MODES OF MICROWAVE HYDROTHERMAL METHOD FOR GROWING ZINC OXIDE NANORODS AND NANOTUBES

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The hydrothermal method for growing zinc oxide (ZnO) nanostructures has many advantages, such as the simplicity of the procedure, which does not require high temperatures and gives high yields at low costs, as well as the controllability of technology, which allows the creation of nanostructures of a given shape and good quality. By varying the composition of the solution, the temperature and pressure in the autoclave reactor, and also the deposition time, it is possible to change the morphology and properties of thin-film zinc oxide arrays from nanowires and nanorods to nanotubes and nanosheets. However, in the conventional hydrothermal method, deposition of ZnO nanostructure arrays takes tens of hours. Recently, microwave hydrothermal synthesis as an ultrafast method has attracted much attention due to its advantages over the conventional method, such as increasing the volume and quality of nanostructured zinc oxide arrays. With this approach, microwave heat is released throughout the material as the electromagnetic energy of the microwaves is converted through dipole polarization and conduction into thermal energy, which ensures uniform heating in the reactor. Due to this, in the microwave hydrothermal process, a high supersaturation of the reagents and their uniform distribution throughout the autoclave are achieved, which accelerates the growth process and facilitates the production of nanostructures with a small particle size distribution and a high degree of crystallinity. In addition, microwave heating allows precise timing and temperature planning, and microwave exposure can influence chemical reaction pathways, for example, reducing the dielectric constant of water and turning an aqueous solution into a supercritical fluid.

Herein, arrays of zinc oxide nanorods and nanotubes oriented vertically on the surface of carbon fabric fibers were grown by microwave hydrothermal method from aqueous solutions of zinc nitrate and hexamethylenetetramine. The method provided real-time pressure control of ~15-18 bar, microwave heating to temperatures in the range of 100-132°C for 25 minutes and controlled residence time of the samples inside the reactor while the solution was cooling. SEM images revealed a strong dependence of the morphology of zinc oxide nanostructures on the residence time of the samples in the reactor at the solution cooling stage. Analysis of optical diffuse reflectance showed that the band gap of direct optical transitions in both nanostructured zinc oxide layers is close to the typical value for bulk ZnO. Raman spectroscopy revealed peaks characteristic of the wurtzite-type ZnO crystal lattice, which are shifted towards low frequencies due to the presence of impurity defects and the small size of ZnO crystals.