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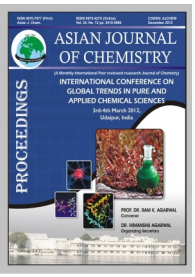

V_2O_5-P_2O_5 Glass Ceramic as a Resistive Solid-State CO_2 Gas Sensor†

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Now a day the specific demand for gas detection and monitoring has emerged particularly as the awareness of the need to protect the environment has grown. The solid-state gas sensors, based on a variety of principles and materials, are the best candidates to the development of commercial gas sensors for a wide range of applications. The resistive type solid-state gas sensor of V_2O_5-P_2O_5 (1:1 M) glass ceramic was fabricated by melt quenching method on silica substrate. The sensor was investigated for CO_2 gas environment at lower ppm level (up to 70 ppm) at room temperature. The response (sensitivity) of sensor at 70 ppm of CO_2 was found to be 0.3. The sensing parameters such as resolution limit, resistance change/ppm and response time were observed to be 5.5 ppm, 195×10^6 Ω/ppm and *ca.* 2 min respectively. The sensor material was characterized by XRD and SEM analysis.

Key Words: V_2O_5, P_2O_5, Solid-state gas sensor, Sensitivity, Resolution limit, CO_2 gas.

INTRODUCTION

The structure of V_2O_5 is built of octahedrons, where the pentavalent vanadium has 5-coordination with oxygen atoms, the V_2O_5-rich glasses in which V_2O_5 acts as the network former have the network structure mainly consisting of corner-sharing branched VO_4 tetrahedral of the same structural units as found in phosphate glasses. The network structure was reported to be made-up unaffected VO_5 groups as in vitreous V_2O_5 and affected VO_5 groups with alkaline earth ions in contrast to the vanadate glasses formed by conventional network formers in which only unaffected VO_5 groups are present. These glasses are known to contain V^{4+} and V^{5+} ions where the electrical conduction was attributed to the hopping of $3d^1$ unpaired electron from V^{4+} to V^{5+} site, which induces a polarization of the vanadium ion around it and forms a polarons. Vanadate has received greater attention as a new branch in semi-conducting glasses because of its wider glass-forming region in the phase diagram and its possible technological applications in threshold switching, memory switching, electrical threshold, electrochemical batteries and optical switching devices[1].

Solid-state gas sensors (SGS), also known as chemoresistive gas sensors, are typically based on metal oxides. Solid-state gas sensors are one of the most widely studied groups of chemiresistive gas sensors. These sensors are designed to react with one class of gases, whereby the solid-state gas undergoes reduction and oxidation. This process causes the solid-state gas sensors to exchange electrons with the target gas at a certain characteristic rate, thereby affecting the sensor's resistance and yielding a certain signal[2,3]. During the past few decades, solid-state gas sensors have become a prime technology in several domestic, commercial and industrial gas sensing systems. Among the available gas sensing methods, the solid-state gas sensor devices have several unique advantages such as low cost, small size, measurement simplicity, durability, ease of fabrication and low detection limits (< ppm levels). In addition, most solid-state gas based sensors tend to be long-lived and somewhat resistant to poisoning. For these reasons, they have rapidly grown in popularity[4]. Several theoretical and applied articles have shown the advantage of reducing the metal oxide grain size down to nanometer scale in order to improve the sensing properties (mainly sensitivity and selectivity) as well as stability over time of the oxide layer[5].

Several materials are fabricated to enhance the sensing characteristics of the solid-state gas sensors (SGS). Various metal oxides mixed with different dopants, catalysts, adhesives, binders, volatile fillers and electrodes all have been studied. In addition to the variations in the composition of the metal oxide materials, their film deposition methods provide another variable for sensor design. These deposition methods include

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pyrolysis, oxidation of metallic films, reactive sputtering, chemical vapour deposition (CVD), laser ablation and electron-beam evaporation techniques[6-8]. Metal oxides exhibit various electro-physical features, ranging from insulators to wide band-gap semiconductors. The non-transition metal oxides contain elements with one oxidation state because they require a large amount of energy to make other oxidation states that would bind to the oxygen ion ligand. In contrast, because of the various oxidation states that might form on transition metal oxides compared to non-transition metal oxides, the surface properties and the types of chemisorptions that occur on the surface are important and have been widely studied. This variation in the oxidation states causes significant changes in the surface chemistry response toward oxygen and other target gaseous molecules[9].

In the present investigation, the resistive type solid-state gas sensor of V_2O_5-P_2O_5 (1:1 M) glass ceramic was fabricated by melt quenching method on silica substrate. The sensor was investigated for CO_2 gas environment at lower ppm level at room temperature. The sensing parameters such as resolution limit, resistance change/ppm and response time were determined. The material was characterized by XRD and SEM analysis.

EXPERIMENTAL

The glass sample of V_2O_5-P_2O_5 with 1:1 M was prepared by the melt quenching technique. Required quantities of V_2O_5 and P_2O_5 were mixed together by grinding the mixture repeatedly to obtain a fine powder. The mixture was dried at 373 K for 0.5 h. Then the mixture was melted in a silica crucible in an electrically heated furnace under ordinary atmospheric conditions at a temperature of about 1100 K for 3 h to homogenize the melt. The sensor film was fabricated by melt quenching method on silica substrate. The melted mixture was poured on mercury leveled 2 × 1 cm^2 silica substrate. For electrical contact, the silver electrodes were deposited on adjacent sides. The sensor film was kept at 400 K for drying the silver contacts. The sensing characteristics of sensor were investigated in CO_2 environment at room temperature. The sensing measurements were described in details elsewhere[10]. The X-ray diffraction pattern of film was recorded on Rigaku X-ray diffractometer using CuK_α radiation (λ = 1.54 Å). The diffractogram was in terms of 2θ in the range 10º-90º. The morphology of film was investigated by using JEOL-JSM SEM instrument.

RESULTS AND DISCUSSION

Fig. 1 shows the X-ray patterns of V_2O_5-P_2O_5 glass ceramic sensor film. The XRD spectra of sample was recorded between 2θ = 10-90º. The X-ray pattern of sample shows noisy spectra and the peak was observed at the position 25º, which is attributed to the amorphous halos nature of glass ceramics.

The surface morphology of V_2O_5-P_2O_5 glass ceramic sensor film was analyzed by SEM micrograph and shown in Fig. 2. The amorphous nature seen in the XRD is reflected in the SEM micrograph also. The surface morphology of sample appears flat and firm in picture.

The sensitivity is defined as in eqn. 1[11].

$$S = R_g - Ra/Ra \quad (1)$$

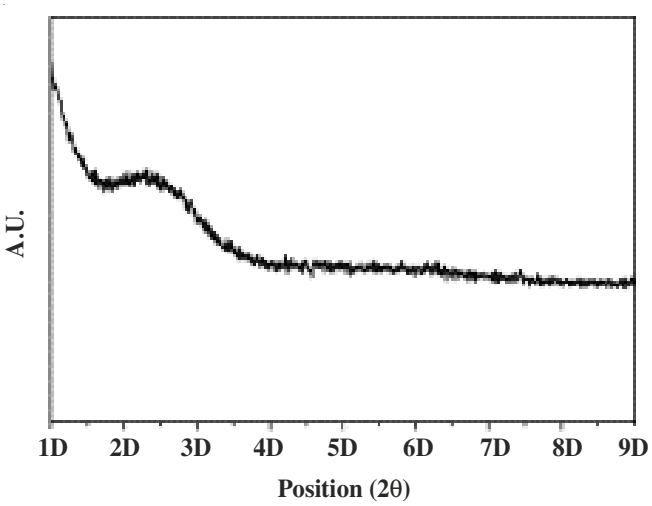

Fig. 1. XRD spectra of glass ceramic sensor film

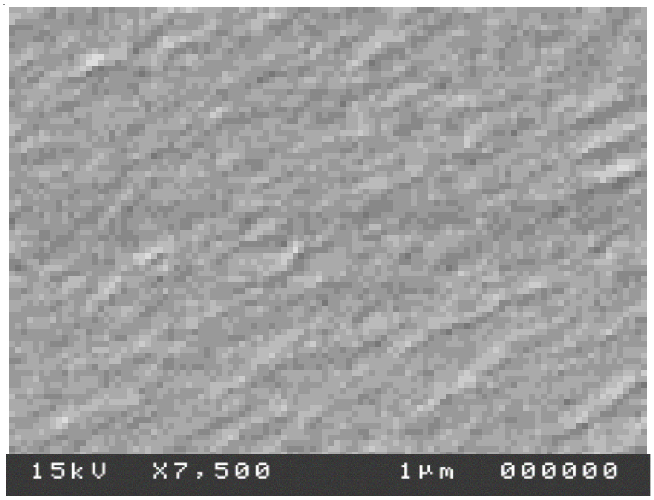

Fig. 2. SEM picture of glass ceramic sensor film

where, Ra and Rg are the resistance of sensor in air and the CO_2 gas respectively.

The resistance of the film was found to be increases linearly with CO_2 gas concentration at room temperature. The resistance change/ppm was found to be 195 × 10^6 W/ppm. The sensitivity (response) of sensor film was determined from eq. 1. and found to be linear in nature (Fig. 3). The saturation effect was not seen upto 70 ppm of CO_2 gas. During experiment, it is observed that the response time of film was -2 min.

The possible mechanism for CO_2 gas detection in material is based on reactions that occur at the sensor surface, resulting in a change in concentration of adsorbed oxygen. At lower temperature (< 150 ºC), oxygen preadsorption at the surface is mainly in the form of O^{2-}. Oxygen ions adsorb onto the surface of material removes electrons from the bulk and create a potential barrier that limits electron movement and resistivity. When exposed to an oxidizing gas such as CO_2 then it is chemisorbed on bridging oxygen atoms with the formation of a surface carbonate, subsequently increasing the barrier height and resistivity[12].

The resolution limit is lowest concentration difference that can be distinguished by sensor[13]. The resistance of V_2O_5-P_2O_5 glass ceramic sensor film in air was found to be 32257 × 10^6

Ω and for different ppm as listed in Table-1. From Table-1, it is clear that the resolution limit of sensor was found to be 5.5 ppm.

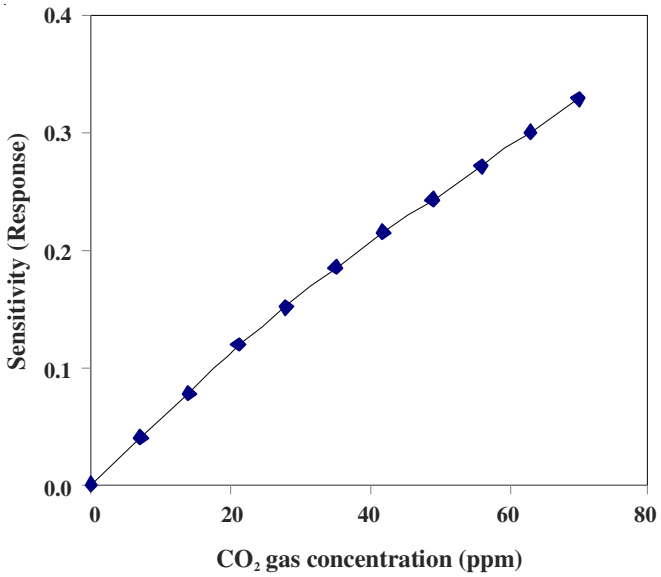

Fig. 3. Sensing response of sensor film

TABLE-1
RESOLUTION LIMIT OF SENSOR FILM

Concentration of CO_2 (ppm)	Resistance of sensor ($\times 10^6$ Ω)
0	32257
1	32257
2	32257
3	32257
4	32257
5.5	33252

Conclusion

The sensitivity (response) was found to be linearly increases with different concentration of CO_2 gas at room temperature. The saturation effect was not seen upto 70 ppm of CO_2 gas. The higher value of CO_2 gas sensitivity was found to be 0.3 at 70 ppm. The resistance change/ppm was found to be 195×10^6 W/ppm. The resolution limit for sensor towards CO_2 was found to be 5.5 ppm. The response time of film was found to be -2 min. The amorphous nature was seen in the XRD spectra. The SEM micrograph reveled that the surface morphology of sample was appeared flat and firm.

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