Direct Organization of Morphology-Controllable Mesoporous SnO₂ Using Amphiphilic Graft Copolymer for Gas-Sensing Applications

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ABSTRACT: A simple and flexible strategy for controlled synthesis of mesoporous metal oxide films using an amphiphilic graft copolymer as sacrificial template is presented and the effectiveness of this approach for gas-sensing applications is reported. The amphiphilic graft copolymer poly(vinyl chloride)-g-poly(oxyethylene methacrylate) (PVC-g-POEM) is used as a sacrificial template for the direct synthesis of mesoporous SnO₂. The graft copolymer self-assembly is shown to enable good control over the morphology of the resulting SnO₂ layer. Using this approach, mesoporous SnO₂ based sensors with varied porosity are fabricated in situ on a microheater platform. This method reduces the interfacial contact resistance between the chemically sensitive materials and the microheater, while a simple fabrication process is provided. The sensors show significantly different gas-sensing performances depending on the SnO₂ porosity, with the highly mesoporous SnO₂ sensor exhibiting high sensitivity, low detection limit, and fast response and recovery toward hydrogen gas. This printable solution-based method can be used reproducibly to fabricate a variety of mesoporous metal oxide layers with tunable morphologies on various substrates for high-performance applications.

KEYWORDS: amphiphilic graft copolymer, sol–gel, mesoporous materials, SnO₂ microheater, gas sensor

INTRODUCTION

Porous materials are attractive for many applications, such as batteries,¹−³ supercapacitors,⁴−⁷ and gas separators,⁸−¹¹ owing to their remarkable structure and morphology, such as tunable pore size and volume, and high surface area. The ability to tailor the pore size is critical for many of these applications. The smaller the pore size, the higher the surface area for gas/ion penetration or for absorption onto active sites. However, very small pore size may inhibit molecular diffusion or surface reaction, resulting from bottleneck phenomena of the large flux of molecules or deficiencies in active sites. Among the porous materials, mesoporous materials with 2−50 nm pore size offer excellent properties, including high surface area and proper pore size, leading to high performance in many applications.¹²

Mesoporous metal oxides have been widely pursued as promising candidates for gas-sensing applications due to their ease of fabrication, good chemical/thermal stability, and high sensitivity. On the basis of gas absorption and diffusion mechanisms, the architectures of mesoporous metal oxides significantly promote the mass transfer of gaseous reactants toward active sites through rapid gas diffusion enabled by their suitable pore size with extremely high surface area, thus resulting in high sensitivity. Various mesoporous metal oxide nanostructures have been developed, such as hierarchical nanostructures,¹³,¹⁴ nanotubes,¹⁵,¹⁶ hollow spheres,¹⁷,¹⁸ or nanowires,¹⁹,²⁰ to achieve rapid response and fast electron transport through interconnected, porous, two- or three-dimensional structures, allowing excellent responses for gas-sensing applications.

However, metal oxides need to be heated to elevated temperatures (250−500 °C) to activate or enhance their sensing performance. Giving that many sensing applications require low-power consumptions and small device footprints,²¹,²² it remains a challenge to effectively integrate presynthesized mesoporous nanomaterials with tailored porosity onto low-power microheaters. The typical integration
method is drop-casting the nanomaterials dispersed in solution onto the microheater chip, a process that is hard to control, resulting in the variable structure of the deposited materials, a reduction of active surface area due to aggregation, and poor interfacial contact with the sensing electrodes. There have been recent reports on new strategies for direct mesoporous metal oxide deposition, such as hydrothermal growths of chemically sensitive materials, chemical vapor deposition methods, and in situ polymerization techniques of sensing materials. Although these novel approaches show outstanding gas-sensing performances, they suffer from a number of limitations, such as complicated control processes during fabrication, difficulty in scaling up for commercialization, and the need for special instrumentation or environmental conditions. Therefore, it is crucial to develop a simple and scalable strategy for the synthesis of well-organized mesoporous materials with controllable morphology on suitable substrates for different applications. In contrast to previous methods, the use of a sacrificial template to fabricate mesoporous materials offers an easier approach toward control over the morphology and pore size of mesoporous materials.

Here, we report a novel technique based on the direct formation of morphology-controllable mesoporous metal oxide layers using amphiphilic graft copolymers as a sacrificial soft template for gas-sensing applications. This new method uses a metal oxide salt as a precursor together with graft copolymer as a sacrificial soft template to generate mesoporous metal oxide layers in situ on a low-power microheater platform, which significantly reduces the interfacial contact resistance between chemically sensitive materials and a microheater with a simple fabrication process. The self-assembly of microphase-separated amphiphilic graft copolymer leads to a controllable nanostructure, pore distribution, and surface area of the active materials. Poly(vinyl chloride)-g-poly(oxyethylene methacrylate) (PVC-g-POEM) and SnO2 are chosen as representative sacrificial template and metal oxide, respectively, to demonstrate the validity of this strategy. The resulting SnO2 gas sensors with varying mesopores present significantly different gas-sensing performances, with the highly mesoporous SnO2 sensor exhibiting high sensitivity, a low detection limit, and fast response and recovery toward hydrogen gas.

**EXPERIMENTAL SECTION**

**Synthesis of PVC-g-POEM Graft Copolymer.** PVC-g-POEM graft copolymers were synthesized via an atomic transfer radical polymerization (ATRP) process, schematically shown in Figure S1a of the Supporting Information (SI). This graft copolymer was chosen due to its relatively simple and low-cost synthesis compared to that of other microphase-separated copolymers (i.e., block copolymers). Briefly, 6.0 g of poly(vinyl chloride) (PVC) was dissolved in 50 mL of N,N-dimethyl-2-pyrrolidone (NMP) in a 250 mL round flask and 15 g of poly(oxyethylene methacrylate) (POEM) was added to the solution. After stirring for several hours, 0.10 g of CuCl and 0.23 mL of 1,1,4,7,10,10-hexamethyltriethylenetetramine (HMTETA) were added, and the reaction was cooled down. After passing the solution through a column of activated Al2O3 to remove the catalyst, the polymer was precipitated into methanol (MeOH). The polymer was purified by repeated washing in MeOH to eliminate all residues. Finally, the PVC-g-POEM graft copolymers were dried in a vacuum oven at 50 °C for 24 h.

**Preparation of Mesoporous SnO2 Precursor Solution.** There are several crucial parameters that can control the pore size and structure of mesoporous SnO2, such as polymer concentration, amount of metal oxide precursor, and solvent composition. Two kinds of mesoporous SnO2 precursor solutions are presented here: (1) a less mesoporous SnO2 (LM-SnO2) precursor solution and (2) a highly mesoporous SnO2 (HM-SnO2) precursor solution. For LM-SnO2 precursor solution, 0.0125 g of PVC-g-POEM was completely dissolved in 2.5 mL of THF solvent, and 0.2 g of SnCl2 was added to as-prepared polymer solution, with stirring for 3–4 h to homogenize the solution. For HM-SnO2 precursor solution, 0.025 g of PVC-g-

**Figure 1.** (a) FT-IR spectra of PVC, POEM monomers, and PVC-g-POEM graft copolymer. (b) 1H NMR spectrum of PVC-g-POEM graft copolymer. (c) DSC analysis of PVC-g-POEM graft copolymer. (d) TGA curve of PVC-g-POEM graft copolymer.
POEM was fully dissolved in 5 mL of THF solvent. Then, 0.2 g of SnCl₂ was added as-prepared polymer solution, and after 10 min, 0.1 mL of deionized (DI) water was added to the precursor solution. This solution was stirred for 3–4 h to make a homogeneous solution.

Materials Characterization. To confirm the successful polymerization of PVC-g-POEM graft copolymer, the synthesized graft copolymer was characterized via FT-IR spectra (Excalibur Series FT-IR, DIGLAB Co.), ¹H NMR measurement with a 600-MHz high-resolution NMR spectrometer (AVANCE 600 MHz FT-NMR, Bruker), and differential scanning calorimetry (DSC) measurement (DSC 2920, TA Instruments, Inc.) by second sequence from 80 to 200 °C at a heating rate of 20 °C/min under an air atmosphere. Thermogravimetric analysis (TGA, Mettler Toledo TGA/SDTA 851e, Columbus, OH) was conducted under an air atmosphere to determine the thermal stability of PVC-g-POEM graft copolymer. The morphology and crystal structure of the mesoporous SnO₂ layers and Mesoporous SnO₂. This solution was stirred for 3 h to make a homogeneous solution. The carbonyl (C=O) vibration band in the PVC-g-POEM graft copolymer is investigated by FT-IR measurement software, was used to collect data from the source-meter and gas delivery system, including gas concentrations and flow rates.

The ¹H NMR analysis is conducted to determine the actual weight ratio of PVC and POEM in PVC-g-POEM graft copolymer. As shown in Figure 1b, the strong signal at 4.6 ppm can be assigned to the protons in the CHCl groups of PVC, whereas the signals at 3.6 and 3.4 ppm are attributed to the protons in the CH₂CH₂O and CH₃ groups of POEM, respectively. The actual grafting ratio is calculated by integrating the peak area assigned to each group. The weight ratio of PVC to POEM in PVC-g-POEM graft copolymer is found to be approximately 2:1. The amphiphilic PVC-g-POEM graft copolymer possesses glassy PVC main chains and rubbery POEM side chains simultaneously, which can produce well-developed microphase separation. Due to these characteristics, the PVC-g-POEM graft copolymer shows two different glass transition temperatures (Tg) in Figure 1c. The Tg at 83 °C originates from the mobility of PVC main chains, whereas Tg at −58 °C indicates from the mobility of POEM side chains. Moreover, the absence of an endothermic melting temperature (Tm) amorphous state of the PVC-g-POEM graft copolymer.

This amphiphilic PVC-g-POEM graft copolymer with well-developed microphase separation is a good sacrificial template with which to fabricate mesoporous structure metal oxides. To be used as a sacrificial template, the thermal stability of PVC-g-POEM graft copolymer is investigated by TGA measurement in Figure 1d. The PVC-g-POEM graft copolymer is thermally stable up to 250 °C without any noticeable weight loss change. A two-step decomposition pattern is observed at around 300 and 400 °C. Less thermally stable groups (i.e., chlorine) in PVC-g-POEM graft copolymer start to decompose at 300 °C, whereas more thermally stable groups (i.e., hydrocarbons) in PVC-g-POEM graft copolymer start to decompose at 400 °C. At 500 °C, PVC-g-POEM graft copolymer is decomposed completely. These results reveal that PVC-g-POEM graft copolymer can be used as a sacrificial template that requires a heating temperature of ≥500 °C to remove all the polymer.

Transmission electron microscopy is employed to understand the effect of various components in the sol–gel process on the resulting morphology. Figure 2a shows the TEM image of pure PVC-g-POEM graft copolymer in THF solvent. The THF solvent has a neutral nature, and thus, it is able to dissolve both hydrophobic PVC and hydrophilic POEM. In this way, the PVC-g-POEM graft copolymer develops evident microphase separation in THF solvent. The dark regions represent the PVC domain and the bright regions represent the POEM domain, due to the higher electron density of chlorine in PVC than in POEM. The morphology of PVC-g-POEM graft copolymer can be controlled by addition of H₂O on the basis of polymer–solvent interactions. Since H₂O is a strongly hydrophilic polar solvent that has a good interaction with the POEM domain, it leads to well-developed POEM domains in PVC-g-POEM graft copolymer, as shown in Figure 2b. The PVC-g-POEM/H₂O/THF mixture not only produces continuous interconnectivity between POEM domains but it also enhances the solubility of the hydrophilic SnCl₂ precursor. These synergistic effects help to fabricate a highly well-developed mesoporous SnO₂ structure.

Based on the above characterizations, the amphiphilic PVC-g-POEM graft copolymer is used as a soft template through sol–gel process for the synthesis of the mesoporous SnO₂ layer. Figure 2c shows the morphology of PVC-g-POEM/SnCl₂/THF mixture without water, which possesses less-
ordered microphase separation, attributable to the low solubility of SnCl$_2$ in THF solvent. The dissolution of hydrophilic SnCl$_2$ in PVC-$g$-POEM graft copolymer with THF solution needs several hours to adhere to the POEM domain and to self-assemble. However, when SnCl$_2$ is added in PVC-$g$-POEM/H$_2$O/THF solution, SnCl$_2$ dissolves immediately due to the presence of H$_2$O. Thus, the PVC-$g$-POEM/H$_2$O/SnCl$_2$/THF exhibits well-organized microphase separation, as shown in Figure 2d, compared to PVC-$g$-POEM/SnCl$_2$/THF. These two recipes are used for the fabrication of mesoporous SnO$_2$ layers with different morphology, referred to as less mesoporous SnO$_2$ (LM-SnO$_2$) (see Figure 2c) and highly mesoporous SnO$_2$ (HM-SnO$_2$) (see Figure 2d).

The two prepared solutions are drop-cast on silicon substrates and then the substrate temperature is raised at a rate of 3 °C/min and held at 500 °C for 2 h to obtain mesoporous layers with different morphologies. The LM-SnO$_2$ and HM-SnO$_2$ layers show comparatively controlled mesoporous structures with crack-free conditions, as displayed in Figure S2 (SI) in low magnified images. Figure 3a,b presents SEM images of the LM-SnO$_2$ layer under different magnifications, where closely an aggregated SnO$_2$ layer with a less-developed mesoporous structure is observed. The pore size of LM-SnO$_2$ is around 5–10 nm, and pores are observed sparsely. In contrast, the HM-SnO$_2$ layer exhibits a well-developed mesoporous structure with 30–40 nm pore size, as shown in Figure 3c,d. Moreover, the SnO$_2$ skeleton is observed to be composed of interconnected small SnO$_2$ nanoparticles in HM-SnO$_2$, exhibiting a large density of nanopores with small diameters with high surface area, while the LM-SnO$_2$ layer shows a much denser skeleton.

To characterize the mesoporous SnO$_2$ layers using TEM, the SnO$_2$ layers are synthesized using the same deposition method directly onto TEM grids consisting of high-temperature-resistant silicon nitride ($Si_3N_4$) windows. The properties of the LM-SnO$_2$ and HM-SnO$_2$ layers, including porosity, domain size, and the lattice $d$-spacing value, are clearly detectable in TEM images (Figure 4). The TEM image of the LM-SnO$_2$ layer (Figure 4a) shows aggregated and large SnO$_2$ domains with low surface area. This is consistent with the TEM image presented in Figure 2c, showing aggregation in the microphase separation of PVC-$g$-POEM/SnCl$_2$, and with the SEM image presented in Figure 3b, showing closely aggregated SnO$_2$ domains in LM-SnO$_2$. Aggregated domains much larger than 20 nm are observed in the magnified TEM image in Figure 4b, with the inset showing the Fourier transform (FT) pattern of the selected area. The (110) plane of LM-SnO$_2$ is clearly observed, corresponding to a lattice spacing of 0.34 nm, which confirms the formation of crystalline SnO$_2$. In comparison with LM-SnO$_2$, the TEM image of HM-SnO$_2$ in Figure 4c exhibits a large number of small domains with extremely large surface area, matched with well-developed microphase separation in PVC-$g$-POEM/H$_2$O/SnCl$_2$ (Figure 2d) and a well-organized, interconnected structure in HM-SnO$_2$ (Figure 3d). In Figure 4d, the domains less than 10 nm show multiple active sites and contacts between adjacent domains, which are advantageous for gas absorption and conductive sensing path. The HM-SnO$_2$ presents a 0.34 nm lattice spacing, in accordance with the (110) plane of SnO$_2$ through the FT pattern in the selected area of the TEM image, demonstrating that LM-SnO$_2$ and HM-SnO$_2$ possess identical crystal structures.

The crystal structure of the mesoporous SnO$_2$ layer is also characterized by Raman and X-ray diffraction measurements. Figure 5a shows the Raman spectra of LM-SnO$_2$ and MH-SnO$_2$ layers, both exhibiting the SnO$_2$ characteristic peaks, located at 475, 632, and 777 cm$^{-1}$ attributed to the E$_g$ A$_{1g}$ and B$_{2g}$ vibration modes of mesoporous SnO$_2$, respectively. Furthermore, the peak found at 591 cm$^{-1}$ indicates the Sn–O surface vibration mode. Figure 5b presents the XRD spectra, showing that both LM-SnO$_2$ and HM-SnO$_2$ exhibit similar characteristic peaks corresponding to polycrystalline rutile SnO$_2$ phase (JCPDS No. 41-1445). Applying Scherer’s equation to the strongest peak (110), which is also the exposed plane from the TEM analysis, the average crystalline size of mesoporous SnO$_2$
is approximately 17 nm, consistent with the aggregated particles size observed from SEM and TEM images. The Raman spectra and XRD pattern results reveal that two types of mesoporous SnO₂ have identical crystalline phases but different morphologies.

**Gas Sensor Performance.** For gas sensor applications, heating the sensing materials to high temperature is often used to activate the sensing mechanism and enhance the response and recovery rates. To demonstrate the application of the synthesis method, a commercial low-power microheater is used, as shown in Figure S3 (SI). (The power consumption characteristics of the microheater is presented in Figure S4, SI.)

The mesoporous SnO₂ sol−gel solution is drop-cast onto the microheater at room temperature. The THF solvent evaporates because of its low boiling point, and it can help with the evaporation of water resulting from the favorable interaction between THF and water. The mesoporous SnO₂ layer is then directly generated with a gradual increase of microheater temperature up to 500 °C, and the temperature is maintained for several hours to remove the polymer template and develop SnO₂ materials. This mesoporous SnO₂ fabrication process provides good interfacial contact with sensor and chemical-sensitive materials, which can reduce the sensor resistance.

The as-prepared mesoporous SnO₂ based sensors are tested toward hydrogen gas. Hydrogen is an important industrial gas used in a number of applications, such as a replacement for fossil fuel and an alternative fuel for transportation. Since hydrogen is odorless and colorless, but flammable, it is critical
to install hydrogen gas monitoring systems that can detect potentially explosive situations. Speed and accuracy are needed to detect it in a short time, because even at a low concentration (lower explosive limit = 4%) it can lead to an explosion.

Operation temperature is one of the key factors for a gas sensor, due to its major influence on sensing performance. The response to hydrogen gas is measured at various heater temperatures to determine the optimum temperature. Figure 6a shows the effect of temperature on the sensor response, calculated as \( R_{\text{air}} / R_{\text{gas}} \), where \( R_{\text{air}} \) is the average resistance in air and \( R_{\text{gas}} \) is the average resistance during exposure to 3% \( \text{H}_2 \) in air. Both LM-SnO\(_2\) and HM-SnO\(_2\) record the highest sensitivity with the microheater at 300 °C. Consequently, this temperature is chosen as the optimum temperature, and further sensing tests are conducted at this temperature.

Figure 6b,c represents real-time resistance changes of the mesoporous SnO\(_2\) sensors to various hydrogen concentrations at microheater operating temperature of 300 °C. The HM-SnO\(_2\)-based sensor shows higher sensitivity and faster response and recovery times during exposure to various hydrogen concentrations. Regardless of hydrogen gas concentration, the resistance recovers to the baseline level after stopping \( \text{H}_2 \) exposure. The average time to reach 90% of the stable sensor signal (\( t_{90} \)) for the response is 5 s and for recovery is 97 s. In comparison, the LM-SnO\(_2\)-based sensor shows slower response and recovery times, with \( t_{90} \) for the response being about 36 s and the \( t_{90} \) for recovery being about 165 s. The sensitivity of the HM-SnO\(_2\)-based sensor shows approximately a linear dependence on the hydrogen concentration, as shown in Figure S5 (SI). The detection limits, calculated using the conventional signal-to-noise of 3, are 70 ppm for HM-SnO\(_2\) and 140 ppm for LM-SnO\(_2\) sensors. Thus, the HM-SnO\(_2\) shows superior sensing performance, including higher sensitivity, lower detection limit, and shorter response and recovery times, compared to LM-SnO\(_2\). We attribute these characteristics to a number of effects.\(^{30-41}\) One factor is the greater porosity of the HM-SnO\(_2\) sensing layer consisting of well-connected SnO\(_2\) nanoparticles. Although the high surface area might not necessarily lead to more active sites due to the Weisz limit\(^{42}\) of the gas sensor, these pores provide more channels for gas diffusion toward the mesoporous SnO\(_2\) surface and greater likelihood for sorption. Furthermore, the HM-SnO\(_2\) sensing layer retains good interconnectivity between SnO\(_2\) nanoparticles, which can provide faster electron transport, leading to better sensor performance. Another factor leading to the improved sensing characteristics of the HM-SnO\(_2\)-based device is the better electrical contact between the HM-SnO\(_2\) sensing material and the electrodes.

The selectivity of mesoporous SnO\(_2\) based sensors is also investigated with two hydrocarbon gases, namely, propane and methane. Figure 6d shows the response to these gases and \( \text{H}_2 \) at 3% concentrations, for both HM-SnO\(_2\) and LM-SnO\(_2\) sensors. The responses to propane and methane gases are significantly lower than to hydrogen. Clearly, the sensor shows excellent selectivity to \( \text{H}_2 \) over these other gases. Although the exact mechanism behind this selectivity is not clear, it is well-known that a sensing material’s surface structure and electronic characteristics strongly affect its interaction with various gases. There are thermodynamic considerations in that hydrocarbons commonly need more active sites and energy to react than hydrogen. There are also kinetic considerations in that hydrogen has a much faster rate of diffusion and smaller kinetic size than hydrocarbon gases, allowing more sensitive gas response.

As shown in Figure 6, the resistance of the mesoporous SnO\(_2\) material decreases when the sensor is exposed to \( \text{H}_2 \), consistent with SnO\(_2\) exhibiting n-type sensing behavior. When mesoporous SnO\(_2\) is exposed to air at high temperature (\( \sim 300 \) °C), oxygen molecules are chemisorbed on the mesoporous SnO\(_2\) surface. The adsorbed oxygen molecules trap electrons, creating an electron-depleted region, which leads to a barrier at the interface between contacting nanomateri-
The introduced hydrogen gas reacts with adsorbed oxygen molecules on the mesoporous SnO2 surface, resulting in the formation of water molecules and the release of the trapped electrons. This results in a decrease in resistance in n-type materials in response to H2. The higher porosity of HM-SnO2 allows selective gas-sensing performance with regard to multiple gases, depending on their structure, which can be easily tuned by self-assembly formation of the PVC-g-POEM graft copolymer template.

**CONCLUSION**

A new approach for direct synthesis and integration of mesoporous SnO2 for gas-sensing applications is demonstrated using amphiphilic PVC-g-POEM graft copolymer as a sacrificial template. The pore-controllable mesoporous SnO2 exhibits high surface area and multiple pores with great connectivity of materials self-assembled from well-developed microphase-separated PVC-g-POEM graft copolymer. The sensing performance of SnO2 depends on the pore structures dictated by the sacrificial template. High porosity of SnO2 based gas sensors exhibit high sensitivity toward hydrogen, low detection limit (60 ppm), fast response time (∼10 s) and recovery time (∼54 s), and excellent selectivity against other hydrocarbon gases at 300 °C.

This mesoporous material synthesis and integration method can be used to fabricate other types of metal oxide or metal/metal oxide complexes for a variety of gas-sensing applications. Furthermore, this strategy suggests the possibility to design commercial ink for an inkjet-printing-based gas sensor. Commonly, ink is prepared by as-prepared nanomaterials and polymer in solvent. It requires many steps, such as synthesis of nanomaterials, preparation of ink, and fabrication of a gas sensor. However, the novel method presented here, based on graft polymer self-assembly and nanomaterial precursor, can produce gas sensors efficiently with easy control, resulting in high surface area, excellent porosity, and interconnectivity. In addition, the graft copolymer and nanomaterial precursor ink can be used for long periods without special treatment, due to their stability under homogeneous conditions. The condition of the precursor solution is maintained for several weeks without any change, and the mesoporous SnO2 derived from the precursor solution after long times shows a structure identical with that from fresh precursor solution on the microheater for gas sensors.

**ASSOCIATED CONTENT**

Support Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsami.7b07823.

Synthesis procedure of PVC-g-POEM graft copolymer and schematic illustration of PVC-g-POEM graft copolymer, low-magnification SEM images of mesoporous SnO2, microheater platform with cross-sectional schematic image and top-view optical images showing heater and sensing electrodes, characterization of microheater power consumption, and sensitivity of mesoporous SnO2 toward various concentrations of hydrogen gas (PDF)

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**Notes**

The authors declare no competing financial interest.

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