

Flame synthesis of 1-D complex metal oxide nanomaterials

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Abstract

One dimensional (1-D) complex metal oxide nanomaterials, such as ternary oxides, doped oxides, and hierarchical structures containing several oxides, not only benefit from large aspect ratios, but also offer exciting opportunities to design materials with desired properties by tuning their chemical compositions and tailoring their sizes and morphologies at the nanometer scale. Flame synthesis is an attractive method to grow 1-D complex metal oxide nanostructures because of its high temperature, scalability, low-cost and rapid growth rate. Here, we present three new combined flame synthesis methods: (1) simultaneous vapor–vapor growth, (2) simultaneous solid–vapor growth, and (3) sequential solid–vapor growth, to grow 1-D complex metal oxide nanostructures with well-defined compositions and morphologies. These three methods combine the previously reported flame vapor deposition and solid diffusion growth methods that were separately used to grow 1-D simple binary metal oxide nanostructures, and significantly advance the capabilities of existing flame synthesis methods for the growth of 1-D nanomaterials. The first method, simultaneous vapor–vapor growth, combines the flame vapor deposition growth of two different metal oxides by oxidizing and evaporating two different metal sources. With this we have successfully grown W-doped MoO₃ nanoplates and nanoflowers. In the second method, simultaneous solid–vapor growth, one precursor is again provided by oxidizing and evaporating metal oxide from a metal, while the other precursor diffuses out from a different growth substrate. With this we have successfully grown ternary Cu₃Mo₂O₉ nanowires. The third method, sequential solid–vapor growth, essentially uses the 1-D nanostructures firstly grown by solid diffusion as the substrates for subsequent flame vapor deposition. With this we have successfully grown hierarchical CuO/MoO₃ core/shell nanowires and MoO₃-branched CuO nanowires. We believe that these three new combined flame synthesis methods will provide a general platform for the synthesis of 1-D complex metal oxide nanostructures with tailored properties.

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1. Introduction

One dimensional (1-D) nanostructured complex metal oxides, such as doped oxides [1], ter-

nary oxides [2], and hierarchical structures containing several oxides [3], offer exciting opportunities to design materials with desired functionalities by tuning their chemical compositions, sizes and morphologies at the nanometer scale. As such, these nanomaterials have not only large aspect ratios [4] but also different thermal [5],

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electrical [6] and optical properties [7] from those of bulk materials, and hence are promising building blocks for many applications, including lithium ion batteries [8], electroluminescent devices [9], catalysts [10], thermoelectric devices [11], transparent conductive oxides [7] and superconductors [12]. For example, Mo-doped WO_3 photoanodes need smaller applied external bias than pure WO_3 photoanodes for photoelectrochemical water splitting, due to the increased conduction band minimum by doping [13]. In addition, ternary $\text{Cu}_3\text{Mo}_2\text{O}_9$ nanorods exhibited higher catalytic activity for the ignition of soot (Printex U model) than CuO and MoO_3 , due to the easier charge transfer among Cu, O and Mo ions in $\text{Cu}_3\text{Mo}_2\text{O}_9$ lattice [14]. Furthermore, Co_3O_4 nanoparticles decorated WO_3 nanowires (NWs) showed lower onset potential and higher current density than WO_3 NWs in water electrocatalysis, due to the combination of active Co_3O_4 surface properties and good charge transfer process in WO_3 [15].

1-D complex metal oxide nanomaterials have been synthesized by methods including the sol-gel method [16], the hydrothermal method [17], and chemical vapor deposition (CVD) [18]. However, these methods typically have significant limitations. Solution phase methods, such as the sol-gel method and the hydrothermal method, usually need several steps of washing, purification, drying and annealing. In addition, the low growth temperature used for the solution phase growth typically leads to low growth rate and poor crystallinity. CVD growth is typically conducted under vacuum conditions that lead to small material growth rate and difficulty in scaling-up production. Flame synthesis, instead, offers many advantages for the mass-production of 1-D metal oxide nanomaterials, such as atmospheric operation, rapid growth rate, low cost, scalability, fine control over the morphology of the synthesized products, and broad choices of growth substrate materials and morphologies. Recently, flame synthesis has been successfully used to grow many 1-D binary metal oxide nanostructures, such as ZnO NWs [19], Fe_3O_4 NWs [20], $\text{WO}_{2.9}$ nanofibers [21], MoO_2 hollow and solid nanorods and whiskers [22,23], $\text{W}_{18}\text{O}_{49}$ NWs, WO_3 NWs and nanotubes [24], $\alpha\text{-MoO}_3$ nanobelts, nanoflowers and branched nanobelts [25], $\gamma\text{-Fe}_2\text{O}_3$ NWs [26], $\alpha\text{-Fe}_2\text{O}_3$ nanoflakes and CuO NWs [27]. All these previous flame synthesis studies mainly focused on the growth of 1-D *simple binary metal oxide nanostructures* by using either flame vapor deposition or solid diffusion growth mechanism. However, to date, flame synthesis of 1-D *complex metal oxide nanostructures* has not been reported.

Here, for the first time, we present three flame synthesis methods, i.e., (1) simultaneous vapor-vapor growth, (2) simultaneous solid-vapor growth, and (3) sequential solid-vapor growth,

for the synthesis of 1-D complex metal oxide nanostructures by combining flame vapor deposition and solid diffusion growth mechanisms that, previously, were separately used to grow 1-D simple binary metal oxide nanostructures. These three new combined methods significantly advance the capacities of existing flame synthesis methods for the growth of various 1-D complex metal oxide nanomaterials, including doped oxide nanoplates and nanoflowers, ternary oxide NWs, hierarchical core/shell NWs and branched NWs. Moreover, the morphologies and compositions of the 1-D complex metal oxide nanostructures can be controlled by tuning the flame fuel to oxidizer equivalence ratio (Φ), the vapor precursor source temperature (T_{source}) and concentration, the growth substrate temperature (T_{growth}), and the growth time (t_{growth}).

2. Experimental specifications

The 1-D complex metal oxide nanomaterials were synthesized using a 6-cm diameter co-flow flat premixed flame burner (McKenna Burner) [28]. The flat flame was stabilized on a water-cooled porous sintered plug, through which fuels (CH_4 and H_2) and oxidizer (air) were introduced. The flow rates of CH_4 and H_2 were 2.00 SLPM and 4.00 SLPM, respectively, while the flow rate of the air was varied from 20.4 SLPM to 71.4 SLPM, yielding a fuel to oxidizer equivalence ratio range of 0.4–1.4 which the burner can operate. For the synthesis of complex oxide nanostructures reported here, the burner was only operated in the fuel to oxidizer equivalence ratio range of 0.5–0.8. In addition, co-flow air was introduced as a shroud to prevent air entrainment and improve the flame stability. The key merit of the flat premixed flame is that the temperature was nearly constant in any plane parallel to the flame, which provided an ideal environment for the uniform growth of 1-D complex metal oxide nanostructures.

Previously, we and others have mainly used two mechanisms to grow 1-D simple binary metal oxide nanostructures in flame: flame vapor deposition [19–26] and solid diffusion growth [27,29,30]. In flame vapor deposition (Fig. 1a), the flame oxidizes metals (e.g., Mo mesh) and evaporates metal oxide vapors that further condense onto colder growth substrates in the form of 1-D nanomaterials (e.g., $\alpha\text{-MoO}_3$ nanobelts) [25]. In solid diffusion growth (Fig. 1b), the flame rapidly heats up metal substrates (e.g., Cu mesh), from which metals (e.g., Cu) diffuse out to the surface and are oxidized by flame, forming 1-D metal oxide nanostructures (e.g., CuO NWs) on the original metal substrates [27].

Herein, we combined the flame vapor deposition and solid diffusion growth mechanisms, and

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