INFLUENCE OF CVD PROCESS DURATION ON MORPHOLOGY, STRUCTURE AND SENSING PROPERTIES OF CARBONACEOUS - PALLADIUM FILMS

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Nanstructured materials containing different allotropic form of carbon such as foam, graphite, fullerene C60, nanotubes, nanofibers doped with transition metals (Pd, Pt, Cu, Fe, Ni) can be used in various applications e.g. in catalysis, hydrogen storage materials, hydrogen sensors, as electrodes in batteries or in fuel cell. Presently hydrogen technologies awaken of great interest among scientists and entrepreneurs. This interest is related to the potential use of hydrogen as an energy carrier. Recently, the growth of the hydrogen importance in the word economy e.g. in transport, chemical compounds production, in electronic and metalurgical industry has been noticeable.

We present C-Pd films based on nanoporous carbonaceous matrix containing Pd nanograins which are promising materials for hydrogen sensing applications. These films were prepared by PVD method and next were modified in CVD process at different times (5, 10 and 30 minutes). The aim of this work is to study the influence of duration of CVD process on morphology, topography, structure and hydrogen sensing properties of these films. These films are deposited on alumina (Al2O3) substrates and can be used as an active layer in hydrogen sensor.

SE and LABE images of CVD films

SE and LABE images of S5 film

SE and LABE images of S10 film

SE and LABE images of S30 film

TEM image of CVD films

TEM image of CVD films

TEM image of CVD films

Cathodoluminescence studies

SEM (a) and CL (b) images are presented for S10 film

Hydrogen sensing of C-Pd films

Hydrogen sensing properties were performed in different concentration of H2/N2 (0.5vol.%, 1vol.% and 2vol.%). The resistance changes of all films were carried out in the special chamber. The films’ resistance decreases in hydrogen presence.

The sensitivity \( \Delta R / R_i \) was calculated using the following equation:

\[ \Delta R / R_i = 100\% \]

where \( R_i \) - film initial resistance in H2 absence

\( R_f \) - film resistance after exposure to H2

Resistance of C-Pd films in air

<table>
<thead>
<tr>
<th>Films</th>
<th>( R_0 [\Omega] )</th>
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<tbody>
<tr>
<td>S5</td>
<td>2.14 ( 10^4 )</td>
</tr>
<tr>
<td>S10</td>
<td>1.61 ( 10^5 )</td>
</tr>
<tr>
<td>S30</td>
<td>7.36 ( 10^7 )</td>
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Film modified in 30 minutes has the highest sensitivity. This film characterizes the smallest thickness and the largest Pd nanograins and the greatest ratio of Pd/C; thus it can be used in hydrogen sensor applications

Conclusion

• duration of CVD process influences on morphology, topography and structure of carbonaceous matrix and also Pd nanograins in films prepared by PVD process;
• with increasing duration of CVD process the films’ thickness decreases;
• with increasing duration CVD process the size of Pd nanograins increases;
• the ratio of Pd/C increases with increasing time of CVD process;
• the film with the smallest thickness has the highest sensitivity and can be used as an active layer in hydrogen sensor;
• Pd nanograins encapsulated in graphite shells are optical active and show emission bands with maxima at 450 and 750 nm.

Figures:

1. SEM images show boundaries of substrate grains. On substrate surface carbonaceous nanograins are visible. These nanograins have different sizes, poses, rounded shapes and smooth walls.

2. After CVD modification morphology, topography and structure of PVD film are drastically changed.

3. SE and LABE images of PVD film

4. SE and LABE images of S5 film

5. SE and LABE images of S10 film

6. SE and LABE images of S30 film

7. Parameters of CVD process

8. EDS measurements

9. Hydrogen sensing properties of C-Pd films

10. Cathodoluminescence studies

11. TEM image of CVD films

12. Resistance of C-Pd films in air

13. Film modified in 30 minutes has the highest sensitivity. This film characterizes the smallest thickness and the largest Pd nanograins and the greatest ratio of Pd/C; thus it can be used in hydrogen sensor applications

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