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Facile controlled synthesis and magnetic properties of high-aspect-ratio nickel nanowires prepared by the dropping method

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ABSTRACT

The high aspect ratio nickel nanowires (NWs) with the different sizes and morphologies were synthesized by the improved chemical reduction method, dropping method. By simply adjusting the reaction conditions of the Ni^{2+} concentration and the precursor solution addition, the average diameter of Ni NWs can be tuned from 85 nm to 350 nm, and the length was about 40 μm. Moreover, the diameter of Ni NWs was more influenced by the Ni^{2+} concentration than the precursor solution addition and a simple empirical model has been proposed for this dependency. Meanwhile, the morphology change was also systematically investigated. Magnetic measurements showed that the diameter and aspect ratio of Ni NWs have no regular impact on the saturation magnetization and the squareness of Ni NWs. The Hc of Ni NWs was, however, a function of the aspect ratio rather than diameter, which is different from that of Ni NWs prepared by template method. The variation of Hc of Ni NWs can be described using the chain-of-sphere model.

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1. Introduction

Nickel nanowires (NWs) have been recognized as important building blocks for the fabrication of electronic devices, magnetic sensors, catalysts, supercapacitors, microwave absorbing materials, and batteries [1–7]. Since the electronic, optical, catalytic and magnetic properties of magnetic nanowires are highly dependent on the morphology of NWs, extensive efforts have been devoted to afford morphological control of NWs during synthesis [8–11]. Advanced synthesis approaches provide numerous possibilities for synthesizing novel functional NWs with precisely controlled size, structural and functional features, useful for practical applications. However, it is still a challenge faced by nanotechnology [12–15]. Several methods have been used to synthesize Ni NWs which include sol–gel method, template method and chemical reduction method etc [16–18]. Among these methods mentioned above, template synthesis is the most commonly used method for synthesizing Ni NWs due to its versatility in controlling of the growth and morphology of NWs. Unfortunately, template synthesis is complicated, high-cost and low yield, which further limit the potential application [19–22]. In this sense, a facile template-free synthesis of Ni NWs is more attractive.

Chemical reduction method has been shown to be advantageous over other methods in synthesis NWs as it is low cost, facile process, environmental friendly and high-yield [23–26]. The synthesis conditions, such as reaction temperature, initial concentration of Ni^{2+} ions, pH or external magnetic field play the critical role in determining the structures and morphologies of NWs. Without the assistance of external magnetic field, Ni et al. synthesized Ni nanorods with a diameter 8–10 nm and length of 100–200 nm [17]. By adopting the Ni ion concentration, Krishnadas et al. have successfully synthesized the Ni NWs with the diameter and length of 60 nm and 2 μm, 100 nm and 3.5 μm, 130 nm and 6 μm, respectively [27]. Using the different complexant, the nanochains with an average diameter of ~140 nm and length of ~10 μm, and the NWs with a mean diameter of ~480 nm and length of ~30 μm were prepared [28]. After optimizing the synthesis parameters, Zhang et al. have prepared the optimal Ni NWs with a diameter of ~200 nm and length up to 200 μm [26]. It can be seen that from

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these literature, most of Ni NWs have a lower respect ratio (<100), and the diameter of NWs is much larger than 100 nm, indicating that it is very difficult to control the size of Ni NWs prepared by the conventional chemical reduction method. In previous work, we have reported the synthesis of Ni NWs with a high respect ratio (>600) using the improved chemical reduction method: dropping method, and the diameter and length of the optimal Ni NWs are 70 nm and 45 μm, respectively [29].

The magnetic properties of the Ni NWs were extensively investigated by many groups, but the research efforts are mainly focused on the Ni NWs prepared by the template method [20–22,30]. For the Ni nanomaterials prepared by the chemical reduction method, the investigation of magnetic properties is concerned with the different nanostructures such as nanoflowers, nanochains, nanorings, and nanochain with hollow spheres etc [16,23,31]. However, to our knowledge, no study on the magnetic properties of Ni NWs with the variation of NW size has yet been reported in the literature.

Herein, we have systematically investigated the controlled synthesis of Ni NWs with a gradient in diameter sizes via the dropping method. To the best of our knowledge, the dropping method is a unique approach in the chemical reduction method to synthesize Ni NWs with diameter less than 100 nm and length larger than 20 μm. In addition, the effects of reaction conditions on the morphology of Ni NWs were discussed. Furthermore, the relationship between the magnetic properties and the size of Ni NWs has been investigated in detail.

2. Experimental section

2.1. Materials

Nickel chloride hexahydrate ($NiCl_2 \cdot 6H_2O$), ethylene glycol (EG), hydrazine hydrate ($N_2H_4 \cdot H_2O$, 80 wt%) and sodium hydroxide (NaOH) were purchased from Guangfu Chemical Co. Ltd. (Tianjin, China). All chemicals used in this experiment were analytical grade and used without further purification.

2.2. Ni NWs preparation

Ni NWs were synthesized by the improved chemical reduction method: dropping method [29]. Compared to the synthesis process of conventional chemical reduction method, the different of dropping method is that the $NiCl_2 \cdot 6H_2O$ in EG as a solvent was added drop by drop into the hydrazine hydrate solution as the deducing agent. Typically, 0.03 mol NaOH and 35 ml EG were mixed in a beaker. Then 11 ml hydrazine hydrate solution as a reducing agent was added rapidly into the NaOH solution. The as-prepared precursor solution was then placed into a constant temperature bath under a magnetic field at atmospheric pressure. The strength of magnetic field in the beaker is ~0.05T generated by NdFeB magnet. The $NiCl_2 \cdot 6H_2O$ dissolved in 5 ml EG was added dropwise into the precursor solution using a micropipette. After 10 min, the reaction was completed and loose grey-black flocculent product was formed on the inner surface of the beaker. After the reaction, the Ni NWs were collected and washed with double-distilled water and ethanol, then dried in a vacuum oven at 60 °C for 12 h.

In order to investigate the variation of sizes and morphologies of the Ni NWs, we synthesized the Ni NWs with different Ni^{2+} concentration (from 0.01 M to 0.125 M) and the precursor solution addition (from 0.03 ml to 0.6 ml). In addition, the Ni NWs were prepared at 70, 80, and 90 °C for understanding the influence of temperature. The experimental conditions and results are summarized in Table 1.

2.3. Characterization

X-ray diffraction (XRD) pattern of the Ni NWs was collected by a Bruker AXS, D8 Discover diffractometer with a Cu K_α radiation ($\lambda = 1.5418$ Å). The 2θ was scanned over the wide-angle range of 20–90° at a rate of 1°/min to identify the structure. Scanning electron microscopy imaging and energy-dispersive X-ray spectrometry (EDS) measurements were conducted performed using field emission scanning electron microscope (SEM, Hitachi SU8010, Japan) operating at 5 kV. For SEM measurements, Ni NWs were drop-cast on a Si substrate, aligned by magnetic field and dried. High-resolution transmission electron microscopy (TEM) and selected area electron diffraction (SAED) were carried out on a JEOL JEM 2100LaB_6 transmission electron microscope operating at 200 kV. For TEM characterization, the samples were dispersed in ethanol and dropped onto copper grids covered with a holey carbon film. The magnetic properties of Ni NWs were measured at 300 K using PPMS with a maximum magnetic field of 10 k Oe.

3. Results and discussion

3.1. Structure and morphology analysis

Fig. 1 shows a large-area SEM image of Ni NWs of S3. It can be seen that the NWs were formed exclusively, and no spherical nanoparticles occurred. The average length of Ni NWs is about 38 μm. The EDS spectrum of a single Ni NW is given in the Supporting Information (Fig. S1). Fig. 2(a) is a TEM image of S3 sample, and the inset shows the selected area electron diffraction (SAED) pattern of the Ni NW. From the TEM image it can be seen that the Ni NWs are polycrystalline, composing of lots of acicular nanoparticles and the directions of nanoparticles' long axes are different. The acicular nanoparticles are about 75 nm in length and 10–50 nm in diameter. The crystalline orientation relations of these nanoparticles are analyzed by XRD (see Fig. 2(b)). In the XRD spectrum, three characteristic diffraction peaks centered at 44.6° (111), 51.9° (200) and 76.5° (220) of crystalline Ni, respectively, which can be readily indexed to pure face-centered cubic nickel (JCPDS 04–0850, space group, Fm3m (225)). Moreover, the amplitude of the (111) peak is much higher than that of other peaks, which indicated that the (111) direction is the preferred crystal orientation, which is aligned along the wire axis. In addition, the EDS and XRD analysis show no impurities such as NiO, $Ni(OH)_2$ and other inorganic ions are observed, indicating that phase-pure Ni NWs were obtained via dropping method.

3.2. Size and morphology control of the Ni NWs

Fig. 3(a–d) shows the SEM images Ni NWs with different Ni^{2+} concentrations in solution. The dimensions were reported from the analysis of about 100 individual NWs. The results are summarized in Table 1. Average diameter of NWs prepared at Ni^{2+} concentrations of 0.01 M, 0.05 M, 0.1 M and 0.125 M were 85 ± 1.5, 140 ± 2.5, 185 ± 2.8, and 200 ± 3.7 nm, respectively. Lengths of these NWs were 24 ± 2.5, 35 ± 3.4, 38 ± 3.9, and 40 ± 4.2 μm, respectively. It can be seen that the length and diameter of NWs increase with the Ni^{2+} concentration increasing. The influence of the precursor solution addition on the dimensions of Ni NWs has been observed. The results are also summarized in Table 1. SEM images (Fig. S2(a-d), Supporting Information) showed that the relationship of dimensions of Ni NWs as a function of the precursor solution addition is similar with that versus the Ni^{2+} concentration; i. e., the higher precursor solution addition, the larger the length and diameter of Ni NWs. Average diameters of NWs synthesized at the precursor solution addition of 0.03, 0.15, 0.3, and 0.6 ml were 140 ± 2.5,

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