Aligned Growth of ZnO Nanorods

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Background

In recent years, ZnO nanostructures have attracted a lot of research interests due to their unique structure- and size-dependent electrical, optical, and mechanical properties and great potentials in a wide range of applications. Our research focuses on the fabrication of vertically aligned and uniform ZnO nanorods, which is considered effective for the assembly of nanodevices and applications in light emitting and field emissions. We employ the vapor-liquid solid (VLS) process for the growth of the nanostructures because of its simple and efficient method. By varying the conditions – temperature, pressure, substrate position, and reactant gas partial pressure – in synthesis, different results can be obtained. The objective of our research is to determine the best condition(s) for growing perfectly aligned and uniform ZnO nanorods and provide a foundation for large-scale, controlled synthesis of ZnO nanorods to be used in practical applications.

Synthesis

Various substrates are used in our experiments, including sapphire, GaN, and AlGaN/AlN. These substrates have ordered surfaces that allow for the aligned and upward growth of the nanorods. For the VLS process, a layer of gold, used as a catalyst that initiates the growth, is plated on the substrates. The source material is prepared by mixing equal amounts of ZnO powder and carbon powder, which catalyzes the vaporization and reduce the vaporization temperature from ~1300°C to ~800°C. Below is the reaction process:

$$ZnO(s) + C(s) \rightarrow Zn(v) + CO(v)$$

Synthesis is conducted in a tube furnace (Figure 1) where the source material, loaded on a boat, is placed in the middle of the alumina tube while the substrate is placed somewhere downstream as shown in Figure 2.



Figure 1 - Barnstead International Low Temperature Tube Furnace



Figure 2 – Tube furnace setup

After the source material and substrate are loaded into the tube, the ends are sealed with steel caps, which are passed through with cooling water. This creates a temperature gradient inside the tube as the furnace heats. The caps also provide the carrying (Ar) and reactant (O2) gases that flow from one end of the tube to another. When all setups are finished, the furnace is heated to a certain temperature where the source material will begin to vaporize. The vapors are carried by the flowing gas to a lower temperature region where it is deposited on the substrate. The synthesis will continue until the furnace is stopped or when the source material is exhausted.

In past experiments, it is observed that chamber pressure is the most determining factor of the result quality. The pressure controls the growth by affecting the saturation of the vapor in the carrying gas and the reaction with the O2 reactant gas. The pressure fluctuates during synthesis as temperature changes and when the partial pressure of the vaporizing ZnO begins to contribute to the overall pressure. One of my main responsibilities in the research is to constantly adjust the pressure to a certain point, depending on the experiment, to ensure the best precision.

Growth Mechanisms

The vapor-liquid-solid (VLS) growth process is determined by the introduction of a metal catalyst. Gold is used as the catalyst in our experiments. Figure 3 demonstrates the VLS process:



Figure 3 – VLS growth process.

- [Left] A thin layer of gold is plated on the substrate.
- [Middle] As temperature heats up, the gold forms into nanosized droplets, and begins to absorb the incoming Zn vapors.
- **[Right]** ZnO ultimately supersaturates in the droplet as vapors are continued to be absorbed. When this happens, ZnO is precipitated out at the solid-liquid interface and forms wire- or belt-like structures.

Results and Discussion

Each experiment is recorded in a log and all parameters are written down. Below is a sample entry: ZnO - B46 7-22-2005 Source Materials: ZnO - 0.6g C - 0.6g Substrate: AlGaN:Mg catalyst, sputter 30" Au 25cm from the end Low temperature furnace: Small valve: 0.1 mbar Big value: 4.0×10^{-2} mbar Heat rate: 50°C/min Keep at 950°C for 30 minutes Gas: Ar - 49.0 sccm O2 - 1.0 sccm Keep pressure at 30 mbar Products from the experiments are checked with a scanning electron microscope (SEM) (Figure 4).



Figure 4 – SEM

Our experiment data is unlike others in that it cannot really be expressed in charts and graphs. Rather, the quality of the products we produce are determined visually and images are taken at different regions of the samples for records. Figure 4 is an example result from an experiment:



Figure 5

In Figure 5, it can be seen that a very high percentage of the nanorods are aligned and perpendicular to the surface. In addition, their dimensions are very much identical. Although several nanorods are slanted or tipped over, this is considered to be a very good result. Referring back to the information recorded in the logs helps us determine the most ideal conditions to consistently grow products of similar (or better) quality.

Conclusion

So far, we have been able to define a rough "phase diagram" for the synthesis of aligned ZnO nanorods at different levels of quality from numerous experiments at various conditions. In the future, we hope to use this as a foundation for the large-scale production of perfectly aligned and uniform ZnO nanorods for application in nanodevices. Also in the future, our work may benefit significantly from several important developments, including the development of a more robust method of synthesis and a more precise furnace system. These will enable us to achieve better consistency and have more control over the kinetics and conditions of the nanorod growth, as well as quality of the products.

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References

 Wang, Xudong. Patterned and Aligned ZnO 1D Nanostructures: Fabrication, Characterization, and Applications. School of Materials Science & Engineering, Georgia Institute of Technology.
Song, Jinhui; Xudong Wang; Elisa Riedo; Zhong L. Wang, The Journal of Physical Chemistry B Letters, 2005, 109, 9869-9872.