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# Comparative and Efficient Ammonia Gas Sensing Study with Selfassembly-Synthesized Metal Oxide-SiC Fiber-Based Mesoporous SiO<sub>2</sub> Composites

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**ABSTRACT:** Self-assembled-assisted ternary nanocomposite  $In_2O_3$ -SiC,  $CuO_2$ -SiC, and  $MnO_2$ -SiC semiconductors were mixed with SiO<sub>2</sub> to enable gas sensing using cyclic voltammetry. The results of TEM (transm  $In_2O_3$ -SiC-SiO<sub>2</sub> ion electron microscopy), X-ray diffraction spectroscopy, and Raman spectra analysis affirm the closeness of few layers between SiO<sub>2</sub> and SiC in  $In_2O_3$ -SiC,  $MnO_2$ -SiC, and  $CuO_2$ -SiC. Among the electrochemical impedance spectra curves of the nanocomposites, none of the samples had a semicircle profile, which indicates the existence of a higher charge-transfer resistivity behavior between the electrolyte and the sample electrode with charge carrier and transport effects, which is related to the well-developed porous structure of synthesized composites.  $CuO_2$ -SiC-SiO<sub>2</sub> and  $MnO_2$ -SiC-SiO<sub>2</sub> showed high resistivity and a quite significant response for NH<sub>3</sub> gas at room temperature. While there was a response for NH<sub>3</sub> gas for  $In_2O_3$ -SiC-SiO<sub>2</sub>, the sensor showed a low response for the gas. From the sensing test, correspondences between the chemical



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structure of the sensor and the molecular structure of the gases have been found. The surface reactions between the sensor surface and the gas with a pore structure, along with the electron receiver/donor phase are observed from the results of gas sensor tests, and all factors are determining the precise state. Finally, the adsorption of  $NH_3$  molecules and the alteration of the electronic resistance of  $In_2O_3$ -SiC-SiO<sub>2</sub>,  $MnO_2$ -SiC-SiO<sub>2</sub>, and  $CuO_2$ -SiC-SiO<sub>2</sub> were presented that include various thicknesses of charge to represent which are achieved by the connection with the substrates and the particles.

# 1. INTRODUCTION

Although various applications of SiC fibers were fabricated, various significant methods need to be examined. Due to continued increase in human population, more and more industrial toxic gases—such as CO, CO<sub>2</sub>, NO, NO<sub>2</sub>, NH<sub>3</sub>, SO<sub>2</sub>, H<sub>2</sub>S, and volatile organic compounds—are discharged to the environment. Figuring out such gases is an important aspect of ensuring human health. Many sensors have been made to detect these toxic gases, including electrochemical sensors, biosensors, surface acoustic wave sensors, and immunosensors, and among them, chemiresistive sensors have been developed as one of the most common established sensors due to their simplicity, rapid response, and low-cost of procurement.<sup>1-5</sup> The adsorption capacity of NH<sub>3</sub> increased while the NH<sub>3</sub> and O2 mixture increased. To explain the sensing mechanism of unfunctionalized SiC-based composites, synergistic adsorption was used, where abundant unoccupied active sites can capture more gases. It could lead to higher adsorption capacity compared to the single components.

Sensing the toxic and harmful gases from the environment has received extensive attention in recent years.<sup>6-13</sup> NH<sub>3</sub> and NO<sub>2</sub> are typical environmental pollution gases, and the

effective detection of such gases is becoming increasingly urgent.<sup>14–18</sup> SiC-based composites have recently been developed as an effective candidate for next decade roomtemperature NH<sub>3</sub> gas sensors. However, the surface-active sites of two-dimensional porous composites are mainly concentrated at the edge of lamella, while the content of active sites on the base plane is reduced,<sup>19–21</sup> thus resulting in low NH<sub>3</sub> sensing response at room temperature. SiC-based gas sensors also have the problems of poor recovery ability, poor selectivity, and stability at room temperature. Therefore, the NH<sub>3</sub> sensing performance of SiC-based gas sensors at room temperature is still far from the theoretical value and practical applicability. High-temperature assistance has proven to be an effective method for improving the NH<sub>3</sub> sensing performance

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of SiC fiber-based composites.<sup>22-27</sup> Some reports have shown that high-temperature assistance could substantially improve the recovery speed of metal oxide-SiC-based NH<sub>3</sub> gas sensors.<sup>28</sup> The results of a mechanism study showed that high-temperature assistance and polymer base could change the number of surface-active centers,<sup>29–33</sup> which was the key to improving the gas sensing performance of semiconductors at high temperature and polymer-based affinity.<sup>34</sup> However, to recombine in pure metal oxides, implemented temperature is an easy step, which results in a limited number of NH4<sup>+</sup> and O<sub>2</sub><sup>-</sup> and contributes to the gas sensing reaction. Therefore, the gas sensing performance of pure metal oxides is still unsatisfactory, even under the condition of low-temperature assistance. To solve these problems, the porosity of the base material is important for increasing the gas sensing effect. Moreover, semiconductor-based heterojunctions have been modified to sense traditional metal oxide gas sensing materials on the surface of SiO<sub>2</sub>, which is an attractive method for improving its high-temperature gas sensing performance.<sup>35–38</sup> On the one hand, the large specific surface characteristics of 2D In2O3-SiC-SiO2, MnO2-SiC-SiO2, and CuO2-SiC-SiO<sub>2</sub> nanocomposites provide significant supports for oxide nanocrystals and improve the use of sensitive bodies. Moreover, it is assumed that the recombination of electrons and holes could be prevented to a certain extent through the effect of the built-in electric field in heterojunctions. Studies have shown that the surface fabrication of SiO<sub>2</sub> can improve the response and stability of pure metal oxides active materials in the detection of NH<sub>3</sub> at high temperatures.<sup>39-</sup>

However, overall, there are still few studies examining the room-temperature gas sensing heterojunctions under hightemperature assistance. The relationship between the surface/ interface structure of heterojunctions and their gas adsorption activity/interface charge transfer behavior to this point is still unclear. The high-temperature gas sensing mechanism also needs to be studied. In fact, because of the weak interface interaction and mismatch of energy band structure, it is often hard to form effective heterojunctions between SiO<sub>2</sub> and metal oxides,<sup>44-46</sup> which makes it hard to achieve significant separation of hole-electron carriers at the interface. Therefore, the charge signal of the gas sensing reaction cannot be efficiently converted into resistance signal, which prevents any further development of the room-temperature gas sensing performance of In2O3-SiC-SiO2, MnO2-SiC-SiO2, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub> metal oxide heterojunctions.

# 2. EXPERIMENTAL SECTION

**2.1. Materials.** Copper(II) sulfate pentahydrate (CuSO<sub>4</sub>·  $SH_2O$ ) has been obtained from Duksan Pure Chemicals Co. Ltd., Korea. Manganese(II) chloride tetrahydrate MnCl<sub>2</sub>· $4H_2O$  and indium(III) oxide In<sub>2</sub>O<sub>3</sub> have been purchased from Sigma-Aldrich Co. Ltd. Triblock copolymer Pluronic F-127, hydrochloric acid (99%) HCl, tetraethyl orthosilicate (TEOS), ethanol 98%, and SiC fiber have also been purchased. Commercial grade chemicals were purchased and used without further purification.

**2.2.** Synthesis of Mesoporous  $SiO_2$ . To begin, 1.1 g of triblock copolymer Pluronic F-127 was consolidated to 15 mL of deionized water containing 2 M HCl at 40 °C. Then, it is required to mix until the copolymer was completely broken down. After that, 3.20 g of TEOS was drowned and blended at 40 °C for 12.5 h. The mixture was transferred to a fixed holder and warmed to 100 °C in a dry oven for 20 h. Then, it was

sifted and washed with water and ethanol. In the next step, it is needed to dry at 65  $^{\circ}$ C for a short amount of time. Finally, the copolymer was cleared by calcination in air at 550  $^{\circ}$ C for 3 h.

2.3. Synthesis of In2O3-SiC-SiO2, MnO2-SiC-SiO2, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub>. First, three different solutions have been prepared with deionized (DI) water, which contain Cu, In, and Mn containing precursor. 1 mol of copper(II) sulfate pentahydrate has been stirred with 40 mL of DI water. Next, 1.0 g of SiC powder has been treated with 20 mL of ethanol. Then, both the solutions have been mixed via stirring. The temperature condition was 40-60 °C. We followed this condition until the water vaporized. This paste has been marked as solution A. Then, we added mesoporous SiO<sub>2</sub> in this paste. The previously prepared 1.0 g of the SiO<sub>2</sub> sample was supposed to be treated with 40 mL of ethanol and 60 mL of DI water. Then, this solution needed to have paste solution A added to it. After stirring for 30 min, the solution was transferred in 100 mL Teflon to a stainless-steel autoclave machine, and the hydrothermal process was followed for 5 h at 120 °C. Next, the solution was washed with DI water several times and dried at 60 °C for 3 h. Then, it had to be calcined at 600 °C for 3 h. The collected sample was marked as CuO<sub>2</sub>-SiC-SiO<sub>2</sub>. The same experimental conditions have been applied to prepare the In2O3-SiC-SiO2 and MnO2-SiC- $SiO_2$  samples, where the manganese(II) chloride tetrahydrate and indium(III) oxide amounts were 1 mol.

2.4. Characterization. The structural morphology of crystals of the product were analyzed by powder X-ray powder diffraction (XRD; Rigaku, X-beam Diffractometer) with Cu K $\alpha$ radiation ( $\lambda$  = 1.5406 A) at 40 kV and 30 mA over a 2 $\theta$  range of (20-70)°. The prepared sample powder for XRD analysis was acquired using the General Structure Analysis System (GSAS, A.C. Larson and R.B. Von Dreele, Los Alamos National Laboratory). The organization of the as-integrated product was investigated using vitality dispersive X-beam spectrometry (JSM-76710F, JEOL, Tokyo, Japan). Filtering electron microscopy (SEM JOEL 6701, JEOL Ltd, Tokyo, Japan) has been used to clarify the morphology of the images. The size and the morphology of the high-resolution transm  $In_2O_3$ -SiC-SiO<sub>2</sub> ion electron microscopy (TEM) were examined by using transmission electron microscopy (TEM; JSM-76710F, JEOL, Tokyo, Japan) at a 300 kV excitation voltage. Electrochemical impedance spectra (EIS, using an electrochemical analyzer, Zahner, Germany) has been used to carry out the resistivity of the sample. Nitrogen adsorption/ desorption isotherms were analyzed by Micro Active for ASAP 2460, and the pore size distribution was figured out according to the Barrett-Joyner-Halenda method. X-ray photoelectron spectroscopy analysis was performed using electron spectrometry (WITec alpha 300 arrangement). The current-voltage (I-V) were recorded using a PG201 (Potentiostat, Galvanostat, VoltaLab, Radiometer, Copenhagen) in a protected and clean box at RT.

**2.5. Electrical Performance Test.** Cyclic voltammetry (CV) and estimations were performed under a two-electrode electrochemical arrangement to examine the current and voltage profiles, and this arrangement used  $In_2O_3$ -SiC-SiO<sub>2</sub>,  $MnO_2$ -SiC-SiO<sub>2</sub>, and  $CuO_2$ -SiC-SiO<sub>2</sub> as working electrodes. Here, a working electrode has been used as a cathode and anode electrode. The working electrode is  $2.5 \times 2.5$  cm<sup>2</sup>, which was coated on nickel foam. For this measurement, the sample electrode was placed in a tube-type chamber to measure the gas sensor capacity, and at this time, ammonia gas sensing

performance was measured under conditions with constant temperature and humidity.

## 3. RESULTS AND DISCUSSION

XRD patterns show the crystal phase information of the different samples, which are shown in Figure 1. It can be seen



Figure 1. XRD patterns of  $In_2O_3$ -SiC-SiO<sub>2</sub>,  $Cu_2O$ -SiC-SiO<sub>2</sub>, and  $MnO_2$ -SiC-SiO<sub>2</sub>.

that  $In_2O_3$ -SiC-SiO<sub>2</sub>,  $MnO_2$ -SiC-SiO<sub>2</sub>, and  $CuO_2$ -SiC-SiO<sub>2</sub> samples show typical diffraction peaks of  $In_2O_3$ -SiC,  $MnO_2$ -SiC,  $CuO_2$ -SiC, and SiO<sub>2</sub>. The XRD patterns of  $In_2O_3$ -SiC-SiO<sub>2</sub>,  $MnO_2$ -SiC-SiO<sub>2</sub>, and  $CuO_2$ -SiC-SiO<sub>2</sub> show mixed phases of hexagonal  $In_2O_3$ -SiC,  $MnO_2$ 

 $CuO_2$ -SiC, and SiO<sub>2</sub>.<sup>47,48</sup> The diffraction peaks of hexagonal In<sub>2</sub>O<sub>3</sub>-SiC, MnO<sub>2</sub>-SiC, and CuO<sub>2</sub>-SiC are relatively weak due to the two-dimensional lamellar structure, and the strong diffraction peak of SiO<sub>2</sub> nanocrystals masks it to a great extent. Based on the XRD data, three types of samples as an active material form a distinct crystal structure, which is believed to have an effect of maximizing the sensor effect. In particular, the distinct SiO<sub>2</sub> crystal structure is believed to increase the ammonia reactivity.

Figure 2 shows the structural morphology and elemental information of the three samples. As can be seen from Figure 2a-c, there is a typical two-dimensional lamellar structure, and the nanocomposites are intertwined to form a rodlike structure. It can be seen that metal oxides and SiO<sub>2</sub> compounds are uniformly distributed around rodlike SiC. These results show that rodlike SiC can play an important role as a substrate material, and that it may play a central role in localization and reactivity with metal oxides and SiO<sub>2</sub> compounds in the formation of nanocomposites. This can clearly be seen from Figure 2a-c. The energy-dispersive X-ray spectroscopy (EDS) spectra show the presence of In, Cu, Mn, Si, C, and O in the In<sub>2</sub>O<sub>3</sub>-SiC-SiO<sub>2</sub>, MnO<sub>2</sub>-SiC-SiO<sub>2</sub>, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub>. The higher amount of O indicates high oxidation in composites.

It can clearly be seen in the TEM images in Figure 3a,c,e that the nanocomposites are well aggregated. The spacing of the (222,002,110) crystal facets of typical hexagonal  $In_2O_3$ -SiC-SiO<sub>2</sub>, MnO<sub>2</sub>-SiC-SiO<sub>2</sub>, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub>, respectively, can clearly be shown in the high-resolution TEM (HRTEM) image, as shown in Figure 3b,d,f. TEM images clearly show that excellent heterogeneous interface contact is formed between  $In_2O_3$ -SiC, MnO<sub>2</sub>-SiC, CuO<sub>2</sub>-SiC nanocomposites, and SiO<sub>2</sub> nanocrystals, as can be found in Figure 3a,c,e, which is a key aspect of the effective charge transfer



Figure 2. SEM images and EDS of (a) In<sub>2</sub>O<sub>3</sub>-SiC-SiO<sub>2</sub>, (b) Cu<sub>2</sub>O-SiC-SiO<sub>2</sub>, and (c) MnO<sub>2</sub>-SiC-SiO<sub>2</sub>.



Figure 3. TEM and HRTEM images: (a,b)  $In_2O_3-SiC-SiO_2$ , (c,d)  $CuO_2-SiC-SiO_2$ , and (e,f)  $MnO_2-SiC-SiO_2$ .

between heterojunctions. Furthermore, using a simple selfassembled method, we have synthesized  $In_2O_3$ -SiC-SiO<sub>2</sub>,  $MnO_2$ -SiC-SiO<sub>2</sub>, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub> heterojunction materials with clear crystal facet exposure, which provides an ideal model for studying the structure–activity relationship between the surface/interface and the properties of heterojunction materials.

Furthermore, the Raman spectra of the  $In_2O_3$ -SiC-SiO<sub>2</sub>,  $MnO_2$ -SiC-SiO<sub>2</sub>, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub> nanocomposites with various G contents, as shown in Figure 4, reveal that



**Figure 4.** Raman spectra of  $In_2O_3$ -SiC-SiO<sub>2</sub>,  $MnO_2$ -SiC-SiO<sub>2</sub>, and  $CuO_2$ -SiC-SiO<sub>2</sub>.

SiC had a spectral peak of about 1354 cm<sup>-1</sup>, which was attributable to the D band associated with the nano-auxiliary deformities of carbon spaces and generated by interactions in the carbon plane. The crests at 1569, 1577, and 1585 cm<sup>-1</sup> are related to the G band, which results from the main distribution of the phonon  $E_{2g}$  of the C sp2 molecules.<sup>49,50</sup> As a rule, a Gband configuration involves a charge exchange, which produces a range of graphene electrical properties. Shifts in the red G band can be seen with a greater measure of G, which declared the arrangement of new areas of carbon during heat treatment. Meanwhile, the decreased strength of the D/G ratio confirmed a slight low peak in G, which then made greater carbon crystallinity by constantly increasing the G. For the compound In2O3-SiC-SiO2, MnO2-SiC-SiO2, and CuO2-SiC-SiO2, the groups D and G expanded and decreased significantly. The downward change in the G band has been ascribed to the incomplete interaction of the metal particles, thus suggesting that the nanocrystals bond weakly to the carbon. These results suggest that the nanocomposite represented a channel for transporters and the prevailing electrical and charge-sharing properties.51,52

Figure 5 shows the EIS spectroscopy results of the  $In_2O_3$ -SiC-SiO<sub>2</sub>, MnO<sub>2</sub>-SiC-SiO<sub>2</sub>, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub> samples.



Figure 5. Nyquist plots of  $In_2O_3$ -SiC-SiO<sub>2</sub>, MnO<sub>2</sub>-SiC-SiO<sub>2</sub>, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub> samples.

It can clearly be seen that those samples have high resistivity. Compared to In2O3-SiC-SiO2 and CuO2-SiC-SiO2 samples, the MnO<sub>2</sub>-SiC-SiO<sub>2</sub> composite has shown low resistivity. Thus, the MnO<sub>2</sub>-SiC-SiO<sub>2</sub> sample has large electron-transfer carrier. Among the EIS curves of the nanocomposites, none of the samples had a semicircle profile, which indicates the existence of a higher charge-transfer resistance between the sample electrode and the electrolyte with charge carrier and transport effects. Further analysis has been conducted to show the porous structure of the synthesized composites. From Figure 6a, it is apparent that the MnO<sub>2</sub>-SiC-SiO<sub>2</sub> sample has a high nitrogen absorptiondesorption isotherm. The In<sub>2</sub>O<sub>3</sub>-SiC-SiO<sub>2</sub> sample shows H1type adsorption isothermal curves, and this hysteresis curve does not show a percolation effect due to the metastable pore fluid and the narrow pore-size distribution. On the other hand, CuO<sub>2</sub>-SiC-SiO<sub>2</sub> and MnO<sub>2</sub>-SiC-SiO<sub>2</sub> show H2-type adsorption isothermal curves. It is considered that their hysteresis curves show the pore fluid and percolation effect



Figure 6. (a) Nitrogen adsorption–desorption isotherms and (b) pore distribution of  $In_2O_3$ –SiC–SiO<sub>2</sub>,  $MnO_2$ –SiC–SiO<sub>2</sub>, and  $CuO_2$ –SiC–SiO<sub>2</sub> sample.

due to the presence of both metastable states and broad poresize distribution. However,  $CuO_2-SiC-SiO_2$  and  $In_2O_3 SiC-SiO_2$  composites have a small loop between the 0.2 and 0.8 cm<sup>2</sup>/g region, whereas the MnO<sub>2</sub>-SiC-SiO<sub>2</sub> sample shows bigger hysteresis loops than the others. Thus, the MnO<sub>2</sub>-SiC-SiO<sub>2</sub> sample has a small pore diameter, and the sample will show a larger light absorption effect. In the pore size distribution results, it can be seen that the MnO<sub>2</sub>-SiC-SiO<sub>2</sub> sample contains larger pore levels than the  $In_2O_3-SiC-SiO_2$ and  $CuO_2-SiC-SiO_2$  samples, as shown in Figure 6b. Due to the large pore volume, the MnO<sub>2</sub>-SiC-SiO<sub>2</sub> sample has a large active surface area, which will be efficient for gas sensing.

Figure 7a-c, respectively, shows the X-ray photoelectron spectroscopy (XPS) results of the  $In_2O_3$ -SiC-SiO<sub>2</sub>, MnO<sub>2</sub>-SiC-SiO<sub>2</sub>, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub> composites. XPS spectroscopy has been conducted to confirm the binding energy region of each element. It is also used to declare the presence of each element in the composite material. Figure 7a shows the binding energy survey results of the  $In_2O_3$ -SiC-SiO<sub>2</sub> sample. The binding energy of In is 463.5 eV, which is declared to be the In 3d orbital region. The presences of Si, C, and O are also observed in the  $In_2O_3$ -SiC-SiO<sub>2</sub> sample, as confirmed by the

binding energy regions at 151.89, 298.53, and 584.8 eV, respectively. In Figure 7b, the orbital positions are apparent at the Si 2p, C 1s, O 1s, and Cu 2p numbers with the binding energy regions of 101.93, 259.65, 598.41, and 998.53 eV, respectively. The binding energy region for the  $MnO_2$ -SiC-SiO<sub>2</sub> sample is shown in Figure 7c. The binding energy for Mn is 638.98 eV, and the orbital region is Mn 2p. For O, the Si 2p spectra are shown with a binding energy region of 101.59 eV. Moreover, the C 1s and O1s orbital numbers are shown in the 298.53 and 559.46 eV binding energy regions.

Figure 8 shows the ammonia gas sensing result of the In<sub>2</sub>O<sub>3</sub>-SiC-SiO<sub>2</sub> composite. From the CV data, it is apparent that the In<sub>2</sub>O<sub>3</sub>-SiC-SiO<sub>2</sub> sample exhibits constant current density results for 6 and 24 h treatment, and the current amount is around 0.8 mA cm<sup>-2</sup>. On the other hand, the Cutreated sample has a low current density result with 6 and 24 h treatment, as shown in Figure 9. Thus, further experimentation has not been applied for the CuO<sub>2</sub>-SiC-SiO<sub>2</sub> sample. Finally, ammonia gas sensing has been tested on the  $MnO_2-SiC-SiO_2$ sample with different time conditions. The Mn-treated sample showed good current density results compared to the Cutreated sample. In Figure 10, it can be seen that the current density amount is  $1.94 \times 10^{-4}$  mA cm<sup>-2</sup>, which is larger than that of the CuO<sub>2</sub>-SiC-SiO<sub>2</sub> sample. Thus, it is apparent that the In<sub>2</sub>O<sub>3</sub>-SiC-SiO<sub>2</sub> sample will show a high sensing result. For further confirmation of the electrical response, resistance values have been calculated for In2O3-SiC-SiO2, MnO2-SiC-SiO<sub>2</sub>, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub> composites. Figure 11 shows the resistance effect of the In2O3-SiC-SiO2 sample for ammonia sensing as a resistance value with increasing time. The figure shows that the resistance value gradually decreases with time. It is believed that ammonia decomposes with increasing time or that the ammonia adsorption properties decrease on the sample surface.

Figure 11 also shows the resistance effect of CuO<sub>2</sub>-SiC-SiO<sub>2</sub> on ammonia sensing as a resistance value for a function of time. It can be seen that the resistance value gradually increases with increasing time. It appears that the resistance value increases due to the accumulation of the adsorption amount of ammonia with increasing time, and it is believed that the ammonia adsorption reactivity on the sample surface is increased. In addition, due to the increase in porosity, many ammonia molecules are continuously filled in the pores, and the adsorption amount is increasing as a result. The resistance effect of MnO<sub>2</sub>-SiC-SiO<sub>2</sub> on ammonia sensing as a resistance value as a function of time is presented in Figure 11. At the initial step, the resistance value is shown to increase slowly and gradually. However, with increasing time, the resistance value shows a tendency to rapidly increase. This indicates that the adsorption amount of ammonia is not very large in the initial time, and the adsorption amount shows a rapid increase with increasing time, which is correlated with the increase in the resistance value. In addition, due to the increase in porosity, many ammonia molecules are continuously stacked in the pores, and the adsorption amount is therefore increasing. This is inefficient for initial sensing effects, but devices requiring long-term sensing effects are expected to show excellent efficiency. Thus, the CuO<sub>2</sub>-SiC-SiO<sub>2</sub> sample will show higher sensing response than other synthesized samples. These redox peaks in the curve could be due to the immediate electrontransfer behaviors of In2O3-SiC-SiO2, MnO2-SiC-SiO2, and  $CuO_2$ -SiC-SiO<sub>2</sub>. Here, all samples show small redox curves, and  $CuO_2$ -SiC-SiO<sub>2</sub> composites show smaller redox curves



Figure 7. XPS spectra of (a) In<sub>2</sub>O<sub>3</sub>-SiC-SiO<sub>2</sub>, (b) CuO<sub>2</sub>-SiC-SiO<sub>2</sub>, and (c) MnO<sub>2</sub>-SiC-SiO<sub>2</sub> survey.





Figure 8. Ammonia sensing effects with  $In_2O_3-SiC-SiO_2$  as active material for 6-24 h.

than  $In_2O_3$ -SiC-SiO<sub>2</sub>, and  $MnO_2$ -SiC-SiO<sub>2</sub> thus indicating high resistivity. The presence of NH<sub>3</sub> gas has prevented hole to electron generation.<sup>53</sup> The sensing mechanism also rely on the charge-transfer process based on oxidation and reducing the behavior of the target gas, and also regarding the atomic surface of the nanostructures.<sup>54,55</sup> In the present study, bare  $In_2O_3$ -SiC,  $MnO_2$ -SiC, and  $CuO_2$ -SiC are amalgamated with SiO<sub>2</sub>, which can demonstrate a reduction in electrical noise and a constant enhancement in current conduction. Figure 9. Ammonia sensing effects with  $Cu_2O-SiC-SiO_2$  as active material for 6-24 h.

However, there is a low electron generation presence of  $NH_3$ , thus leading to a low redox reaction.<sup>56</sup>

To assess the concentration-dependent electrochemical reactions of  $In_2O_3-SiC-SiO_2$ ,  $MnO_2-SiC-SiO_2$ , and  $CuO_2-SiC-SiO_2$  samples under different concentrations of NH<sub>3</sub> gas, as shown in Figure 12, we recorded it at a connected potential of 0.2 V. A stepwise increment in current density was observed upon the expansion of ammonia gas at increasing concentrations. The  $In_2O_3-SiC-SiO_2$  sample exhibited a high



Figure 10. Ammonia sensing effects with  $MnO_2$ -SiC-SiO<sub>2</sub> as active material for 3-12 h.



Figure 11. Resistance values with time condition of  $In_2O_3$ -SiC-SiO<sub>2</sub>, Cu<sub>2</sub>O-SiC-SiO<sub>2</sub>, and MnO<sub>2</sub>-SiC-SiO<sub>2</sub> with ammonia.



Figure 12. Current variation under different  $NH_3$  concentrations (0.1–1.0 mL/s).

current value in the whole range along with an increase in concentrations, and it maintained a constant current value. The  $MnO_2-SiC-SiO_2$  sample showed a high current value from the initial concentration value, and as the time and concentration increased, the current value showed a tendency to slowly decrease. The  $CuO_2-SiC-SiO_2$  sample maintained a low current value throughout the whole range, and it showed a tendency to maintain a constant current value along with increasing time and concentration. Among the three types of samples,  $CuO_2-SiC-SiO_2$  showed a stable sensing effect, while  $MnO_2-SiC-SiO_2$  showed a gradually stable sensing

effect. Therefore, MnO2-SiC-SiO2 is expected to show excellent characteristics when measuring sensor effects for a long period of time. Interestingly, we observed an enhancement in the sensing effect within the greatness of the standard deviation in moving toward higher ammonia concentrations, which may correspond to an increase in the sensing effect within the inconstancy of detection as the samples approached the upper concentration limit of ammonia. The sensor effect has multiple variables, and it is correlated with the conductivity of the constituent element and the compound, the adsorption characteristics due to porosity, and the gas reactivity. Here, one can see that the response range corresponds to the concentration extent. After observing this proportion that can effortlessly reach to this choice that able to degree of ammonia gas sensing test with this sensor for subjective and quantitative analysis.<sup>5</sup>

Furthermore, Scheme 1 depicts a mechanism showing the working material preparation and their working principal. It is

Scheme 1. NH<sub>3</sub> Adsorption and Chemisorption Mechanisms

and Design of Electrodes for the Electrical Performance



apparent that the active materials have been coated on the surface of nickel foam. Generally, an active material produced hole-electron and transfer electron from the valance band to the conduction band. As the samples have a mesoporous structure and as metallic In<sub>2</sub>O<sub>3</sub>-SiC, MnO<sub>2</sub>-SiC, and CuO<sub>2</sub>-SiC have been added on the surface of mesoporous  $SiO_{2}$ , samples have active surface area containing pores. Although the samples also have semiconductive behavior, the presence of NH4<sup>+</sup> prevented electron production by blocking active pores and increasing the resistive active surface. Thus, these samples will show high resistivity at room temperature and high temperature. This experimental condition declared the gas sensing efficiency of the sample NH<sub>3</sub>.<sup>53,57,58</sup> The fibrous base materials used so far have good conductivity and durability, but they lack pore and gas affinity.<sup>59,60</sup> Therefore, in this study, fibrous SiCs with good properties were used to form complex SiO<sub>2</sub> with porosity and a metal oxide with gas affinity.

#### 4. CONCLUSIONS

In summary,  $In_2O_3-SiC-SiO_2$ ,  $MnO_2-SiC-SiO_2$ , and  $CuO_2-SiC-SiO_2$  composites were prepared through a selfassembled method on the SiC fiber surface. The roomtemperature NH<sub>3</sub> sensing properties of the heterojunctions were studied under low-temperature assistance. It was found that CuO<sub>2</sub>-SiC-SiO<sub>2</sub> and MnO<sub>2</sub>-SiC-SiO<sub>2</sub> heterojunctions showed a significantly higher sensing response than In<sub>2</sub>O<sub>3</sub>-SiC-SiO<sub>2</sub> composites, thus suggesting a strong synergistic effect. The high gas sensing performance of the heterojunction can also be related to adsorption capacity with pore structure and porosity for NH<sub>3</sub> gas. Current density was obtained from I-V curves and EIS spectra to clarify the gas sensing behavior. The gas sensing test found a correlation between the molecule structure, the electron donor/acceptor phase of the gases, and the chemical structure of the sensor. The estimations have been presented to include various thicknesses of charge to represent the adsorption of NH<sub>3</sub> molecules, and the alteration of the electronic resistance of In2O3-SiC-SiO2, MnO2-SiC-SiO<sub>2</sub>, and CuO<sub>2</sub>-SiC-SiO<sub>2</sub> was achieved by the connection with the substrates and the particles. The results provide a new model through which to improve the room-temperatureassisted gas sensing performance of SiC-based gas porous sensors through the construction of porous heterojunctions, which will essentially increase the development of NH<sub>3</sub> gas sensors.

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#### Notes

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