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Growth of zinc oxide nanowires by a hot water deposition method

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Abstracts

Recently, various methods have been developed for synthesizing zinc oxide (ZnO) nanostructures, including physical and chemical vapor deposition, as well as wet chemistry. These common methods require either high temperature, high vacuum, or toxic chemicals. In this study, we report the growth of zinc oxide ZnO nanowires by a new hot water deposition (HWD) method on various types of substrates, including copper plates, foams, and meshes, as well as on indium tin oxide (ITO)-coated glasses (ITO/glass). HWD is derived from the hot water treatment (HWT) method, which involves immersing piece(s) of metal and substrate(s) in hot deionized water (DI) water and does not require any additives or catalysts. Metal acts as the source of metal oxide molecules that migrate in water and deposit on the substrate surface to form metal oxide nanostructures (MONSTRs). The morphological and crystallographic analyses of the source-metals and substrates revealed the presence of uniformly crystalline ZnO nanorods after the HWD. In addition, the growth mechanism of ZnO nanowires using HWD is discussed. This process is simple, inexpensive, low temperature, scalable, and eco-friendly. Moreover, HWD can be used to deposit a large variety of MONSTRs on almost any type of substrate material or geometry.

Keywords: Metal oxide nanostructures, zinc oxide, nanowires, deposition, hot water treatment, substrate, low temperature.

Introduction

Zinc oxide (ZnO) nanostructures, such as nanowires, nanorods, and nanoneedles, have received great interest because of their unique optical, electronic, magnetic, mechanical, and antimicrobial properties [1-7]. With a direct wide bandgap (3.37 e V) and high exciton binding energy (60 me V) at room temperature [8-10], ZnO has been extensively investigated because of its potential use in various applications, including optoelectronic, photonic, field emission, energy storage and conversion, catalysis, and sensing devices [11-16]. Different methods for synthesizing ZnO nanostructures have been reported in the literature, some of which involve either high-temperature or high-vacuum physical and chemical vapor deposition techniques [17-22], that are commonly employed for producing various types of thin films [23-25]. However, these approaches are generally expensive and energy-demanding, because they typically require relatively more complicated equipment and high-temperature growth conditions. Alternatively, various low-temperature solution-based approaches (*e.g.*, hydrothermal method) have recently been developed to fabricate ZnO nanostructures with promising potential for scaled-up production and commercial feasibility [26-31]. However, such wet-chemical synthesis methods still require potentially toxic chemicals and additives.

Recently, formation of metal oxide nanostructures (MONSTRs) on metal substrates using a simple hot water treatment (HWT) method has been reported [32-37]. HWT is a solvent-free method that involves immersing a metal in hot water at a relatively low temperature as low as ~50 ^{o}C. HWT can also be used to grow nanostructures on a wide variety of metallic materials including elemental metals, alloys, and compounds [38]. The surface morphology, crystal structure, and chemical composition of the substrates treated with hot water showed the growth of well-developed nanoscale features of the thermodynamically stable metal oxides [39]. Various nanostructures were observed on the substrate surface after the HWT, including cubes, pyramids, plates, wires, spheres, and leaf-like nanostructures. As illustrated in Figure 1, a hot water treatment growth mechanism that includes the combination of surface diffusion and a dissolution-precipitation process called "plugging" was proposed to explain the growth of nanoscale features as opposed to smooth thin films [40-42]. Briefly, during the HWT, metal oxide molecules form on the surface of a metallic substrate through a water-metal surface reaction. This is followed by plugging, which involves the release of metal oxide molecules from the metal surface, migration through water, and re-deposition onto another metal surface point. Re-deposited molecules can initiate the formation of isolated MONSTRs. However, the random nature of plugging might not be sufficient to explain the smooth crystalline surfaces observed in HWT nanostructures. Therefore, surface diffusion along with plugging is believed to be the main mechanism behind the formation of MONSTRs with smooth crystal facets. Nevertheless, HWT is limited to the re-deposition of MONSTRs on the metal source itself. The synthesis of metal-oxide nanostructures on different substrates is essential because the substrate can significantly influence the properties of the nanomaterial, such as its electronic and optical characteristics [43]. Different substrates allow the production of nanostructures with tailored functionalities suited for specific applications, including flexible electronics and high-performance sensors. Additionally, selecting an appropriate substrate enhances the stability of nanostructures and facilitates their incorporation into devices, which is vital for their real-world application in areas such as microelectronics and photonics [44, 45]. Therefore, there is still a need to grow MONSTRs on a variety of substrates that are different from the source metal utilizing a hot water process without the need for any chemical additives.

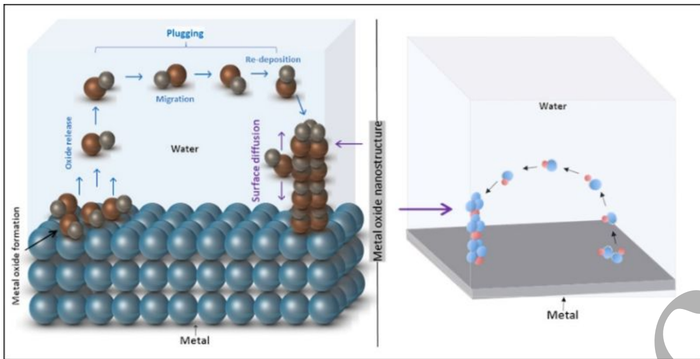

Figure 1: HWT method to grow MONSTRs on a metal surface, which acts both as the source and substrate, through plugging and surface diffusion mechanisms.

In this study, inspired by HWT, we developed a new method called hot water deposition (HWD) for growing MONSTRs of a metal oxide on a substrate surface of various types. HWD involves growth mechanisms similar to those of HWT, with the main difference being the migration and deposition of metal oxide molecules on a substrate that differs from the source metal. HWD offers advantages similar to those of HWT, including simplicity, low cost, low temperature, scalability, high-throughput, and does not involve any chemical agents or surface activators. Moreover, HWD can be used to deposit a large variety of MONSTR on almost any type of substrate material or geometry.

Methods and Materials

Commercial-grade Zn plates and powders (99% purity) were used as the source materials, while Cu mesh, foam, plates (99% purity), and indium tin oxide (ITO)-coated glasses (ITO/glass) (9-12 ohm/sq) served as the substrate materials. The HWD process for Zn and Cu plates (Figure 3) was performed for 2 hours by immersing the Zn source and Cu substrate in 100 mL of ultra-pure deionized water (DI) water (18.2 MΩ. cm) and maintaining the water temperature at 75 °C using a thermocouple. For the remaining experiments, the same parameters and setups were employed with a longer treatment time of 3 hours. To deposit ZnO on the Cu plate, mesh and ITO/glass substrates, a Zn plate was used as the source material, facing the substrate materials and separated by two inert nonconductive polymeric spacers with a diameter of 4 mm. The setup was held together using polymeric clips and positioned vertically by touching the bottom of the beaker. With Zn powder as the source material, Cu foam was placed directly on the Zn powder without the use of a spacer. After HWD, substrate samples were dried with nitrogen. Morphological analyses were performed using a scanning electron microscope (SEM, JOEL JSM-7000F, Tokyo, Japan) and transmission electron microscopy (TEM; Hitachi HF3300, Ibaraki Prefecture, Japan located at Oak Ridge National Laboratory). The crystal structures and compositions of the as-grown ZnO nanorods were analyzed using X-ray diffraction XRD (Rigaku Miniflex 600, Tokyo, Japan) and energy dispersive spectroscopy (EDS, EDAX Apex).

Results and Discussion

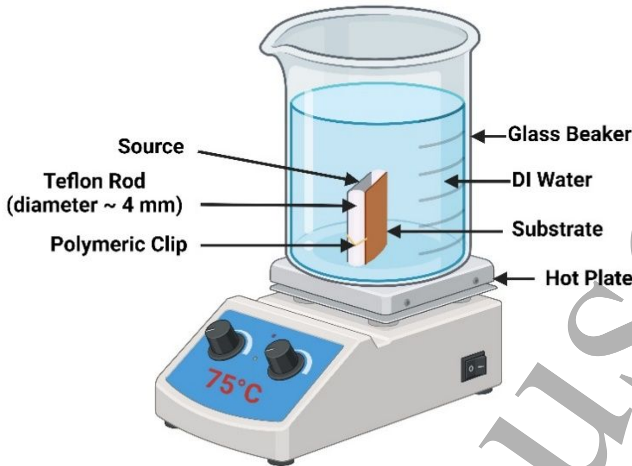

Figure 2: HWD method to grow MONSTRs on a substrate surface using metal as the source.

As illustrated in Figure 2, the HWD process involves a source metal and a target substrate that are both immersed in hot water facing each other. Water temperature between 50 - 95 ^{o}C is expected to be sufficient for the growth of MONSTRs by HWD, based on the results reported for HWT [38]. To demonstrate HWD, we chose ZnO nanowires as the material of interest because of their potential applications, as summarized above. For this purpose, we first used commercial grade plates of Zn as the source metal and Cu as the substrate, on which the ZnO MONSTR was to be deposited. The polished Cu and Zn plates were positioned vertically, as shown in Figure 2, facing each other in hot DI water for 2 hours at 75 ˚C and separated by 4 mm. In our earlier work [38], we observed that Zn has a faster response to HWT than Cu and can form ZnO nanowires with hexagonal cross-sections after approximately 30 - 45 minutes of treatment. In contrast, Cu takes approximately 4 hours to grow Cu_2O nanocubes and 16 hours to grow CuO nanoleaves [46]. Therefore, it is expected that zinc oxide molecules will form before copper oxide molecules during the HWD experiments. It is also expected that ZnO molecules will migrate from Zn toward the Cu surface as part of the plugging mechanism and deposit on Cu as ZnO nanowires.

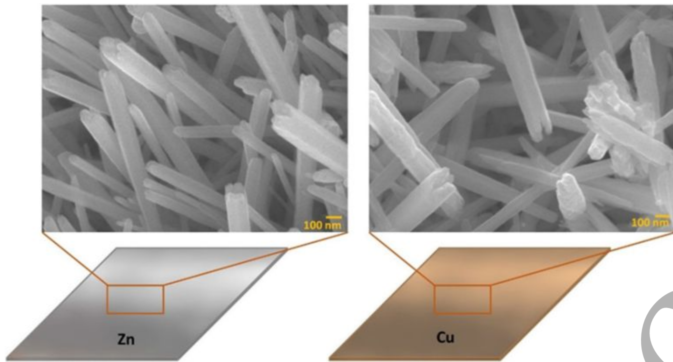

Figure 3: ZnO nanowires were deposited on a Cu plate substrate (right) after 2 hours of HWD at 75 ^{o}C using a Zn plate as the source (left). ZnO nanowires also grew on the Zn plate through the HWT mechanism.

SEM images of the Zn and Cu plates, shown in Figure 3, after 2 hours of the HWD process revealed that ZnO nanowires with lengths of a few hundreds of nanometers and hexagonal cross-sections of approximately 75 - 150 nm grew on both the Zn plate (source metal) and Cu plate (substrate) following the proposed growth mechanisms of HWT (re-deposition) and HWD (deposition), respectively. SEM images also showed that the ZnO MONSTRs uniformly covered the entire Cu substrate surface. EDS was used to analyze the chemical composition of the as-grown ZnO nanorods on top of Cu plates. The distribution of Zn, Cu, and O was determined by EDS elemental mapping, as shown in Figure 4. The presence of 14.6 % Cu may be attributed to the underlying Cu substrate, as evidenced by the gaps between the nanorods in Figure 4(a). Figure 4(b) presents a depiction of Zn, Cu, and O molecules superimposed on the SEM image. This figure illustrates that the majority of the Cu molecules are situated in the interstices between the ZnO nanowires, while the oxygen molecules are distributed uniformly across the substrate, primarily resting on the nanorods. The atomic percentages of Zn, Cu, and O are displayed in Figure 4(c) as 31.6%, 14.6%, and 53.8%, respectively. Additionally, the higher atomic percentage of O can be attributed to the native oxide layer beneath the ZnO nanorods, which may have formed after HWD when the samples were exposed to the environment. To assess the purity of the ZnO nanowires and analyze the spatial distribution of elements, we conducted line scan analysis using EDS (see Supplementary Information). The ZnO nanowires were detached from the Cu substrate and deposited onto carbon tape for analysis. The results confirmed the absence of Cu contamination in the nanowires, indicating that the nanowire synthesis was successful and free from substrate material interference. This ensured the structural integrity and purity of the ZnO nanowires for further characterization.

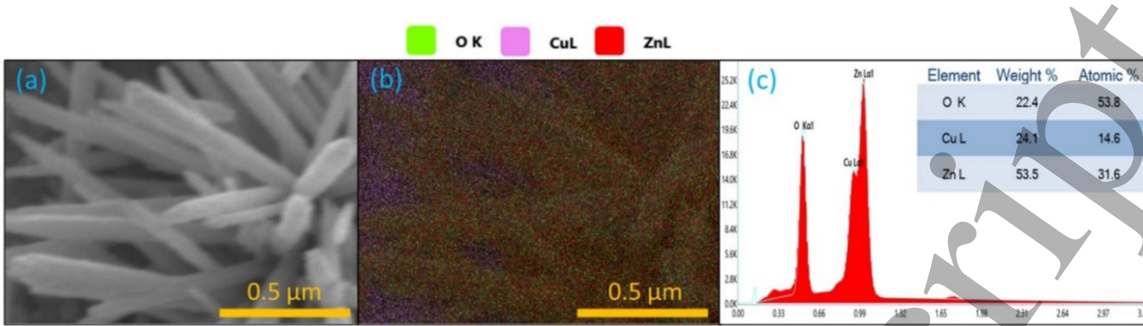

Figure 4: (a) SEM image, (b) corresponding superimposed EDS elemental map on the SEM image, and (c) elemental composition of ZnO nanostructures grown on Cu plate by HWD.

The crystallographic information of the ZnO nanowires was analyzed by XRD. (Figure 5(a)). The sharp ZnO <100> peak at 2θ = 32.07˚ [crystallography open database entry 1011258] indicates that the ZnO nanowires had a well-developed, highly oriented crystal structure. The more dominant Cu <111> and <200> peaks at 2θ = 43.72˚ and 50.92˚, respectively [crystallography open database entry 4313203], are attributed to the underlying Cu substrate. The XRD pattern of ZnO exhibited a single prominent peak, which was attributed to the preferred orientation of the ZnO nanostructures grown on the Cu substrate. In contrast to the randomly oriented crystallites in the powdered samples that produce multiple diffraction peaks, the ZnO nanostructures on the Cu substrate are potentially preferentially aligned, resulting in the predominance of a single peak in the diffraction data. TEM was used to further investigate the crystallinity of the ZnO nanowires grown on the Cu plate. The ZnO nanowires were scraped off from the Cu plate after HWD and placed on a Cu mesh TEM grid for TEM analysis. The low-magnification TEM image shown in Fig. 5(b) shows an average ZnO diameter of approximately 110 nm, which is consistent with the values found in the SEM images (Figure 3). The dark stripes observed in Fig. 5(b), oriented perpendicular to the nanorod axis, are likely the result of nanoscale stacking faults, potentially involving dislocations, grain boundaries, or twin boundaries [47]. Such planar defects disrupt the periodic atomic arrangement, leading to increased electron scattering in comparison to undisturbed regions, which appear as dark bands. The TEM image in Figure 5(c) reveals that the nanowire is a single crystal. The lattice spacing was measured to be 0.26 nm using ImageJ software, which corresponds to the (002) plane of wurtzite ZnO, according to the Crystallography Open Database (entry 1011258). This result, in conjunction with the XRD data, suggests that the ZnO nanowires likely exhibit a hexagonal wurtzite structure with a preferred orientation along the c-axis. However, further characterization, such as SAED analysis, is necessary to conclusively confirm the crystal structure and orientation of the ZnO nanowires. The hexagonal wurtzite structure of ZnO offers the highest thermodynamic stability among its various structural configurations [48]. Due to the elevated surface energy of the axial crystal facet of ZnO nanowires, this facet exhibits a higher growth rate compared to the lateral facets, promoting the preferential addition of atoms along the c-axis [49]. As a result, during HWD, ZnO molecules likely adhere more readily to the axial facet, leading to the preferential formation of the nanowire morphology.

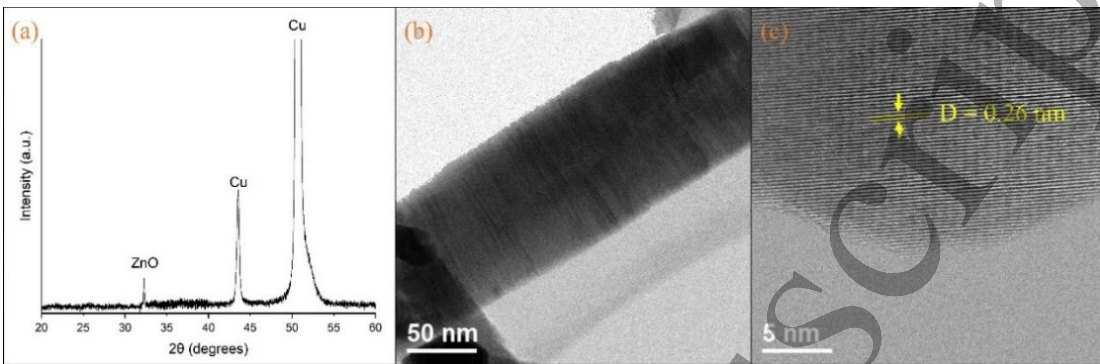

Figure 5: (a) XRD profile of ZnO on Cu plate, (b) TEM image and (c) high resolution TEM image of ZnO nanowire after 2 hours of HWD.

Moreover, we investigated the ability of HWD to grow ZnO MONSTRs on different substrate types. In addition to the flat Cu plate, 3D substrate geometries, including Cu mesh and foam, as well as ITO/glass as an oxide material, were used as substrates for the HWD of the ZnO nanowires. The HWD experiments were conducted for 3 hours at 75 ˚C. The SEM images in Figure 6 reveal the uniform deposition of ZnO nanowires on all the substrates studied. Our results also show that different forms of metal sources, e.g., metal powders, can be used in HWD. As shown in Figure 6, commercial grade microparticles of Zn powder successfully acted as the source for growing ZnO nanowires on Cu foam. Depending on the Zn source (plate vs. powder) and substrate type, ZnO nanowires had different diameters in the range of 50 - 150 nm on Cu mesh, 25 - 75 nm on Cu foam (Zn powder source), 100 - 200 nm on Cu plate, and 150 - 200 nm on ITO/glass substrates.

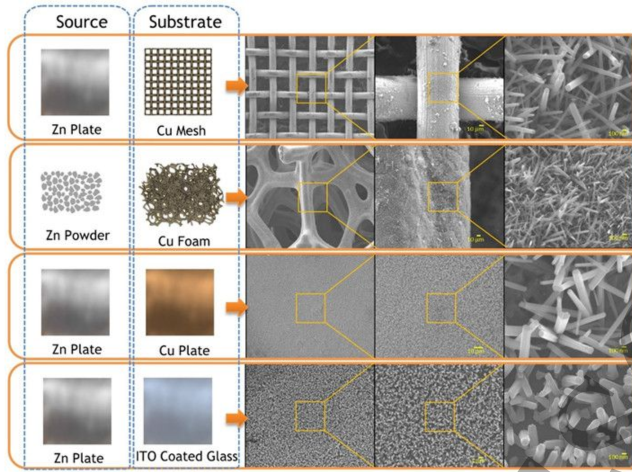

Figure 6: ZnO nanowires deposited on several types of substrate geometries and materials including Cu mesh, Cu foam, Cu plate, and ITO/glass after 3 hours of HWD at 75 °C. Zn plate or Zn powder was used as the source.

Figure 7 illustrates a possible growth mechanism by which the HWD technique. As previously discussed, ZnO molecules near the source metal (Zn plate or powders) must have formed prior to any copper oxide molecules from the substrate (Cu mesh, foam, or plate) during the brief period of the HWD in this study. Moreover, our research found that ITO/glass were not reactive to the HWT (see Appendix). Therefore, during the HWD process, the ZnO molecules may have been released and migrated into the water, similar to the regular HWT process. However, migrated ZnO molecules can now be deposited on both the substrate and the source (Zn or powder) to form MONSTRs. As shown in Figure 3, hexagonal ZnO nanowires are observed on both the Zn and Cu substrates. The vast majority of ZnO molecules are likely entrapped within the confined space situated between the plates, as this area was only accessible to the outside water from the top, whereas the remaining sides were obstructed by Teflon rods or the base of the beaker. This trapping of metal oxide molecules may have contributed to the dense growth of the ZnO nanostructures, as evidenced by the SEM images.

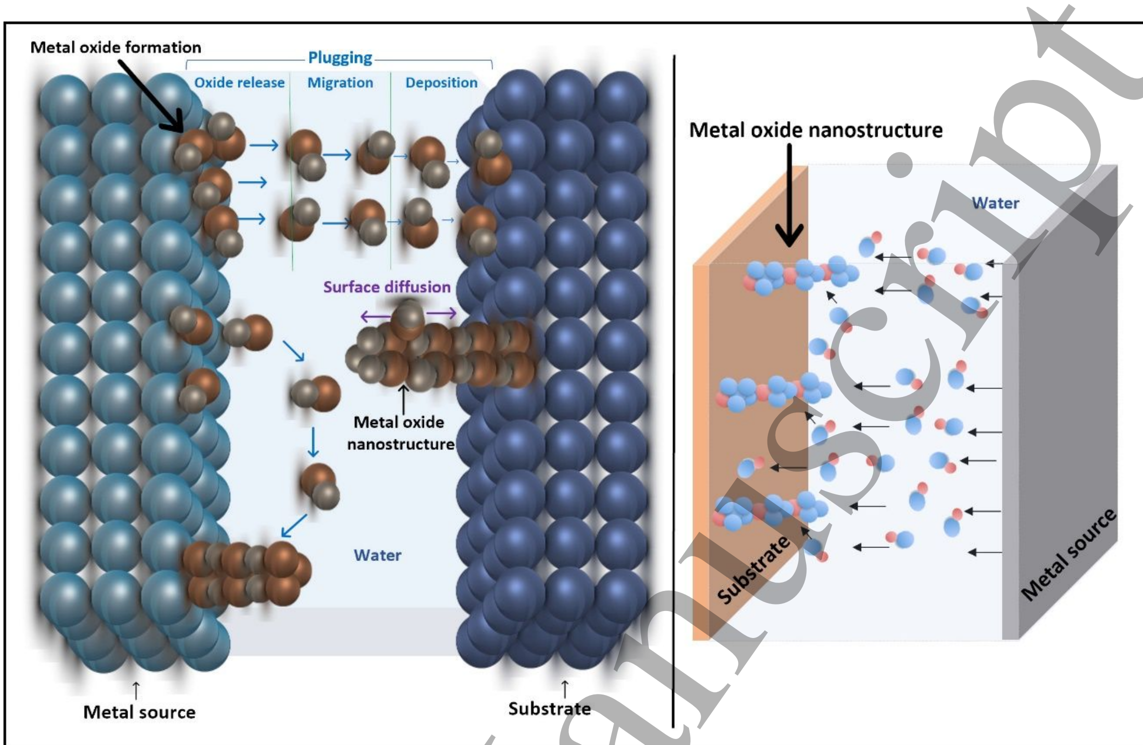

Figure 7: Proposed growth mechanism of HWD method to deposit MONSTRs on a substrate surface, in addition to source itself (re-deposition) through plugging and surface diffusion.

Furthermore, the smooth facets of these hexagonal nanowires are believed to be due to the surface diffusion of the metal oxide molecules on the nanostructure surface after the rough surface is created by the random nature of plugging (i.e., release, migration, and re-deposition) [38]. The variation in the diameters of the ZnO nanowires is attributable to the surface-area-to-volume ratio (SA/V) of the different substrates. When Zn plates are utilized as the source, the concentrations of the released ZnO molecules are expected to be similar. Additionally, the geometrical shapes of the ITO/glass and Cu plates are analogous; hence, their SA/Vs values might also be comparable. As a result, the diameters of the ZnO nanowires on these substrates were within a similar range, as depicted in the SEM images in Figure 6. However, the Cu mesh had a larger SA/V than the ITO/glass and Cu plates. Consequently, the Cu mesh should possess shorter nanowires with smaller diameters given that the same amount of released and migrated ZnO molecules are now being deposited on a larger surface area than in the cases of ITO/glass and Cu plates. We observed shorter ZnO diameters on Cu mesh, as shown in Figure 6. In contrast, the ZnO nanowires observed on the Cu foam substrates where we used Zn powder as the source had the shortest lengths and smallest diameters. It is reasonable to expect that Zn powder could provide a more substantial release of ZnO molecules because of its larger surface area compared to Zn plate, thereby enhancing the deposition rate. However, it appears that the Zn particles were not small enough or their surface was passivated with a thicker native oxide layer that might have hindered the release of ZnO molecules and consequently the deposition rate, leading to smaller nanowires. Further research is required to determine the growth mechanism of the HWD process. In addition, substrates with other shapes and compositions should be investigated in the future.

Conclusion

In summary, we presented a novel HWD method for producing nanostructured metal oxides by immersing a source metal and a substrate in hot water. We used Zn plates and powder as the source materials, and Cu foam, mesh, plates, and ITO/glass substrates. SEM images revealed that uniform nanowires were formed on all the substrates studied after only 2-3 hours of HWD, and elemental mapping showed the presence of Zn and O on the surface of the substrates. XRD and TEM results demonstrated the excellent quality of the wurtzite ZnO nanowires. The growth mechanism of MOSNTRs during HWD can be attributed to the plugging process and the surface diffusion of metal oxides. Additionally, the variations in the surface-area-to-volume ratio of the source and substrate materials could account for the differences observed in the diameters of the grown ZnO nanowires. Our study demonstrates that HWD is a low-temperature process that does not require any special environments, chemicals, or processing techniques, such as vacuum, acidic or alkaline solutions, catalysts, or lithographic processing. Furthermore, our study showed that MOSNTRs can be grown using HWD on substrate materials with different configurations. Finally, HWD presents the possibility of growing MOSTRs on a range of substrate geometries, including 1D (e.g., wires and rods), 2D (e.g., plates, foils, and thin films), and 3D (e.g., powder, pipe, mesh, and foam).

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