Chapter

Ultrathin Metal Hydroxide/Oxide Nanowires: Crystal Growth, Self-Assembly, and Fabrication for Optoelectronic Applications

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Abstract

The fundamental understanding of transition metal oxides nanowires’ crystal growth to control their anisotropy is critical for their applications in miniature devices. However, such studies are still in the premature stage. From an industrial point of view, the most exciting and challenging area of devices today is having the balance between the performance and the cost. Accordingly, it is essential to pay attention to the controlled cost-effective and greener synthesis of ultrathin TMOS NWs for industrial optoelectronic applications. This chapter provides a comprehensive summary of fundamental principles on the preparation methods to make dimensionality controlled anisotropic nanowires, their crystal growth studies, and optical and electrical properties. The chapter particularly addresses the governing theories of crystal growth processes and kinetics that controls the anisotropy and dimensions of nanowires. Focusing on the oriented attachment (OA) mechanism, the chapter describes the OA mechanism, nanocrystal’s self-assembly, interparticle interactions, and OA-directed crystal growth to improve the state-of-the art kinetic models. Finally, we provide the future perspective of ultrathin TMOS NWs by addressing their current challenges in optoelectronic applications. It is our understanding that the dimension, and single crystallinity of nanowires are the main contributors for building all functional properties, which arise from quasi-1-D confinement of nanowire growth.

Keywords: transition metal oxides, ultrathin nanowires, optoelectronics, oriented attachment, kinetics

1. Introduction

1.1 Transition metal oxides nanowires

Transition metal oxides nanowires are known as an important class of materials with a rich collection of physical and chemical properties due to their superior performances based on quantum confinement effects for various general applications, including electronic devices, optical devices, gas sensors, photovoltaics, photonic devices, energy storage devices, and catalysts [1–5]. The fabrication of one-dimensional (1 D) nanostructures of transition metal oxides semiconductor
(TMOS) nanowires and nanorods has been recently fascinating for the next-generation high-performance “trillion sensor electronics” era for Internet of things (IoT) applications, mainly due to their high surface to volume ratio, high crystallinity, and low power consumption [6–10]. The ultrathin nanowires that have below 10 nm diameter offer interesting characteristics including new surface determined structures by tuning the surface chemistry of surfaces [11, 12]. Furthermore, high surface area and increased colloidal stability are also inherited to ultrathin nanowires that are related to the decreased diameter of nanowires [11]. These improved overall properties of ultrathin TMOS nanowires as an ideal building block will continue the miniaturization and functional scaling of integrated circuits (ICs), nanoelectronics and optoelectronic devices by achieving the limitations of Moore’s law.

A significant research endeavor has been devoted to the fabrication of 1D metal hydroxide/oxide nanowires. However, their controlled fabrication to tailor the shape, size, crystallinity, and anisotropy is remaining a challenge. Tremendous efforts are needed to devote to the development of effective, greener fabrication methods that has scalability, reproducibility, and stability of nanowires for the successful commercialization and integration of devices. The fundamental in-depth understanding of guiding principles such as crystal growth mechanisms, kinetics, and phase transformation during the fabrication of metal–hydroxide/oxide nanowires with different strategies is crucial to accomplish this goal for promoting TMOS nanowire-based optoelectronic devices. In this review, first, we give a comprehensive overview of different synthesis strategies of transition metal-oxide/hydroxide nanowires and their electrical and optical properties. Then we provide the fundamentals of crystal growth mechanisms and a detailed overview of oriented attachment (OA), crystal growth mechanism that makes anisotropic nanowires. We subsequently discuss the state-of-the-art kinetic models that explain the OA crystal growth mechanism. Then we present a greener facile synthesis approach for the fabrication of ultrathin copper hydroxide/oxide nanowires and their optoelectronic properties. Finally, we provide an outlook of the challenges of future prospective to fabricate ultrathin transition metal hydroxide/oxide nanowires for optoelectronic applications.

1.2 Synthesis strategies of transition metal oxide nanowires

The bottom-up techniques are prominent to produce transition metal oxide NWs due to the high purity of the product, low-cost fabrication, and dimension controllability [13]. The controlled fabrication of transition metal hydroxide/oxide nanowires can be done with either vapor or solution phase growth strategies. Shen and coworkers have summarized different 1-D metal oxide nanostructures including nanowires, nanobelts, nanorods and nanotubes synthesized using both vapor or solution phase growth strategies [14]. However, the controlled pressure of the inert atmosphere and high-temperature vapor-phase approaches including vapor phase growth such as vapor–liquid–solid (VLS), solution–liquid–solid (SLS), vapor–solid–solid (VSS) or vapor–solid (VS) process, physical vapor deposition (PVD) and chemical vapor deposition (CVD) are expensive and need sophisticated instruments [15–18]. Therefore, solution-based wet chemical strategies such as hydro-thermal method, thermal decomposition, electrochemical methods, solvothermal methods, sol–gel routes became popular as they are inexpensive, energy savers, excellent control over size and morphologies, with ease of larger-scale production [19].

The wet chemical routes can be performed through precipitation or oxidation of the precursor by the aid of catalyst or surfactants and the aid of heating in an oxygen-rich environment to make metal oxide/hydroxide NWs [20]. In a hydro-thermal route, heating the precursor solution/substrate and then annealing in an oxygen environment is required to form metal hydroxide/oxide nanowires [19].
Solvothermal methods are performed by heating a metal precursor solution to a high temperature with the presence of a solvent [21]. Microwave-assisted methods are another powerful approach that require heating to a higher temperature in a microwave [22]. However, these most wet chemical growth strategies require expensive chemicals, heating procedures, longer reaction times or templates or impurities that needs to remove after the procedure [20].

The sol–gel method is a green and low-temperature method, which is widely employed to make homogeneous, highly stoichiometric and high-quality metal oxide/hydroxide nanowires in a larger scale production [23, 24]. It is often used to fabricate size and shape-controlled nanostructures starting from a metal salt as the precursor and catalyzed by base or acid to form an integrated network or gel. This method is popular to make different solid networks such as inorganic hydroxides/oxides, organic polymers, organic–inorganic hybrids, and composites due to its advantages such as high yield, better reproducibility, low operation temperature and low-cost method of highly stoichiometric and homogeneous products [25–27]. The sol–gel process can be defined as the transition of a liquid solution “sol” into a solid “gel” phase, involving both physical and chemical reactions such as hydrolysis, condensation, drying, and densification, as shown in Figure 1 [28]. When preparing metal hydroxides/oxides, first, the sol is formed from the hydrolysis of metal precursors. Sol is a stable dispersion of colloidal particles in a liquid solution and particles can be amorphous or crystalline. Then the gel is formed through condensation, polycondensation and aging to form a gel network using metal–oxo–metal or metal–hydroxy–metal coordinate bonds. Drying and densification involve densifying the gel by collapsing the porous gel network.

The morphology is influenced either due to different surface energies of the crystal faces or the external growth environment, which combines with different factors such as the precursor to base concentration, solvent polarity, temperature, and crystal growth mechanism. Different types of metal oxides have been synthesized using sol–gel route, exhibiting different 1D morphologies such as nanorods, nanoplatelets, and wires [29–32]. The conventional sol–gel process is hydrolytic, as shown in Figure 2 and oxo ions are originated from water in the reaction medium. When using organic solvents as reagents in the medium, they are nonhydrolytic sol–gel process pathways and the oxygen atoms are originated from the organic O-donor [25, 33–35]. However, both sol–gel processes have their limitations with different metal precursors [25, 35].

1.3 Electronic and optical properties of transition metal oxides

Transition metal oxides offer very diverse and fascinating electrical and optical properties of materials, which arise from the outer d electrons of the transition metal ions. Many transition metals can form binary oxides of the formula $M_x O_y$. The range of electrical conductivity of transition metal oxides is wide and varied from metals to semiconductors and insulators. A few examples of transition metal oxide insulators are CaO, NiO, TiO$_2$ and the semiconducting materials are FeO, ZnO, and CuO, while TiO, NbO, CrO$_2$, ReO$_3$ show the metallic properties

**Figure 1.**
Steps of a typical sol–gel process.
Nanostructured Materials - Classification, Growth, Simulation, Characterization, and Devices

\[ \begin{align*}
\text{Hydrolytic sol-gel formation} \\
\equiv & \text{M-OR} + \text{H}_2\text{O} \rightarrow \equiv \text{M-OH} + \text{ROH} \quad \text{Hydrolysis} \\
\equiv & \text{M-OH} + \equiv \text{M-OR} \rightarrow \equiv \text{M-O-M} \equiv + \text{ROH} \\
\equiv & \text{M-OH} + \equiv \text{M-OH} \rightarrow \equiv \text{M-O-M} \equiv + \text{H}_2\text{O} \quad \text{Condensation}
\end{align*} \]

\[ \begin{align*}
\text{Nonhydrolytic sol-gel formation} \\
\equiv & \text{M-OR} + \text{M-X} \rightarrow \equiv \text{M-O-M} \equiv + \text{R-X} \quad (\text{X} = \text{OR, Cl, OOCR}) \\
\equiv & \text{M-OR} + \text{RO-M} \rightarrow \equiv \text{M-O-M} \equiv + \text{R-O-R}
\end{align*} \]

Here, \( M \) denotes for metal.

Figure 2. The formation of metal hydroxides/oxides via hydrolytic and nonhydrolytic sol–gel process.

respectively [36]. Every 3d transition metals monoxides are widely used in different applications due to their higher abundance and low cost compared to 4d and 5d transition metal oxides.

Among transition metal oxides, copper oxide has received considerable attention in recent years as an alternative element for expensive silver and gold due to its second-highest electrical conductivity and higher abundance [37]. Cupric oxide (CuO) is a p-type semiconductor with a narrow and indirect energy bandgap of \(~1.2 \text{ eV}\) [36]. Copper hydroxide (Cu(OH)$_2$) is the hydroxide form, having an indirect bandgap of 1.97 eV [38, 39]. Zinc oxide (ZnO) is also a widely used and studied material, which is composed of the next element to Cu in the periodic table. It is an n-type semiconductor with a direct wide bandgap of 3.3 eV [40]. The electronic properties have been widely studied for CuO and ZnO nanostructures [41–43]. By fabricating smoother surfaces with minimum defects of metal oxide dielectrics can utilize their good electrical insulation without compensating its high \( k \) [44]. Since metal oxides are typically wide bandgap materials, they show excellent optical properties. For example, ZnO nanostructures have been mainly reported for laser diodes and LEDs that is the potential to operate at room temperature, owing to their higher exciton binding energy [45–47]. Ye Zhao et al. reported the optical properties of MoO$_3$, exhibiting a wide optical band gap of \(~3.05 \text{ eV}\) [48]. The controlled hydrothermal synthesis of ZrO$_2$ 1D nanostructures has shown optical properties, which are suitable in light-emitting devices [49]. In addition, CuO, NiO, SnO, and Ta$_2$O$_5$ are also have shown optoelectronic properties [10, 50–53].

2. Crystal growth mechanisms and kinetics

2.1 Oriented attachment (OA) mechanism

The nonclassical mechanism, named oriented attachment (OA) is the most common crystal growth mechanism to understand the aggregation-based crystal growth of materials at the nanoscale. It is the self-assembly of adjacent nanocrystals to form a secondary crystal through Brownian motion by sharing a common crystallographic orientation (Figure 3(a)) [54, 55]. This OA-based crystal growth was first described in 1998 for hydrothermally synthesized TiO$_2$ nanocrystals. Penn and coworkers observed the anisotropic chain of TiO$_2$ anatase crystals attachment across the \{112\} facets using a high-resolution transmission electron microscope (HR-TEM) [54, 56–58]. The OA-based crystal growth is governed by thermodynamics as the result of the reduction of total crystal surface energy [54, 59]. Highly ordered monocristalline materials can be formed through OA, which is a versatile approach for the preparation of anisotropic 1D nanowires and nanorods.
As described by Moldovan et al., the primary nanocrystal colloids in a solution rotate for a crystal match to achieve a perfect coherent grain–grain interface in nearby crystals and start coalescence of nanocrystals, eliminating the common grain boundaries to form a single nanocrystal, shown in Figure 3(b) [60, 61]. This model is named as grain-rotation-induced grain coalescence (GRIGC) mechanism to describe the crystal growth process of OA-based nanomaterials [62]. This mechanism is based on the reduction of the crystal surface energy by minimizing the area of high-energy surfaces. Leite et al. have observed the OA mechanism experimentally in the growth process of SnO$_2$ nanocrystals at room temperature [63]. With the recent advancement of liquid-phase high-resolution transmission electron microscopy (liquid phase HR-TEM), Li and coworkers directly observed the OA mechanism of iron oxyhydroxide nanoparticles [64].

The ultrathin nanowires produced by the OA process provide unique features such as constant nanowire diameter during the growth by direct attachment of nanocrystals to the tip of the growing nanowire similar to polymerization reactions [65]. Therefore, the diameter of the nanowire can be predetermined by the diameter of the nanocrystals, which are monodispersed. However, the disadvantages of OA-based nanowire synthesis methods are poor yield and having residues of ligands and solvents attached to the nanowire [11].

2.2 Crystal growth kinetic models and prior arts

The crystal growth kinetics is mainly depending on the nature of the material, interface of crystal facets, working temperature, the type of surrounding solution and the concentration of the solution [66]. In a colloidal solution, the crystal growth mechanism for the formation of metal and metal hydroxide/oxide microstructures is often explained by Ostwald ripening (OR) theory. The OR crystal growth is controlled by diffusion, where larger particles grow at the expense of smaller particles [56, 57, 67]. The kinetic model of the OR mechanism is LSW kinetic model, which is attributed to the first-order chemical reactions, as in Eq. (1) [68, 69].

$$D^n - D_0^n = k(t - t_0)$$

where $D$ and $D_0$ are the mean particle sizes at time $t$ and $t_0$, $k$ is a temperature-dependent rate constant, $n$ is an exponent relevant to the coarsening mechanism.
Three kinetic models were developed to explain the OA-based crystal growth of nanocrystals based on their diameter \([69, 70]\). These models can be categorized based on the collision between two primary nanoparticles \((A_1 + A_1 \text{ model})\) or a primary particle and a multilevel particle \((A_1 + A_i \text{ model})\) or two multilevel particles \(((A_i + A_j) \text{ multistep kinetic model})\), as shown in Figure 4(a)–(c), respectively. The aggregation of nanocrystals using the OA mechanism is described in these models using the time evolution size distribution of nanocrystals, based on the Smoluchowski Equation \([69]\). These population growth matrixes of OA kinetic models account for nanostructures with different diameter sizes. The simplest growth model is \(A_1 + A_1 \text{ primary particle model}\) and it can be stated in Eq. (2) \([71]\).

\[
d = \frac{d_0 \left( \sqrt{2} k t + 1 \right)}{\left( k t + 1 \right)}
\]

where \(d_0\) is the mean diameter at time \(t = 0\) (primary nanoparticle), \(d\) is the mean diameter at time \(t\) (secondary nanoparticle).

The prior arts of these existing kinetic models that were used to understand the crystal growth mechanism by fitting the experimental observation of synthesis of different nanocrystals have summarized in the following Table 1. As we can see in these studies, most often, both OR and OA mechanisms occur simultaneously or coexist in the same model. Zhang and coworkers found that the solo OA mechanism causes to grow the nanocrystals by hindering OR mechanism at the initial stages by introducing surfactants to strongly adsorb them onto crystal surfaces \([66, 74]\). Moreover, Zhuang et al. observed that the OA mechanism become dominated under unsaturated solutions by suggesting that OR mechanism can be thermodynamically prohibited without having enough concentration gradient to dissolve nanoparticles in a surfactant-free hydrothermal synthesis route \([76]\).

These state of the art of OA-based nanostructures show that all the existing kinetic studies have been performed based on the nanoparticle’s diameter growth using the modified Smoluchowski equation. In addition, two reports have attempted to explain the kinetic rate for the elongation of 1D nanorods by considering dipole attraction for their alignment \([77, 84]\). The in-depth understanding of the kinetics of crystal growth mechanisms for the fabrication of 1D nanostructures is still in its preliminary stage although there is rapid progress in fabricating semiconductor nanowires. Therefore, it is vital to develop new kinetic models for

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**Figure 4.**
The illustration of (a) \(A_1 + A_1 \text{ model}\), (b) \(A_1 + A_i \text{ model}\), and (c) \(A_i + A_j \text{ model}\) (\(A_i\) is a primary particle and \(A_i\) and \(A_j\) are multilevel particles; \(n = 1, 2, 3, 4, \ldots\)).
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3. Greener synthesis approaches for fabricating ultrathin metal hydroxide/oxide nanowires for optoelectronic applications

Due to the remarkable physical properties of 1D transition metal hydroxide/oxide nanostructures, greener fabrication of anisotropic metal hydroxide/oxide nanowires has attracted increasing attention in many applications. Yang et al. summarized the recent efforts of controlled synthesis of metal oxide and hydroxide 1D nanostructures such as NiO nanorods and Co$_3$O$_4$ nanowires via hydrothermal route for high-performance electrochemical electrodes and catalysts [19]. A wet chemical synthesis method was demonstrated the fabrication of ZnO nanorods, with a diameter of ~15 nm by oriented attachment mechanism [86]. Very recently, our group introduced a versatile sol–gel synthesis combined with the solvothermal process to make ZnO nanorods for optoelectronic devices [87]. Ultrathin ZnO nanorods with a diameter of ~7 nm were fabricated using a modified solvothermal route [88]. These nanorods show a strong UV band edge emission suggesting the applications of photoelectric nanodevices. Furthermore, Chaurasiya et al. synthesized TiO$_2$ nanorods using a wet chemical method for photovoltaic and humidity sensing applications [89], while MnO$_2$ nanorods were fabricated using a hydrothermal synthesis method for supercapacitor applications [90]. The work of hydrothermally growth GaOOH nanorods in width of 200–500 nm presented a low-cost and large-scale production strategy to prepare nanorods for practical applications [91].

Many works have been reported for the greener synthesis of 1D Cu(OH)$_2$ and CuO nanostructures [92–96]. However, only a very few reports have demonstrated
cost-effective and efficient synthesis routes to fabricate ultrathin copper hydroxide/oxide nanowires. In previous reports, ultrathin Cu(OH)$_2$/CuO NWs were synthesized using either both weak and strong bases [97] (i.e., aqueous ammonia and NaOH/KOH solutions) or by the interaction between a copper complex and NaOH at the aqueous-organic interface [98]. Sundar et al. demonstrated a bio-surfactant assisted synthesis method to produce CuO nanowires, suggesting the crystal growth process follows the oriented attachment mechanism [99].

Very recently, our group fabricated scalable and reproducible ultrathin CuO nanowires from self-assembled ultrathin Cu(OH)$_2$ nanowires, using a facile, greener and surfactant-free sol–gel approach, as shown in Figure 5 [100]. As depicted in Figure 6, we observed the nanocrystals self-assembly into a certain crystallographic orientation and after 45 min stirring it forms smoother surfaces of colloidal nanowires with a uniform diameter of 6 ± 2 nm from 1 hour to 4 hours stirring time intervals. The time-dependent X-ray diffractometer (XRD) analysis is supported to identify the crystal growth plane of nanowires along the [020] facet. Upon annealing the ultrathin Cu(OH)$_2$ nanowires on the Si substrate at 300°C for 1 hour, we fabricated high aspect ratio CuO nanowires over a large area, as shown in Figure 7. The respective XRD and XPS spectroscopies confirm the chemical composition of both Cu(OH)$_2$ and CuO nanowires and their purity. The calculated optical band gaps for Cu(OH)$_2$ and CuO nanowires are 1.51 and 1.10 eV, respectively.

Wang et al. fabricated uniform ultrafine Cu(OH)$_2$ and CuO nanowires using a simple wet chemical route for lithium-ion batteries [101]. Another study reported the aqueous phase synthesis of Cu(OH)$_2$ nanowires with diameters of about 10–20 nm [102]. They have initiated to study the OA crystal growth mechanism of Cu(OH)$_2$.
NWs, utilizing the power of high-resolution transmission electron microscopy (HRTEM) analysis. Very recently, Bhusari et al. reported the sol–gel approach to preparing Cu(OH)$_2$ NWs with a maximum diameter of 30 nm by varying the pH and temperature of the solution [103]. However, there is no experimental work reported for in detail investigations of their crystal growth mechanism, reproducibility, scalability and optimization of reaction parameters to get desired dimensions of nanowires for real applications in optoelectronics.

4. Summary and future prospective

One-dimensional ultrathin transition metal hydroxides/oxide nanowires are ideal building blocks for the miniaturization of next-generation high-performance integrated circuits (ICs), nanoelectronics and optoelectronic devices due to their quantum confinement and high aspect ratio. Although, we have witnessed the rapid progress and significant achievements of TMOS nanowire fabrication, still there are certain issues that need to be addressed.

1. Lack of better understanding of the guiding principles that control the size and shape of anisotropic nanowires.

It is crucial to develop more efficient, greener, scalable, and reproducible synthesis methods that can control the size of the nanowires. Aggregation of ultrathin nanowires is the major challenge of most available solution-based synthesis methods that are used to fabricate metal hydroxides/oxide nanowires. Fabrication of single ultrathin nanowires using solution-based strategies has not been studied for efficient and cost-effective devices. The controlled synthesis of ultrathin transition metal hydroxides/oxide nanowires is still in the preliminary stage and the guiding principles that control the size and shape are poorly understood and rarely explored. The existing crystal growth kinetic models have explained the crystal growth of nanostructures based on their nanoparticle diameter changes with time. These kinetic models cannot explain the crystal growth of one-dimensional nanostructures along certain specific directions. The attempts describing the kinetics of OA-based elongation of 1D nanostructures are still untapped and understudied. Therefore, exploration on crystal growth processes to generate effective novel kinetic models from experimental data will provide the prospects for the preparation of size and shape-controlled 1D nanostructures.

2. The insufficient yield and quality of NWs for device fabrication.
In general, each synthetic strategy has its advantages and disadvantages. Some synthesis gives high purity ultrathin nanowires, but the yield is poor. Many laboratory-scale synthesis routes and device fabrication are inappropriate for real commercial device fabrication due to this reason. It is challenging to develop strategies that have all the cost-effective features of commercialization. Therefore, more attention needs to pay to the different types of cost-effective fabrication methods of metal hydroxides/oxide nanowires that yield high quality and yield.

3. Lack of sufficient work for optimizing the fabrication process of NWs with desired electronic and optical properties for commercialization.

Optimization of synthesis routes to reduce the reaction time, better yield with desired dimensions of nanowires and their properties is essential to maximize the performance efficiency of NWs based devices. This is the main reason for the inappropriateness of laboratory-scale nanodevices for practical applications. The performance of optoelectronic devices can be improved by tuning the composition, dimensions, and crystal structure of TMOS NWs. These optimization studies are rarely reported for applications and this area can certainly strengthen by having strong collaborations between industries/entrepreneurs and scientists. When exploring the electronic properties of TMOS NWs, understanding their bandgap, electronic structure, electrical conductivity, dielectric constants and electron and hole mobilities are important to accelerate miniaturization and high-density integration of components of the next decade electronic devices. The factors such as light absorption, regeneration and recombination processes and charge carrier mobility of TMOS NWs are necessary to critically evaluate for future optical devices.

5. Conclusions

The controlled synthesis of anisotropic one-dimensional TMOS nanowires with versatile properties is most fascinating for different applications due to their superior performances based on quantum confinement effects and high aspect ratio. Their ultrathin TMOS nanowires have been attracted recently, however, many nanowires synthesis strategies are not sustainably produced. The fabrication of optoelectronic devices from ultrathin TMOS nanowires is clearly in its infancy. Currently, it is essential to conduct fundamental research on greener synthesis of ultrathin TMOS NWs, and the mechanisms to control the size and shape of 1D nanowires, as well as an evaluation of the optical and electrical properties of these NWs for industrial applications. Augmenting the OA crystal growth mechanism, we successfully fabricated scalable and reproducible sub 6 nm Cu(OH)$_2$ NWs and CuO NWs using a greener sol–gel hydrolysis followed by directed self-assembly and crystal growth of Cu(OH)$_2$ nanocrystals for potential electronic and optical applications. As TMOS NWs are excellent candidates for next-generation optoelectronic devices, their optical and electrical properties need to control by manipulating the fabrication conditions and fundamental understanding of controlling factors that can be optimally designed.

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Conflict of interest

There is no conflict of interest to declare.

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