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Hydrogen Sensing Properties of Mesoporous SnO₂ Loaded with Noble Metal

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Abstract
The m-SnO₂ powders loaded with noble metals (nM/m-SnO₂, n; the amount of noble metals loaded (wt%), M: Pd or Au) have been prepared and their H₂-sensing properties have been investigated in this study. The loading of Pd or Au to m-SnO₂ was very effective in improving H₂ sensing properties of almost all sensors. However, the response of nPd/m-SnO₂ decreased with an increase in the amount of Pd loading at a higher temperature, probably because of a high catalytic oxidation activity of H₂.

Keyword: mesoporous, noble metal, hydrogen sensing, oxidation activity

Introduction
Introduction of well-developed meso- and macro-pores to metal oxides as a gas-sensing material is quite effective for the improvement of their gas-sensing properties [1]. Among them, ordered m-SnO₂ has been of a great interest from the viewpoint of their controlled mesoporous structure, small crystallite size and large specific surface area, while the gas-sensing properties of m-SnO₂ sensors were not as good as those we expected. Thus, we have attempted to enhance the gas-sensing properties of m-SnO₂ by a loading of Pd or Au in this study.

Experimental
Preparation of nM/m-SnO₂ powders
nM/m-SnO₂ powders were prepared from the aqueous precursor solution containing appropriate amounts of n-cetylpyridinium chloride as a template, sodium stannate as a tin source and trimethylbenzene. After Pd(NO₃)₂·nH₂O or HAuCl₄·4H₂O was added in the precursor solution, the pH was adjusted to 10 using an aqueous HCl solution. The precipitates obtained were aged at 20°C for 3 days in the solution, filtered and then treated with 1.67×10⁻³ M aqueous phosphoric acid solution for 2 h to improve the thermal stability. The resultant powders were fired at 600°C for 5 h in air. The amounts of Pd or Au loaded were analyzed by X-ray fluorescence spectroscopy (XRF).

Fabrication of nM/m-SnO₂ sensors
The paste of each nM/m-SnO₂ powder was screen-printed on an alumina substrate, on which a pair of interdigitated Pt electrodes had been printed, to fabricate thick film sensors. Thereafter they were heated at 550°C for 5 h. Sensing properties of the thick film sensors to 1000 ppm H₂ balanced with air were measured in a temperature range of 150–450°C. The response was defined as Rₐ/Rₕ, where Rₐ and Rₕ were the sensor...
Results and Discussions

Figure 1 shows variations in \( \text{H}_2 \) response and sensor resistance in air with the loading amount of Pd or Au of \( n \text{M/m-SnO}_2 \) sensors at 150°C and 450°C. Sensor resistances of \( n \text{Pd/m-SnO}_2 \) and \( n \text{Au/m-SnO}_2 \) in air at 150°C decreased at 0.39 and 1.94 wt% loading, respectively, but tended to increase again with an increase in the loading amount. These variations in sensor resistance in air with the amounts of the noble metal leaded at 150°C were completely different from those expected, and reasons for the behavior are still obscure. On the other hand, their sensor resistances in air at 450°C monotonically increased with an increase in the amount of Pd or Au loading. In addition, the sensor resistance of \( n \text{Pd/m-SnO}_2 \) was larger than that of \( n \text{Au/m-SnO}_2 \), due to a stronger electronic interaction between Pd and m-SnO₂.

The magnitude of \( \text{H}_2 \) response at 150°C was improved by the Pd or Au loading and \( n \text{Au/m-SnO}_2 \) showed much larger response in comparison with those of \( n \text{Pd/m-SnO}_2 \). On the other hand, it increased with the amount of the Au loading even at 450°C, while the Pd loading resulted in no improvement in response. Generally, catalytic combustion properties of \( \text{H}_2 \) over Pd are much excellent than those over Au. Therefore, it is expected that most \( \text{H}_2 \) molecules burn at the upper region of thick \( n \text{Pd/m-SnO}_2 \) films, it contributes to decreasing of the number of \( \text{H}_2 \) molecules at the bottom region of thick and the reduction of the response. In the case of \( n \text{Au/m-SnO}_2 \), on the other hand, more \( \text{H}_2 \) molecules can diffuse into the bottom region of the oxide films, owing to their low catalytic activities, and leading to larger response to \( \text{H}_2 \).

Conclusions

Effects of Pd or Au loading to m-SnO₂ on their \( \text{H}_2 \) sensing properties were investigated in this study. Sensor resistances of \( n \text{Pd/m-SnO}_2 \) and \( n \text{Au/m-SnO}_2 \) in air at 150°C decreased once, and increased again with an increase in the loading amount. In addition, the sensor resistance of \( n \text{Pd/m-SnO}_2 \) was larger than that of \( n \text{Au/m-SnO}_2 \). On the other hand, \( \text{H}_2 \) response of \( n \text{Au/m-SnO}_2 \) was much larger than that of \( n \text{Pd/m-SnO}_2 \), probably because of low catalytic activity of the \( n \text{Au/m-SnO}_2 \).

References