

Article

Diffusional Solid-State Transformation for the Formation of Copper Oxide Nanowires During the Thermal Oxidation Process

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Abstract

The solid-state transformation theory has been used to describe the formation of CuO nanowires during the oxidation of Cu metal in air. In order to fill the gaps of the nucleation mechanism of CuO nanowires, a quantitative model has been founded based on the classical nucleation theory, and the results show that the formation of the CuO nanowires is controlled by a solid solution precipitation process under steady-state heterogeneous nucleation circumstances, which will provide beneficial references for the analysis and preparation of metal oxide nanowires of other metal elements.

Keywords: CuO nanowires; solid-state transformation; nucleation

1. Introduction

Metal compound nanowire materials, owing to their unique properties, have been widely applied [1–3]. As a promising semiconducting material, CuO nanowires have also attracted significant attention. Practical synthesis methods have been developed to attain the required morphologies and structures. To date, there are two mostly used methods for the preparation of CuO nanowires (NWs): the wet chemical method and the thermal oxidation method [4]. Specifically, the latter method can prepare CuO NWs with fine crystalline and large scale easily. The thermal oxidation method can provide appropriate products for fabricating nanosized electrical junctions, optoelectronic, and electromechanical devices [5]. An in-depth study of the formation mechanism of CuO NWs in the oxidation of copper will be helpful to prepare the required products for different applications. To date, there have been many works on the mechanism, and several typical models have been proposed to discuss the growth mechanism of CuO NWs [6–12]. In these models, the stress-induced short-circuit diffusion model shows evidence-based proficiency and is considered outstanding [4]. Almost all the publications until now have just focused on the growth process; however, although the nucleation process of the CuO NWs is still not clear, there have been some conjectures proposed [4].

The vapor–solid growth (VS) model and the stress accumulation–release (SAR) model are the most popular theories to explain the CuO NWs nucleation process. According to Yang’s investigations [7], during the oxidation of Cu metal in an oxygen-containing atmosphere, Cu₂O will form first, and then the CuO layer. When the CuO vapor is saturated above the oxide layer, it will nucleate and precipitate on the CuO layer to form the CuO



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NWs. Therefore, the different vapor pressure of CuO will lead to various morphologies of CuO NWs at different oxidation temperatures. However, the vapor pressure of CuO at 400 °C is only $\sim 10^{-9}$ Torr [13]; even if all the CuO molecules in vapor participate in the formation of NWs, the growth rate is only ~ 0.005 nm/min, which is much lower than the observed value. Considering this, Shalav has modified the Yang's model [14]. They claimed that the reaction involved H₂O and O₂ at the oxide–metal interface, with Cu forming volatile CuOH molecules, to be the precursor to generate the growth of CuO NWs. However, the CuO NWs will still form in large quantities in a dry oxidation atmosphere. Nevertheless, more importantly, no volatile CuOH can be observed during the growth of CuO NWs to support the VS model.

Kumar [15] and Kaur [16] provided a different idea in the view of crystalline growth to explain the formation of CuO NWs, also known as the SAR model. The mismatch at the Cu₂O/CuO phase interface will lead to the generation and accumulation of stress. In order to reduce the surface energy, wrinkle structures and even some “pyramid” structures will be formed during the continuous growth of the oxide layers. As the oxidation reaction progresses, the compressive stress increases. When the stress accumulates to a critical value, the stress will be released at the top of the “pyramid” by forming NWs. The SAR model has been used to explain the growth of the NWs successfully, such as the growth rate and the negative correlation between the diameter and the length of the NWs. However, the SAR model can only provide a semiquantitative analysis method and cannot provide a credible explanation of the nucleation mechanism of NWs.

Here, we synthesized CuO NWs with high straightness and crystallinity by the thermal oxidation method under an air atmosphere and ambient pressure. Both the nucleation and the growth mechanisms are investigated using diffusional solid-state transformation theory and short-circuit diffusion theory, respectively, and the corresponding equations are established to quantitatively analyze the experimental data.

2. Materials and Methods

2.1. Preparation

Cu foils were immersed in 1 M HCl aqueous solution (Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) for 30 min to remove the oxide layers, and then washed with deionized water several times. The dry and cleaned Cu foils were calcined using a muffle furnace (Carbolite Gero, Hope Valley, UK) in air. The heat treatment was performed in the temperature range of 200–900 °C. In order to avoid the effect of water vapor on the oxidation of copper, all the heating process was performed under the condition of a relative humidity of less than 2%.

2.2. Characterization Method

The morphology and structure were observed by scanning electron microscopy (SEM; ZEISS SUPRATM field emission microscope, Carl Zeiss AG, Oberkochen, Germany) and high-resolution transmission electron microscopy (HRTEM; Tecnai G2 F20 UTWIN, Thermo Fisher Scientific, Hillsboro, OR, USA). Crystal phase analysis was characterized by X-ray diffraction (XRD; Rigaku D/max-2500B2+/PCX system with CuK α λ = 1.54 Å, Rigaku Corporation, Tokyo, Japan). The element dispersion was investigated by an energy-dispersive X-ray spectroscopy (EDS; attached to SEM), and the active energy was calculated based on the data of thermogravimetric analysis and differential scanning calorimetry (TGA and DSC; Netzsch STA 449C, NETZSCH-Gerätebau GmbH, Selb, Germany).

3. Results and Discussion

3.1. Microstructure

The morphology characterization by SEM indicates that the entire surface of the oxide scale is covered by dense CuO NWs, which are relatively aligned and perpendicular to the substrate, as illustrated in Figure 1a. Most CuO NWs have smooth surfaces and a uniform diameter along their longitudinal axial direction. Figure 1a also gives a cross-sectional view of the oxide layer, which exhibits a three-layered structure: a thick bottom layer with large, clear, and neat columnar grains (the layer under the white dashed line in Figure 1a), an intermediate layer with small and irregular grains, and a top layer of CuO NW arrays. According to previous reports and the characterization results in this paper, we can consider that the composition of the thick bottom layer is Cu_2O , the intermediate and the top layers are CuO. Figure 1b shows the crystallography and morphological structure of a selected individual CuO NW. No hollow structure is observed in the NW. A twin boundary-like structure can be seen clearly along the growth direction of the NW, which divides the NW into a bicrystalline structure. And each side of the bicrystalline NW shows well-defined crystal lattice planes. The lattice fringes with an interplanar spacing of 0.265 nm and 0.253 nm indicate the (010) and (111) crystal planes of CuO, respectively.

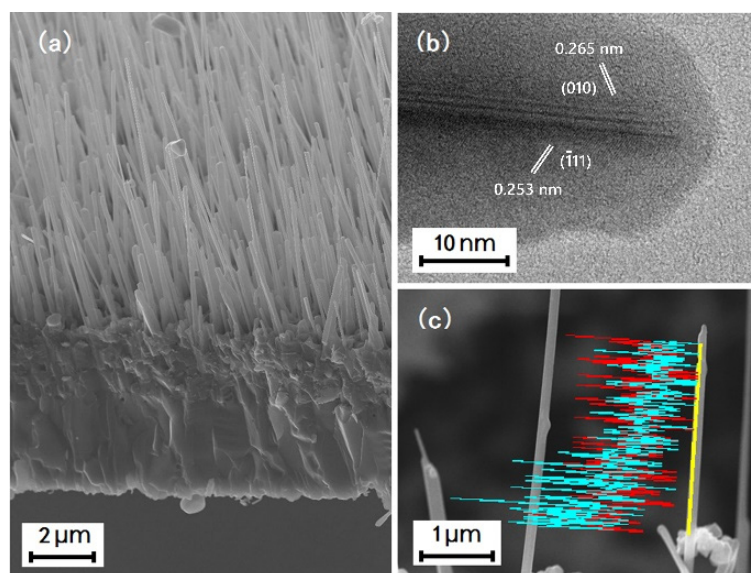


Figure 1. Electron microscopic characterizations of the sample annealed at 500 °C for 6 h: (a) SEM cross-section image of CuO nanowires; (b) HRTEM image of a randomly selected CuO NW; (c) EDS line-scan profile for Cu (blue) and O (red) along the yellow line on the NW.

3.2. Investigation of the Mechanism

Cu forms two common and thermodynamically stable oxide compounds, CuO and Cu_2O , when it reacts with oxygen [17]. The mechanism of the oxidation process of Cu has been extensively studied [18]. The growth of Cu oxides was dominated by the diffusion of Cu cation vacancies and electron holes. During the oxidation of Cu, oxygen is first chemisorbed on the surface of the oxide layer, and then oxygen is fully ionized to react with Cu^{2+} ions to form CuO molecules. This process produces a corresponding quantity of Cu^{2+} vacancies (V_{Cu}^{-2}) and electron holes (h^+) from the surface of the CuO layer [19]. V_{Cu}^{-2} and h^+ will migrate through the oxide layer to the inner interfaces. In the same way, the growth of the Cu_2O layer can also generate V_{Cu}^{-2} and h^+ on the CuO/ Cu_2O interface at the expense of CuO. This also gives a reasonable explanation for why the Cu_2O layer is thicker than the CuO layer. All Cu cation vacancies and electron holes generated at the two interfaces of

oxygen/CuO and CuO/Cu₂O eventually flow to the parent Cu for annihilation to form tiny pores dispersing at the Cu/Cu₂O interface.

In order to investigate the relationship between the growth parameters and the species diffusion process, the average length and aspect ratio of CuO NWs are calculated and plotted as functions of annealing time, and in the inset image, the square of the length of CuO NWs for the sample oxidized at 500 °C versus the annealing time is plotted. As displayed in Figure 2a, the average length of NWs is positively correlated with annealing time, and the curve in the inset image presents a quite near-linear dependency between the square of length and annealing time, indicating that the growth of CuO NW is determined by the diffusion of Cu²⁺ ions [9]. The data in Figure 2a show a good consistency with the parabolic equation:

$$L^2 = kt \quad (1)$$

where L is the length of NWs, t is the annealing time, and k is the constant.

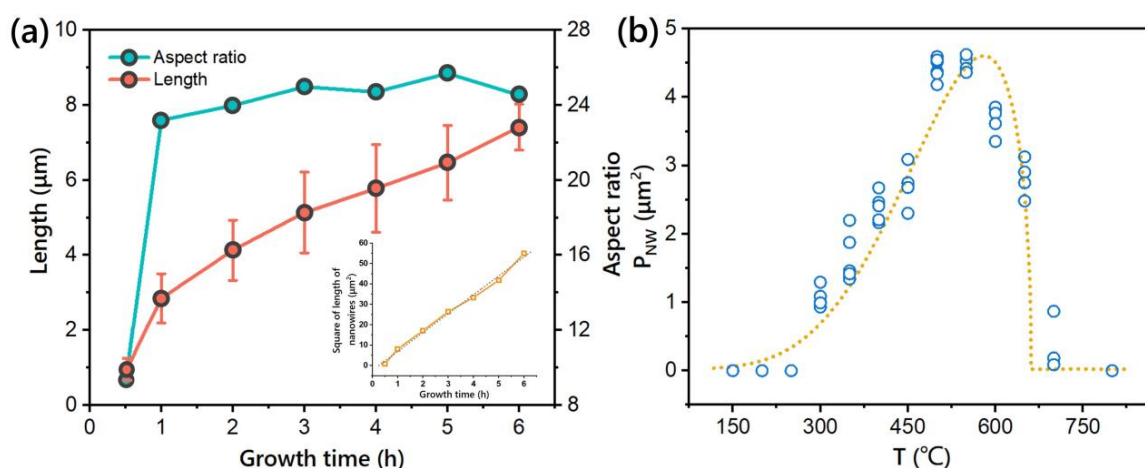


Figure 2. (a) Nanowire length and aspect ratio as functions of growth time (inset shows the square of the length of CuO nanowires (yellow solid line) and the linear fitting result (yellow dashed line) for the square-law relationship), where all the samples were annealed at 500 °C; (b) CuO nanowire density as a function of annealing temperature (small blue circles) and the fitting result with Equation (3) (yellow-dot line).

In addition, a line-scan profile of EDS on a single CuO NW has provided a visualized image of the Cu and O on a selected NW. As illustrated in Figure 1c, the signal intensity of the Cu element on the NW is strong at the root, indicating a high concentration of Cu²⁺, which gradually fades from root to top along the yellow line. However, the signal of the O element shows no remarkable change in the NW along the growth direction. It can be concluded that there exists a concentration gradient of Cu²⁺ ions from root to top on CuO NWs, while the O atoms disperse with an approximately constant concentration along the NWs. Further evidence is the “adhesion phenomena” that the nanowires glue together in parallel and cross, as shown in Figure 3a,b, which could be attributed to the fact that, during the diffusion of the Cu²⁺ flux on the surface of nanowires, part of the Cu²⁺ ion flux reacted with oxygen and “solidified” to form joints between nanowires.

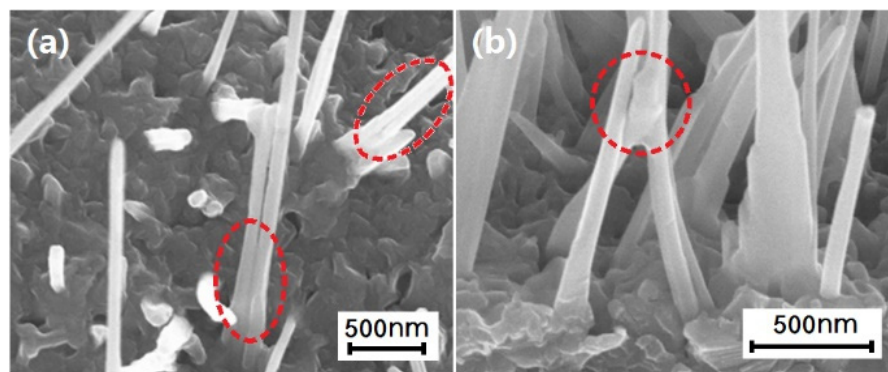


Figure 3. Two typical “adhesion phenomena” of CuO NW morphologies: (a) parallel and (b) cross. The red-dashed ellipses highlight the jointed regions between adjacent nanowires, where the nanowires reacted with oxygen and “solidified” to form joints.

According to the oxidation theory of Cu and the aforementioned evidence from our investigation, it can be identified that the growth of CuO NWs is a component of the oxidation of copper, and the whole process is diffusion-controlled. This indicates that CuO NW growth is determined by the migration of Cu cations. As oxidation involves surface diffusion and formation of oriented oxide nuclei, the formation of Cu₂O at the Cu surface and CuO at the Cu₂O surface can be treated as a heterogeneous nucleation of oxide islands induced by the reaction of gaseous oxygen with the substrate [20]. In addition, the Cu₂O is no longer an unavoidable and useless existence in the oxidation of copper, but a parent phase of the formation of CuO NW nuclei [21].

To investigate the mechanism of CuO nanowire nucleation, a statistical analysis was conducted on the nanowire growth densities, as shown in Figure 2b. It is observed that with increasing annealing temperature, the nucleation density of nanowires rises to a maximum at ca. 600 °C and then declines rapidly. Beyond 750 °C, almost no NW can be observed. Since the nucleation of CuO nanowires can be considered a solid solution precipitation process under steady-state heterogeneous nucleation conditions, solid-state transformation nucleation theory was applied. The nucleation rate I is expressed as follows [22]:

$$I = Z\beta^*N\exp\left(\frac{-\Delta G^* - E_a^{CuO}}{kT}\right) \quad (2)$$

where Z is the Zeldovich non-equilibrium factor, β^* is the frequency of an additional molecule joining a critical nucleus, N is the number of nucleation sites per unit area, and E_a^{CuO} is the activation energy for CuO molecule migration, derived from thermogravimetric analysis of Cu oxidation in air. Here, $E_a^{CuO} = 38.87 \text{ kJ mol}^{-1}$, k is the Boltzmann constant, and T is the annealing temperature. ΔG^* is the critical nucleation free energy, defined as

$$\Delta G^* = -n\Delta\mu + 4\pi r^2\gamma \quad (3)$$

This free energy consists of two parts: one is the nucleation driving force $n\Delta\mu$, where $\Delta\mu$ is the free energy change in the phase transformation reaction; the other is the surface energy of nucleation $4\pi r^2\gamma$, where γ is the surface tension. In the nucleation driving force, n represents the embryo size, which is related to the embryo radius r by the relation:

$$n = \frac{4\pi}{3}r^3\bar{\rho} \quad (4)$$

where $\bar{\rho} = \rho N_A / M$, ρ is the density of CuO, N_A is Avogadro's number, and M is the molar mass. The Zeldovich non-equilibrium factor accounts for the decay of some critical-sized nuclei to smaller sizes and is expressed as

$$Z = \left[-\frac{1}{2\pi kT} \frac{\partial^2 \Delta G^*}{\partial n^2} \right] \quad (5)$$

Substituting Equations (3)–(5) into the nucleation rate expression yields

$$\begin{aligned} J &= Z \beta^* N \exp\left(\frac{-\Delta G^* - E_a^{\text{CuO}}}{kT}\right) \\ &= \left[\frac{\bar{\rho}^2}{64\pi^2 k} \frac{\Delta \mu^4}{T \gamma^3} \right]^{1/2} \beta^* N \exp\left[\left(-\frac{16\pi \gamma^3}{3\bar{\rho}^2 \Delta \mu^2} - E_a^{\text{CuO}}\right) / kT\right] \end{aligned} \quad (6)$$

Here, for computational convenience, two approximations are made. Under experimental temperature conditions, the free energy change $\Delta \mu$ is approximated as

$$\Delta \mu \cong \frac{H_V}{T_C} (T_C - T) \quad (7)$$

where H_V is the latent heat of the reaction; T_C is the critical temperature of the phase transformation reaction. Additionally, within the temperature window for CuO nanowire growth, the surface tension γ is approximately proportional to $(T_C - T)$. For ease of derivation, a proportionality constant b is introduced, giving

$$\gamma \cong b(T_C - T) \quad (8)$$

Finally, substituting Equations (7) and (8) into Equation (6), and since the annealing time is fixed, the nanowire density is proportional to the nucleation rate. Thus, the density of nanowires P_{NW} can be expressed as a function of temperature using the steady-state nucleation model:

$$P_{\text{NW}} \propto \left(\frac{T_C - T}{T} \right)^{1/2} \exp\left[\frac{-\xi(T_C - T) - E_a^{\text{CuO}}}{kT} \right] \quad (9)$$

where ξ is a proportional constant.

As illustrated in Figure 2b, with the increasing of annealing temperature, the density of the CuO NWs increases and reaches a maximum at ca. 550 °C, and then exhibits a sharp decline from 600~750 °C. After 800 °C, CuO NWs are far too subtle to be observed even if one prolongs the annealing times or raises the temperature. The fitting result shows an excellent correlation of the experimental data by Equation (9) shows an excellent correlation between the experimental data and the theoretical curves, indicating that nucleation of CuO NWs is determined by solid solution precipitation under steady-state nucleation circumstances.

An intuitive evidence of the nucleation process of CuO NWs is also provided by SEM images. After oxidation at 500 °C for 12 min, the surface of the Cu slice has already been covered by a CuO layer with an uneven morphology and lots of small knots dispersed on the oxide scale, as illustrated in Figure 4a,b. When prolonging the annealing time to 16 min, the surface of the oxide scale becomes smoother, and the knots become remarkable and neat. The formed CuO NWs can be seen clearly on the substrate after prolonging the annealing time to 20 min (Figure 4d).

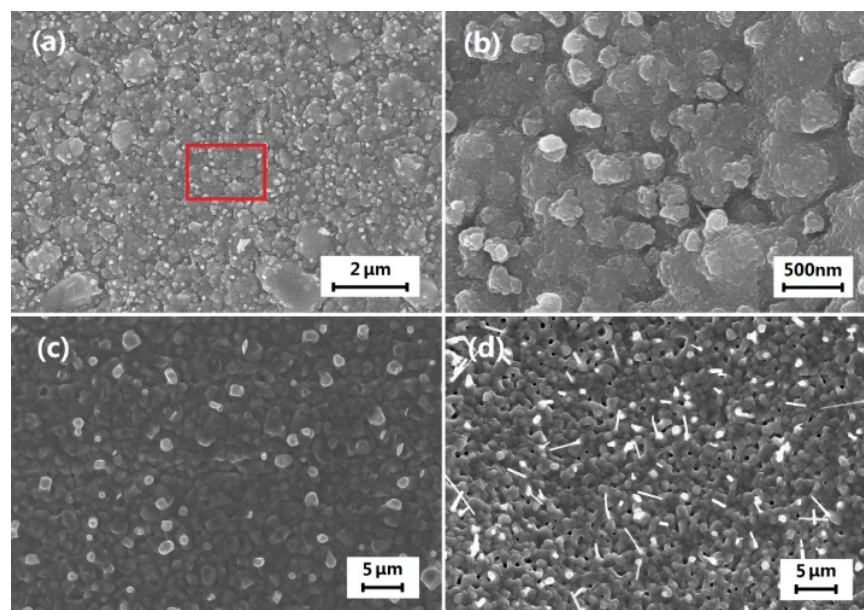


Figure 4. The formation of CuO NWs at the beginning of the oxidation of Cu: (a) 12 min, (b) enlarged SEM image of the selected area (red box) in (a), (c) 16 min, and (d) 30 min.

Regarding the strength of the Cu oxidation mechanism and our analysis, the formation mechanism of CuO NWs can be proposed completely, including a quantitative model for nucleation and growth processes. At the beginning of the oxidation of Cu, a highly protective film of Cu_2O will be formed in a relatively short time, as the reaction equivalent O_2 partial pressure of Cu_2O is much lower than that of CuO. Then, a thin film of black CuO is formed on the surface of the Cu_2O , which can be considered as a solid-state transformation process. The deposited CuO molecules continue aggregating to form monoclinic crystal nuclei. Considering the theory of crystallography, when forming the critical nucleus, monoclinic crystals usually present a rhombic prism shape with the two sharp ends, and nuclei with a certain orientation will grow along the crystallographic axis to generate nanowires, while all the other nuclei will rub shoulders with each other to construct the CuO substrate [23,24]. According to the foregoing description of the kinetics of Cu oxidation, during the growth of CuO NWs, half of the CuO grows out of the oxidation of Cu_2O , and the other moiety is owing to the fully ionized O_2 . Part of the Cu^{2+} ions migrating onto the surface of the CuO layer react with oxygen and incorporate into the CuO crystal, contributing to the growth of the oxide layer, while the remainder will be transported to support the growth of CuO NWs via surface diffusion driven by the chemical potential gradient between the substrate and the nanowire top.

4. Conclusions

We have advanced a complete quantitative kinetic model to explain the formation of CuO NWs during the thermal oxidation of Cu, which shows good agreement with the experimental results. Specifically, we obtained a successful solution for describing the nucleation process by introducing the diffusional solid-state transformation theory. The nucleation of CuO NWs is a process of solid solution precipitation under steady-state heterogeneous nucleation circumstances, which can be described by the classical nucleation rate equation. The growth process of CuO NWs is controlled by grain boundary diffusion and surface diffusion.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/cryst16030194/s1>. Figure S1: XRD pattern of CuO NWs; Figure S2:

SEM cross-sectional image of oxide scale; Figure S3: SEM images of CuO NWs in air at 500 °C for (a) 1 h, (b) 2 h, (c) 3 h, (d) 4 h, (e) 5 h and (f) 6 h; Figure S4: Thermo-gravimetric analysis of Cu slices and the derivative curves of TGA, and Arrhenius plot of $\ln(dw/dt)$ versus the inverse annealing temperature ($1/T$).

Author Contributions: Conceptualization, C.J.; methodology, C.J. and J.L.; validation, J.L.; investigation, J.L. and X.L.; resources, X.L.; data curation, Y.L.; writing—original draft preparation, C.J.; visualization, Y.L.; supervision, X.L. and X.B.; project administration, C.J.; funding acquisition, C.J. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

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