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High Response Hydrogen Gas Sensor Based on Palladium Coated Multi-Walled Carbon Nanotube

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Abstract: H_2, which has a zero-carbon footprint, is expected to be one of the main energy sources in the future. The sensitive detection of H_2 in the transportation, storage and energy production processes will allow the active use of this resource. Recently, there are many studies in which nanotube-shaped structures are used as high-response gas sensors. In this study, H_2 gas response parameters at different temperatures (150, 200 and 250 ºC) of multi-walled carbon nanotube (MWCNT), which were grown on quartz substrate by spin coating method and then Pd coated with DC sputtering, were investigated. The measurements were made at a gas concentration of 1000 ppm with the help of a current-sensitive gas sensor system. The crystallographic structure, elemental content, oxidation levels and surface morphological properties of the produced film were determined by XRS, XPS and SEM analysis. XRD and XPS analyzes support that the MWCNT used in the study is well graphitized and the formation of PdO compound in the structure with Pd coating. The temperature-dependent H_2 gas sensing measurements showed that the produced Pd-MWCNT structure had a very high gas response and the highest response was at 200 °C. Comparing the response values obtained with the results of other Pd-CNT structures in the literature, it was determined that the film produced by the economical spin coating method had a very high gas response.

Paladyum Kaplı Çok Duvarlı Karbon Nanotüp Tabanlı Yüksek Yanıtlı Hidrojen Gazı Sensörü

Anahtar Kelimeler
H_2 gaz sensör, MWCNT, Spin kaplama, XPS, Pd,

Öz: Sıfır karbon ayak izine sahip olan H_2'nin gelecekte ana enerji kaynaklarından biri olması bekleniyor. Taşıma, depolama ve enerji üretim süreçlerinde H_2'nin hassas tespiti bu kaynağın aktif olarak kullanılmasını sağlayacaktır. Son zamanlarda nanotüp şeklindeki yapıların yüksek tepkili gaz sensörleri olarak kullanıldığı birçok çalışma bulunmaktadır. Bu çalışmada, cam altlık üzerinde spin kaplama yöntemi ile büyütülen ve daha sonra DC püskürtme ile Pd kaplanan çok duvarlı karbon nanotüpün (MWCNT) farklı sıcaklıklardaki (150, 200 ve 250 ºC) H_2 gazı tepki parametreleri incelenmiştir. Ölçümler akıma duyarlı gaz sensör sistemi yardımıyla 1000 ppm gaz konsantrasyonunda yapılmıştır. Üretilen filmin kristalografik yapısı, element içeriği, oksidasyon seviyeleri ve yüzey morfolojik özellikleri XRS, XPS ve SEM analizleri ile belirlenmiştir. XRD ve XPS analizleri, çalışmada kullanılan MWCNT'nin iyi grafitleştiğini ve Pd kaplama ile yapıda PdO bileşiği oluşumunu desteklemektedir. Sıcaklığa bağlı H_2 gazı algılama ölçümleri, üretilen Pd-MWCNT yapısının çok yüksek bir gaz tepkisine sahip olduğunu ve en yüksek tepkinin 200 °C'de olduğunu göstermiştir. Elde edilen tepki değerleri literatürdeki diğer Pd-CNT yapılarının sonuçları ile karşılaştırıldığında, ekonomik spin kaplama yöntemi ile üretilen filmin çok yüksek gaz tepkisine sahip olduğu belirlenmiştir.

1. INTRODUCTION

Today, it is extremely important to meet the increasing energy need from energy sources with zero carbon footprint. As an environmentally friendly energy source, H_2 is one of the most important clean energy sources of the future [1,2]. H_2 is a highly flammable and explosive gas with high heat of combustion (120-142 MJ/g). It has a wide flammable range, especially when its amount in the environment exceeds 4% [3]. In addition to these properties of H_2, its colourless, odourless and tasteless nature makes it impossible for human senses to detect it. Parallel to the increasing use of H_2 energy, this gas needs to be carefully monitored during storage, transportation and usage.

In detecting any gas with the aid of a sensor, it is essential that the sensor has fast response (τ_{res}) and recovery times (τ_{rec}), high response magnitude (R) and chemical stability. In this context, it is requested that the gas sensors that are tried to be developed should have the specified features together [1]. Different semiconductor metal oxides (SMO) such as TiO_2 [4–7], MoO_3 [8], WO_3 [9,10], SnO_2 [11,12], ZnO [13,14] and graphene [15] are used in H_2 gas sensor applications. These SMOs can have different structures such as nanotubes or nanowalls. Many studies emphasize the importance of nanotube structures for obtaining high gas response magnitude and low response times [1]. Nanotubes generally exhibit good gas sensing performance, as their large surface area allows H_2 molecules to interact more. The most well-known of these nanotube structures are single (SWCNT) and multi-walled carbon nanotubes (MWCNT). Both SWCNT and MWCNT are basically different shape formations of graphen. While SWCNTs are the rolled-up form of a single graphene layer, MWCNTs are composed of graphene layers that are concentrically nested within each other. The diameters of CNTs are usually in the order of nanometers [16]. CNTs may exhibit metallic or semiconductor characteristics, depending on the direction of graphene. CNTs have high thermal conductivity at room temperature, superconducting characteristics at low temperatures, and a tensile strenght approximately 56 times higher than steel [17]. Both SWCNTs and MWCNTs have a wide range of electro-analytical and radiation applications [18,19]. In addition, there are many studies indicating that CNTs can be used as H_2 gas sensors. Kong et al., published a study suggesting that CNTs could be used as H_2 gas sensors [20]. In that study, SWCNT has been deposited on the silicon substrate by chemical vapor deposition (CVD) method. The authors also coated the film surface they produced with Pd. Response and recovery times for H_2 gas at 400 ppm gas concentration were calculated as 2 and 1.5 s, respectively. Zilli et al., formed Pd-MWCNT nanocomposite structure with CVD growth system and determined the H_2 gas response with a system sensitive to current change [21]. The authors reported that the composite they produced had a response time of 150 s and that the H_2 gas sensing parameters changed significantly with increasing H_2 concentration. Data in the literature indicate that Pd modification is required to increase H_2 gas adsorption [1]. There are many studies examining the response characteristics of the Pd-CNT structure at different gas concentrations. In different studies, the researchers have been stated that the H_2 gas responses of the structures as 407 at 200 ppm [22], 1000 at 311 ppm [23], 1260 at 3000 ppm [24], 2 at 400 ppm [20] and 400 at 10000 ppm [25].

In this study, the structural, morphological properties and temperature dependent H_2 gas sensor detection capabilities of Pd-MWCNT nanocomposite, were investigated. Gas sensor measurements of the produced nanocomposite were taken at 150, 200 and 250 °C under a constant 1000 ppm H_2 gas concentration. The obtained results are given in comparison with the results of Pd nanocomposites previously published in the literature.

2. MATERIAL AND METHOD

In this study, MWCNTs of 99% purity, 10-20 nm in diameter and 10-30 μm in length, purchased from Graphene Chemical Industries, were used. For film growth by spin coating, MWCNT was mixed with ethanol at 0.25 mg/ml. The resulting solution was ultrasonicated for 1 hour. The obtained solution was dropped by a micropipette onto the quartz substrate, which was previously subjected to the conventional cleaning procedure. The dripped solution was spinned for 30 s at 3000 rpm at room temperature. These dripping and rotating processes were repeated 3 times. DC sputtering system was used to make Pd modification to the obtained film. During the sputtering process, the sputtering parameters were set as chamber pressure 3 mtorr, deposition time 120 s, substrate temperature 200 ºC, deposition power 20 W. In order to take the H_2 gas sensor measurements of the produced Pd-MWCNT structure, IDE (interdigitated digital electrode) Pt contacts were made in the thermal evaporation system.

PANalytical/Empryean and Specs-Flex devices were used for XRD and XPS analysis, respectively. The average angular resolution of this XRD device, which has a power of 4kW, is 0.026°. In XRD measurements, a Ni-filtered Cu source with a wavelength of 1.54 nm (Kα) was used to generate X-rays. Obtained XRD and XPS data were analyzed with Origin 8.5 (Demo) and CASAXPS (Demo) and versions. Zeiss Sigma 300 model scanning electron microscope (SEM) device was used to obtain surface morphological images. The gas sensor properties of the synthesized Pd-MWCNT film was determined for H_2 gas for 150, 200 and 250 °C temperature and 1000 ppm gas flow with the Keithley 487 picoammeter in the current-sensitive gas sensor measurement system. In these measurements, a voltage of 1V was applied to the metal contacts on the samples.

3. RESULTS AND DISCUSSION

XRD analysis was carried out to determine whether the Pd coated multi-walled carbon nanotube, which was used as an H_2 gas sensor, was in a crystallographic structure. The XRD profile obtained between $2\theta=10^o$-90^o of the examined sample is given in Figure 1. This figure

shows a dominant peak corresponding to 2θ=26º and low intensity peaks corresponding to 36, 43 and 53º. Except for the peak corresponding to 36º, the others represent the classical crystallographic planes of CNTs (002), (100) and (004). The peak observed at 36º reveals the formation of PdO in the structure during the Pd decoration process. The fact that the 002 plane, one of the crystallographic planes of MWCNT, is very dominant like a single crystal, reveals that the MWCNT examined in the study is well graphitized [26]. The crystallite size for 002 plane of MWCNT calculated as 68.5 nm.

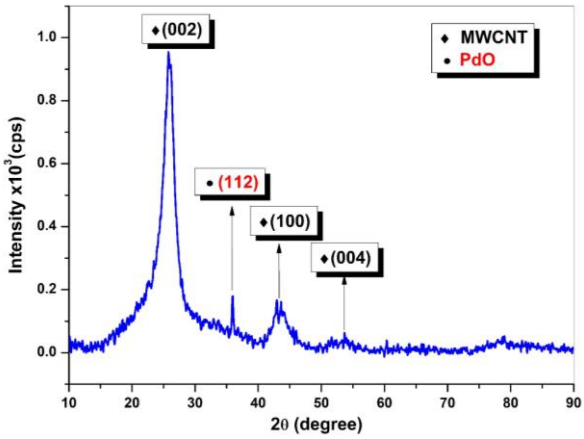

Figure 1. XRD profile of the Pd-MWCNT nanocomposite

Since the gas sensor properties of any material are directly related to the chemical properties and morphology of its surface, both survey and high resolution XPS spectra of the produced film are given in Figure 2 (a-d). From the survey profile given in Figure 2 (a), it is important that only C, Pd and O are present in the structure. This can be considered as an indication that the production stage is designed not to cause any pollution. The high intensities of the peaks of Pd can be seen from Figure 2 (a), as XPS analyses can provide information from the surface of the examined sample to a depth of 10-20 nm. The high resolution XPS spectrum of the Pd 3d orbital is given in Figure 2 (b). From this figure, it is seen that there are two dominant peaks corresponding to Pd 3d3/2 and Pd 3d5/2 peaks resulting from spin-orbit interaction, and 2 low-intensity peaks supporting PdO formation. The good agreement (R^2=0.9975) between the measured raw data and the cumulative peak data obtained after deconvolution can be considered as an indication that the peak separation is done correctly, that is, all formations in the structure of Pd are presented fully. The C1s peak is currently used in almost all XPS studies, both in calibration procedures and in determining the bond structures of C. The specific scanning spectrum of the C1s peak is given in Figure 2 (c). The structure being MWCNT, requires a high proportion of pure C bonds, as expected, with 46.17% of C-C bonds supporting this situation. It was determined that C-C, C-H, C=O and O-C=O formations occurred in the structure, respectively, with the deconvolution of the C1s peak, and their relative distributions were 46.17%, 28.23%, 10.80% and 14.81%. In order to determine the oxidation species and levels of the Pd-decorated MWCNT film, the specific scanning spectrum of the O1s peak of the film is shown in Figure 2 (d). This spectrum reveals that there is only chemical bonding between C, O and Pd in the structure. That is to say, there are only peaks corresponding to C-O, Pd-O, and O-Pd-O in the deconvoluted spectrum. Among these peaks, it can be clearly seen from the figure that C-O and Pd-O peaks are dominant. According to the elemental analysis processes performed according to XPS analysis, the atomic percentages of O, C and Pd elements in the structure were calculated as 69.113%, 15.745% and 15.141%, respectively. As mentioned before, the fact that XPS is only sensitive to the sample surface caused the Pd and O ratios to be higher than expected. In addition, XPS analyses clearly support the existence of PdO formation, which is thought to exist in the structure according to the XRD results.

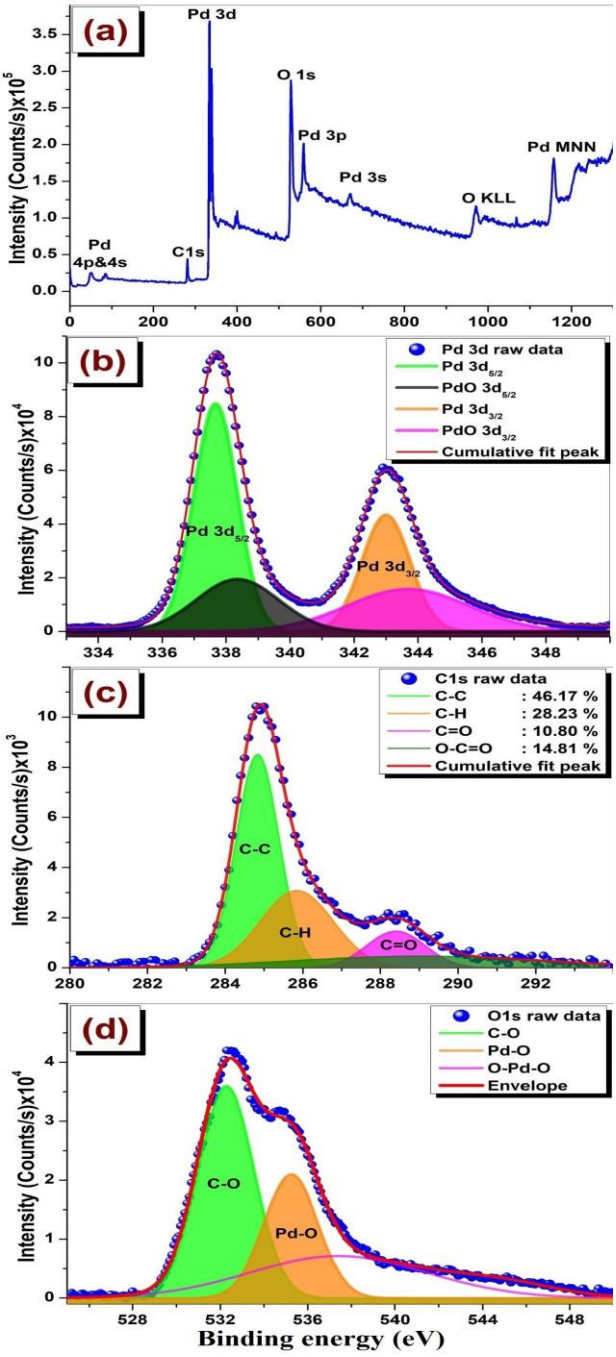

Figure 2. (a) XPS survey spectrum of Pd coated MWCNT (b-d) High resolution spectra of Pd, C and O, respectively

The morphological structure and elemental content of the Pd decorated MWCNT film were investigated using SEM-EDAX. From the 10 KX SEM image given in Figure 3(a), it can be seen that the quartz substrate is almost homogeneously coated with MWCNT. Although it is thought that there may be agglomerations in places, it is clear that MWCNTs completely cover the surface, forming a very tightly packed fibrous structure. From the images in Figures 3 (b) and (c) given for 100 KX and 150 KX magnifications of this film, it is seen that the nanotubes are oriented parallel to the surface and the tube diameters vary between 23.97 nm and 31.99 nm. In addition, it can be seen from Figure 3 (c) that the nanotube surfaces have a porous structure. The energy dependent EDAX spectrum of Pd coated MWCNT is given in Figure 3 (d). The peaks seen in this spectrum belong to C, O and Pd. This supports the qualitative analysis results obtained from XRD and XPS analyses. Elemental percentages by mass obtained by analyzing these peaks were calculated as 83.46%, 6.02% and 10.52% for C, O and Pd, respectively. The fact that the electrons used in EDAX can penetrate more into the structure allows data to be obtained from lower layers compared to XPS. So, this is the main reason why the elemental percentages given look different from XPS.

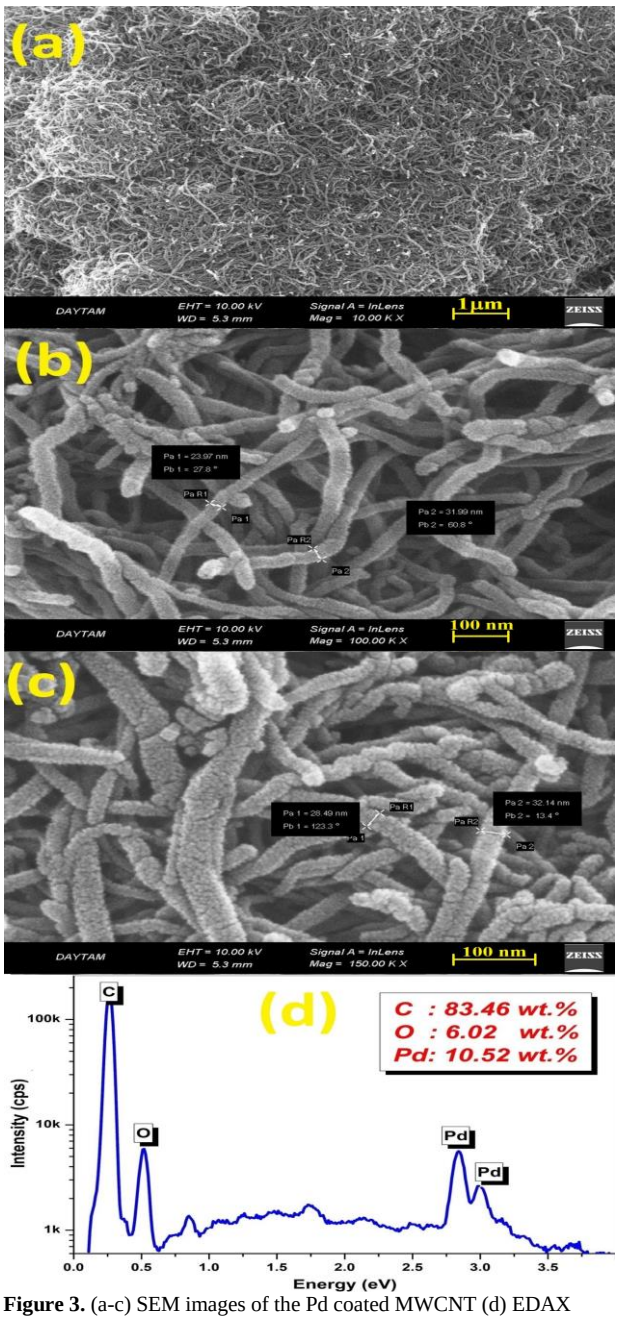

Figure 3. (a-c) SEM images of the Pd coated MWCNT (d) EDAX profile of the MWCNT

The H_2 gas sensor characteristics of Pd coated MWCNT films as a function of temperature are shown in Figures 4 (a) and (b). PdO on the surface of the film exposed to the hydrogen environment undergoes surface reduction. In such a case, palladium hydride is formed by bonding palladium and hydrogen [27]. When the PdO compound homogeneously coated on MWCNTs interacts with H_2, the number of free electrons in the structure increases. This corresponds to an increase in the carrier charge concentration in the structure, that is, to a decrease in the electrical resistance of the structure. In addition, the coating of high conductivity MWCNTs with PdO causes the gaps between nanotubes to be filled. Due to the conversion of PdO to Pd metal in the hydrogen environment, the conductivity of the structure is further strengthened and its electrical resistance is significantly reduced. In a film produced with such a system, in the H_2 gas environment, it is expected that the electrical resistance will decrease, that is, the current will increase.

This described situation is clearly seen in the PdO coated MWCNT film. When Figure 4 (a) is examined, it is seen that the I/I_0 values show a significant increase in the case of the application of H_2 gas, and that the resistance of the structure increases in the case of gas cut-off. This is a proof that the produced thin film giving response for H_2 gas.

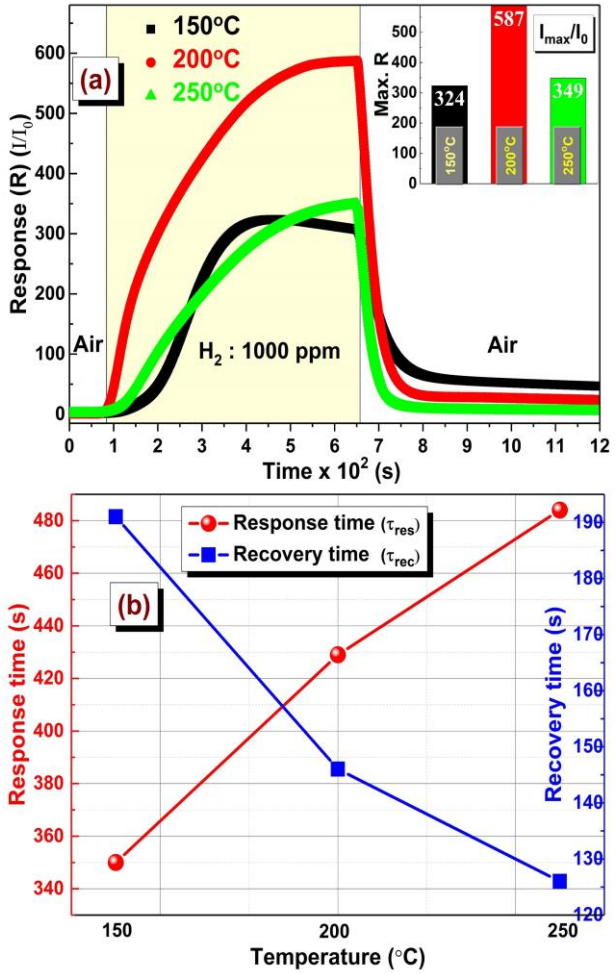

Figure 4. (a) Current-time variations of Pd coated MWCNT for 1000 ppm H_2 gas (b) Variations of temperature dependent response and recovery times

The gas response of any gas sensor device can be determined from changes in current or resistance. An ideal sensor is expected to have a high response value and low response and recovery times. In the calculation of gas responses, some researchers calculate the response values with $100x(I_m-I_0)/I_0$, while others use I/I_0 values directly. Essentially, both of these procedures are accurate in determining the gas response, but a normalization is required to compare the gas responses of the produced materials. In this study, the gas response of the produced film was calculated using I/I_0. In this equation, I represents the current value at any instant, while I_0 corresponds to the minimum current. When the I/I_0 changes of the measurements taken at 150, 200 and 250ºC are examined to determine whether the H_2 gas response of the PdO-coated MWCNT film changes with temperature, it is seen that the highest gas response is 587 at 200 ºC. The inset plot of Figure 4 (a) reveals that the maximum H_2 gas response values of this film first increase and then decrease with increasing temperature. From these data, it can be concluded that in order to obtain the maximum H_2 gas response from PdO coated MWCNT films, it is necessary to operate them at an operating temperature of 200 ºC. However, it cannot be said that a manufactured material is an ideal candidate for a gas sensor based solely on the magnitude of the response value. Another parameter that can be used for this purpose is the response times. In the calculation of the response times, starting from the minimum current, the time it takes to reach 90% of the time corresponding to the maximum response is used, while in the recovery time, the 90% decrease time from the maximum is used. In the light of this information, the temperature dependent changes of the calculated τ_{res} and τ_{rec} times are shown in Figure 4 (b). Accordingly, the minimum response and recovery times were obtained at 150 and 250 ºC, respectively. While the response times have an increase with increasing temperature, the recovery times have an almost regular decrease. From the evaluation of response and recovery times together, it is concluded that the optimum condition is at 200 ºC. At this temperature, response and recovery times were calculated as 429 and 146 s, respectively. Therefore, due to both the high gas response and the response times that can be considered optimum compared to other temperatures, it was concluded that it would be appropriate to operate PdO-coated MWCNT films at 200 ºC for H_2 gas sensing.

In order to make a meaningful evaluation of the data obtained in this study, the sensing parameters were compared with some studies in the literature. Table 1 shows the response values and response calculation formulas for H_2 gas of some Pd decorated structures (SWNT, MoS_2, WO_3, and graphene). When this table is examined, it can be seen that the Pd-MWCNT structure produced in this study has high response values compared to other materials. In some studies, given in the table, it is seen that the equation $(R_a-R_g)/R_a\times100$ is used to calculate the R parameter. Calculating the R value of any material with this method causes the results to be higher than calculations with R_g/R_a or I/I_0. Although the I/I_0 method is preferred for the calculation of R values in this study, it is clear that the H_2 gas R values obtained are higher than almost all other materials. If the data obtained in the study are calculated with a result similar to the other equation, the H_2 gas response of Pd-MWCNT measured at 200ºC will be 58600. In addition, considering that the measurements in this study were taken at 1000 ppm gas concentration, it can be easily expected that much higher responses will be obtained at 3000 or 10000 ppm values.

4. CONCLUSION

In this study, Pd coated MWCNTs with very high H_2 gas response values were successfully fabricated. It has been determined that the produced nanotube film has response values of 324, 587 and 349 for H_2 gas at 150, 200 and 250 °C, respectively, and the response times are competitive with other studies presented in the literature. From the evaluation of response times and response magnitude values together, it was determined that the

use of Pd coated MWCNT film at 200 °C would give better results. XRD and XPS results indicate that PdO compounds are formed on the surface of the structure with Pd coating. This PdO compound interacts with H_2 and the number of free electrons in the structure increases. This corresponds to a decrease in the electrical resistance of the structure. Moreover, the coating of MWCNTs with PdO leads to the filling of the gaps between the nanotubes. Due to the conversion of PdO to Pd metal in hydrogen environment, the conductivity of the structure is further enhanced and the electrical resistance is significantly reduced.

Table 1. Comparison of H_2 gas sensing parameters of produced Pd-MWCNT with some studies in literature

Sample	τ_{res} [s]	τ_{rec} [s]	R	G.C. (ppm)	T (°C)	Definition of R	Ref.
Pd-SWNT	420		1260	3000	RT	$(R_a-R_g)/R_a\times100$	[24]
Pd-SWNT	15	300	400	10000	RT	$(R_a-R_g)/R_a\times100$	[22]
Pd-WO3	42	48	169	1000	80	R_g/R_a	[28]
Pd/MoS2	786	902	35	10000	RT	$(R_a-R_g)/R_a\times100$	[29]
Pd/Graphene	60	--	33	10000	RT	$(R_a-R_g)/R_a\times100$	[30]
Pd-MWCNT	350	191	324	1000	150	I/I_0	This work
Pd-MWCNT	429	146	587	1000	200	I/I_0	This work
Pd-MWCNT	484	126	349	1000	250	I/I_0	This work
R: Response, T: Temperature, RT: Room Temperature, G.C.: Gas Concentration							

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