The shift in the WF, response behavior and recovery details are discussed in the present paper. From the present study the response time values show a downward trend with increase in H_2 concentration values and saturates beyond 500 ppm concentrations. The results suggest that thin Pt film on SiO_2 substrate gives measurable shift in WF on exposure to H_2 and this can be implemented in gate electrode of a MOSFET structure to realize three terminal FET-Hydrogen sensors.

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Figure Captions

Fig.-1: Computer controlled Kelvin probe setup for work function measurements.

Fig.-2: Sensor response: work function shift vs H2 concentration at 20% RH and sample temperature at 30 oC.

Fig.-3: Response curve at H2 concentrations of 1000 ppm.

Fig.-4: Work function shift vs H2 concentration

Fig.-5: The response-time (□ 90) vs H2 gas concentration.

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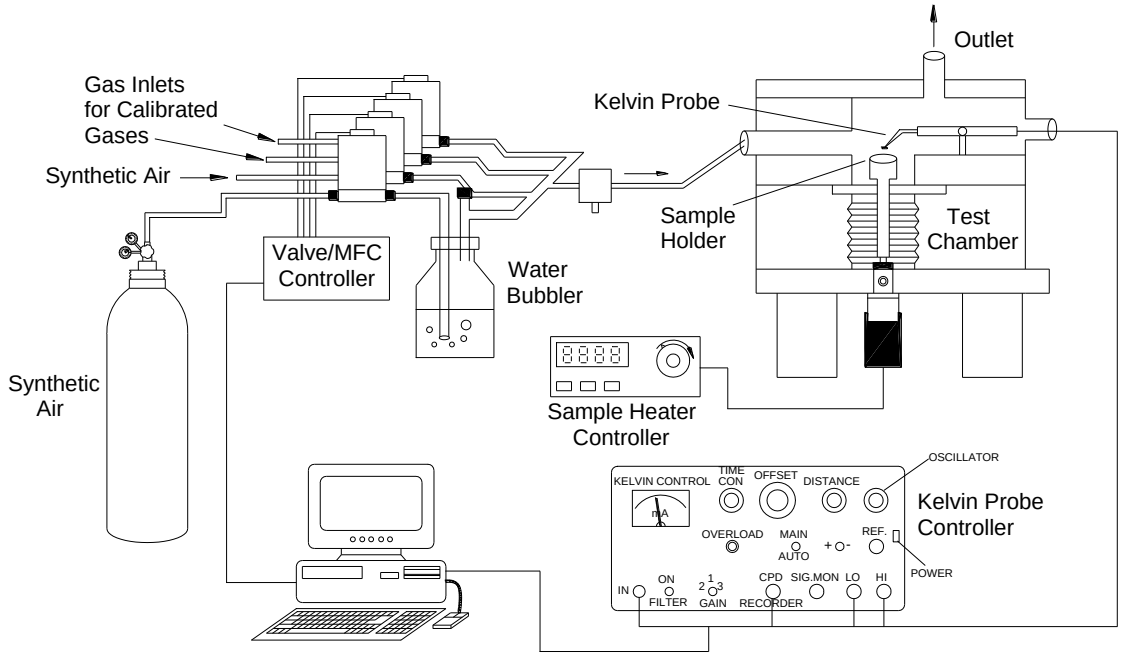

Figure-1

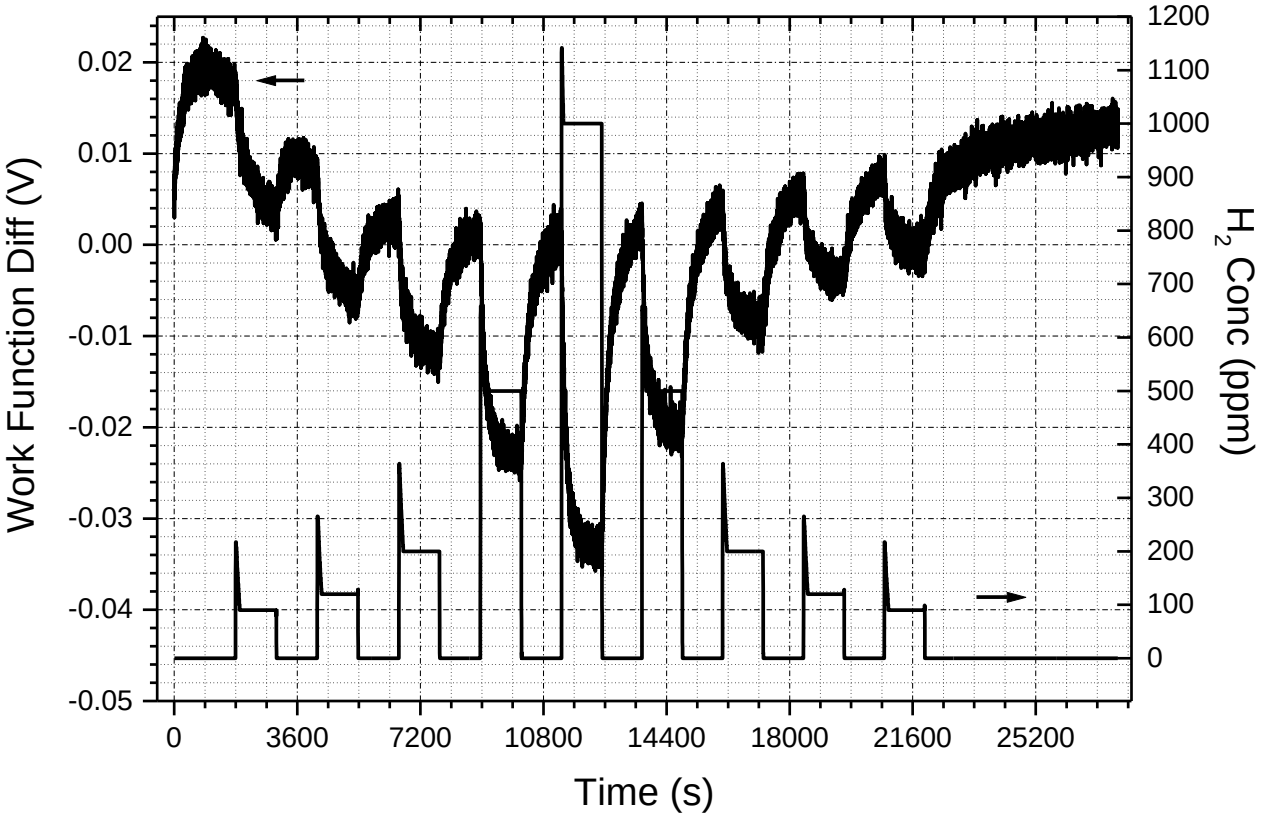

Figure-2

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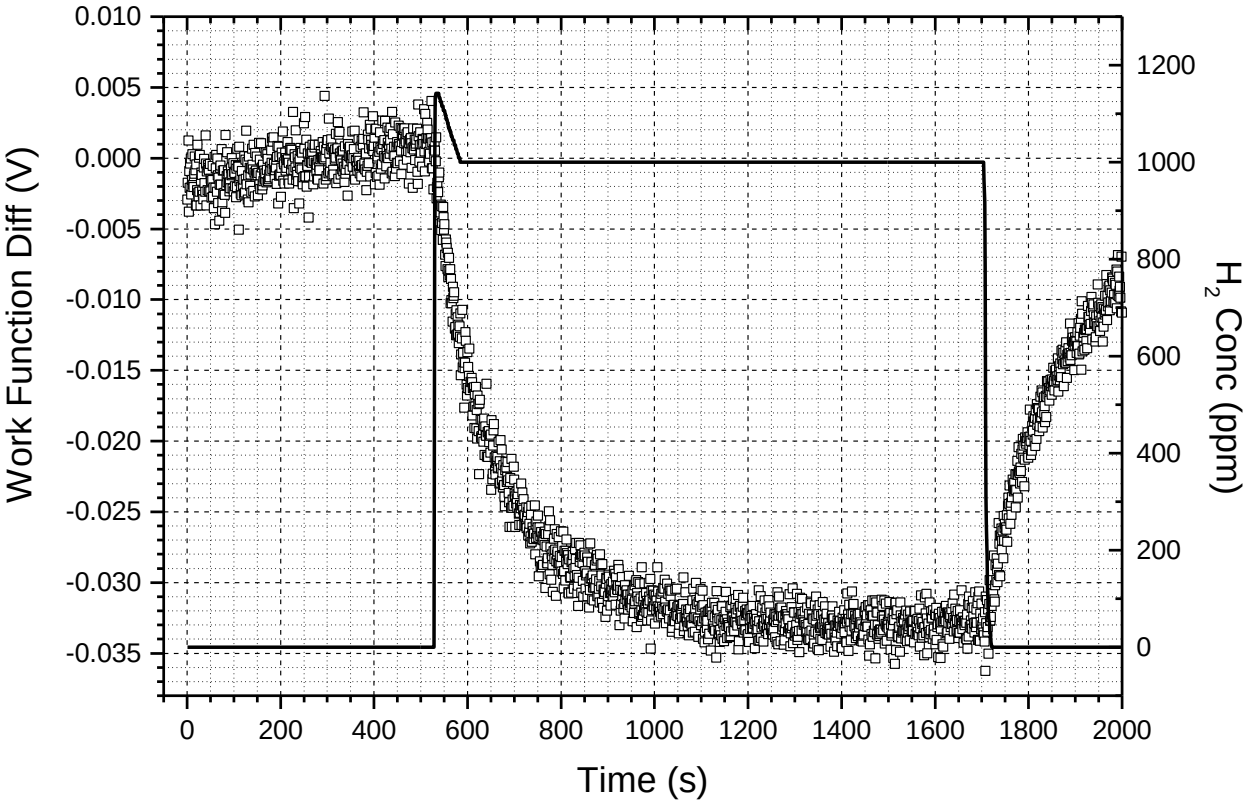

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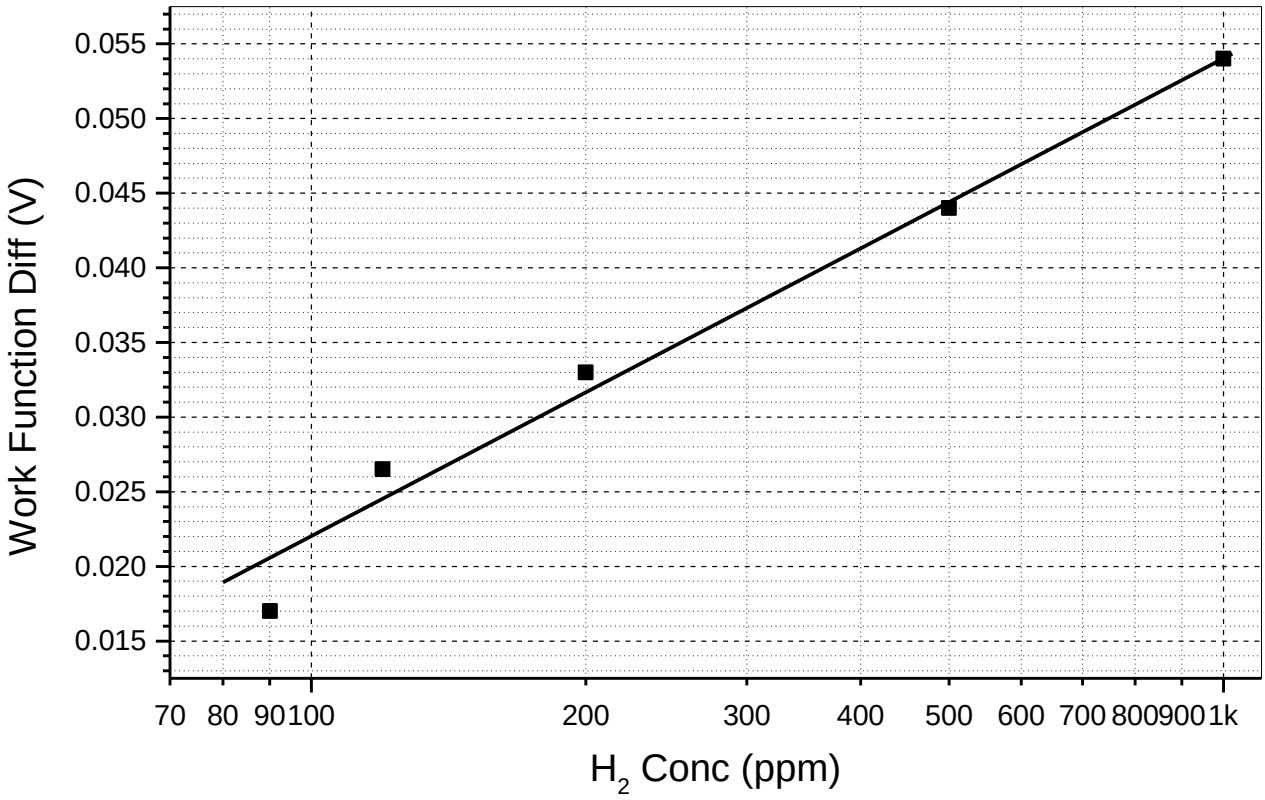

Figure -4

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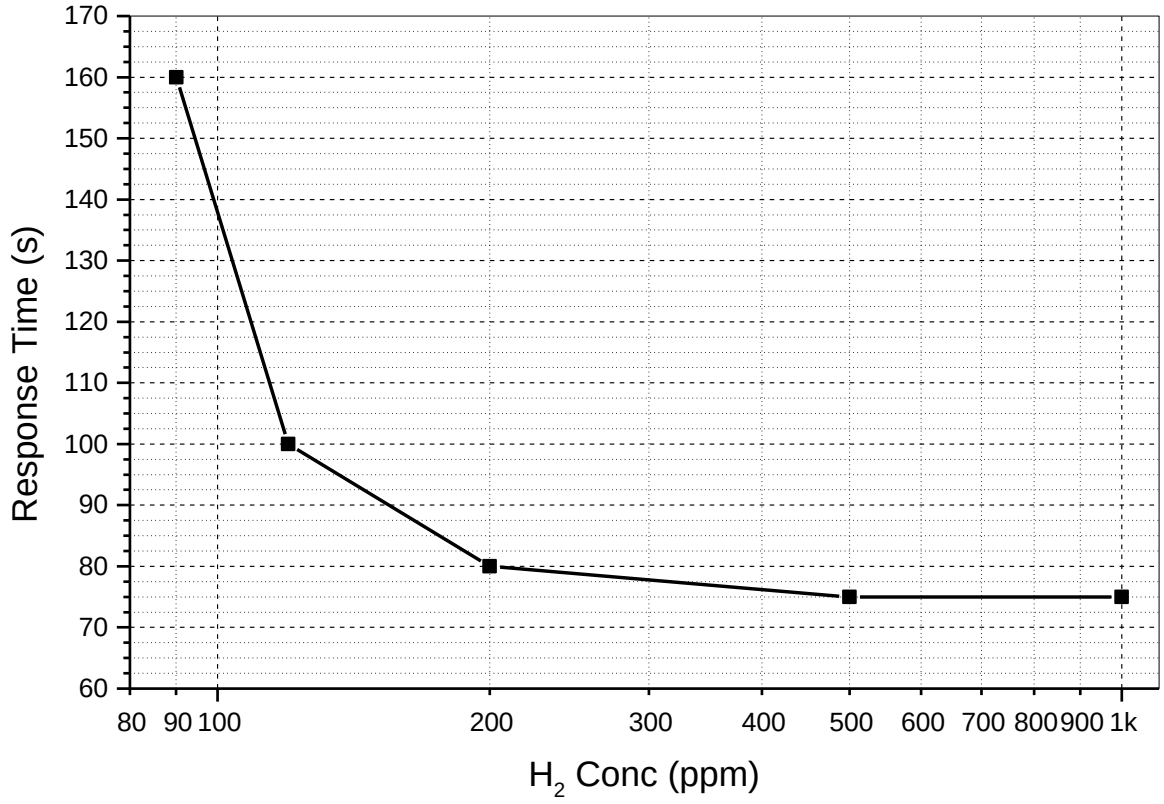

Figure -5