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Synthesis and electromagnetic shielding performance of nickel nanowires with controllable morphology

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ABSTRACT

Nickel nanowires (NiNWs) were synthesized by a simple hydrazine hydrate reduction method. The morphology of NiNWs was controlled via adjusting the molecular weight and amount of polyvinylpyrrolidone (PVP). To demonstrate practical application, NiNWs filled PVDF (NiNWs/PVDF) film was prepared and measured. The result shows that shielding effectiveness (SE) is up to 43 dB for the film with 9 wt% NiNWs (8.2–12.5 GHz), suggesting its great potential application in EMIS for today electronics.

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1. Introduction

As an important magnetic material, 1D structured NiNW has attracted increasingly attentions and interest in catalytic, microwave-absorbing, electrochemical devices and magnetic sensors due to its advantages of anti-oxidation, corrosion resistance and low cost [1–4].

Recently, some strategies have been introduced to synthesize NiNWs, such as chemical reduction, gamma irradiation, CVD, and template method [5–10]. Among these approaches, chemical reduction is a more attractive route to synthesize NiNWs because of low cost, simplicity, ease of process, less byproduct and feasibility for scalable production. Liu et al. prepared NiNWs via a reduction route under external magnetic field, and the pH value of the solution was adjusted to 13.7 by NaOH [11]. Soumare et al. synthesized NiNWs using the reduced Ru as seeds by the polyol process under an external magnetic field of 1.4 T [12]. However, the aforementioned traditional chemical syntheses require a magnetic field to direct the growth of NiNWs, or NaOH as an additive to control pH, which means complicated process, and environmental unfriendliness.

In this work, we reported a simple, modified chemical method to synthesize NiNWs in the presence of PVP without NaOH and magnetic field. Morphology of NiNWs could be controlled successfully via adjusting the molecular weight and amount of PVP. The mechanism underlying this method was analyzed in detail. Moreover, the NiNWs/PVDF films showed excellent electromagnetic interference shielding performance, and the value of SE approached 43 dB in the measured frequency range from 8.2 to 12.5 GHz for the NiNWs/PVDF film with 9 wt% NiNWs.

2. Experimental section

2.1. Synthesis of NiNWs

In the typical synthesis, 75 μl (1 M) $NiCl_2$ aqueous solution and 15 ml PVP ethylene glycol (EG) solution were mixed, and heated to 100 °C. Then, 0.5 ml $N_2H_4{\cdot}H_2O$ was dropwise added to the above solution slowly, and the reaction was kept at 100 °C for 30–60 min until the solution became colorless and the dark gray product floated on the surface. The product was washed several times

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using distilled water and ethanol, and finally redispersed into ethanol for further use.

2.2. Preparation of the NiNWs filled PVDF (NiNWs/PVDF) composite films

NiNWs were added into PVDF solution (100 mg/ml, DMF as solvent) to obtain composite coating and then cured at 90 °C for 1 h to fabricate NiNWs/PVDF films with a thickness of 60 μm.

2.3. Characterization

Morphology of NiNWs was analyzed by scanning electron microscopy (Nova Nano SEM450, FEI) and a Lorentz – Transmission Electron Microscope (JEM-2100F, Japan). The phase structure of NiNWs was characterized by an X-ray polycrystalline diffractometer (XRD) (D/Max2550V, Rigaku, Japan) and selected area electron diffraction. Thermogravimetric analysis (TGA) was performed using TA instruments Q600 under Ar atmosphere. The elemental compositions of NiNWs were identified by X-ray photoelectron spectroscopy (XPS) (VG Scientific ESCA Lab 220I-XL). EMI shielding performance was measured by vector network analyzer (E5071C, VNA, Keysight) in the frequency range of 8.2–12.5 GHz. The magnetic properties of the NiNWs/PVDF films were analyzed using Physical Property Measurement System (PPMS) at room temperature.

3. Results and discussion

Fig. 1(a) and (b) show that 1D structured NiNWs are synthesized successfully by the simple chemical reduction method. PVP as surfactant forms a capping layer on the surface of NiNWs, as shown in the inset of Fig. 1(b). Fig. 1(c) displays that as-synthesized product is attracted by a magnet and aggregates on the wall of vial, suggesting magnetic nature of NiNWs. It is shown in Fig. 1(d) that the diffraction peaks in XRD pattern at 44.5°, 51.8° and 76.4° are consistent with the (1 1 1), (2 0 0) and (2 2 0) planes of typical face-center-cubic (fcc) nickel phase (No. JCPDS 04-0850), coupled with the SAED pattern of the product, which further confirms the synthesis of metal nickel nanowires. We can see from Fig. 1(e), no weight change is observed for NiNWs synthesized without PVP, while the significant weight loss takes place at about 380 °C due to the degradation of the remnant PVP on the surface of NiNWs [13]. XPS spectrum of NiNWs in Fig. 1(f) shows that three peaks at 529 eV, 531 eV and 533 eV are derived from O^{2-} from NiO produced by the oxidation of NiNWs, –C=O in PVP and absorbed H_2O, respectively [14]. The existence of –C=O demonstrates the interaction between PVP and NiNWs during the growth of nanowires.

Evidence of morphology-controlled synthesis of NiNWs is provided by SEM images in Fig. 2. Fig. 2(a)–(c) show that a few thorns appear on the surface of NiNWs synthesized with 0.03 g of PVP-55000, while disappear as the amount of PVP-55000 is increased to 0.50 g and 1.00 g. For NiNWs synthesized with PVP-1300000, dense and sharp thorns are generated on the surface of NiNWs and reduce slightly with PVP-1300000 increased from 0.03 g, 0.50 g to 1.00 g, as depicted in Fig. 2(d)–(f). When PVP-1300000 mixed with same weight of PVP-55000 serves as surfactant to control synthesis of NiNWs, less thorns form on the surface of NiNWs and reduce further with the content of the mixed PVP increased from 0.03 g, 0.50 g to 1.00 g, as shown in Fig. 2(g)–(i). Besides, Fig. 2 displays that the diameter of as-synthesized NiNWs decreases slightly with the increase of PVP regardless of the molecular weight of PVP. These observations demonstrate the vital role of the molecular weight and amount of PVP as capping agent in morphology-controlled synthesis of NiNWs.

Fig. 3 schematically illustrates the growth mechanism of NiNWs controlled with PVP. At the initial stage of reaction, the addition of $N_2H_4{\cdot}H_2O$ would form a soluble complex with Ni^{2+} ions in solution, during which N_2H_4 acted as a bridging bidentate ligand interacting with Ni^{2+}, as depicted in step 1. Meanwhile, N_2H_4 as reductant reduced Ni^{2+} ions into Ni atoms. Newly formed Ni atoms grew into

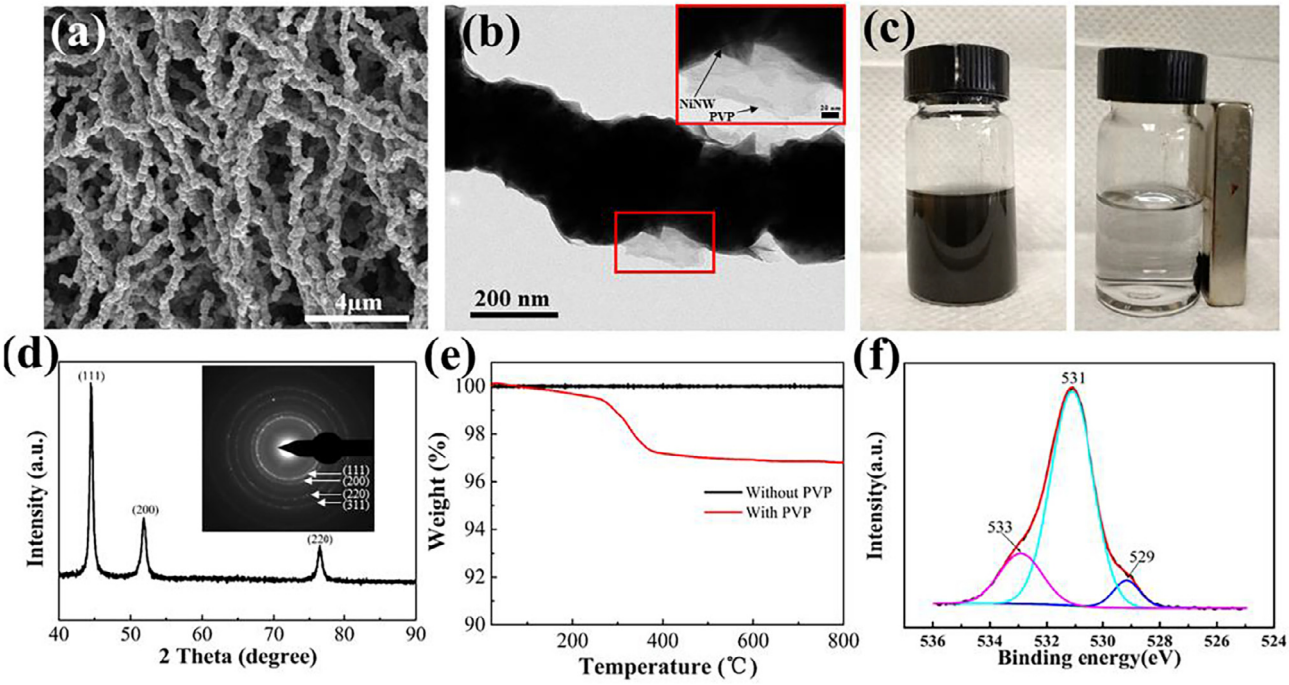

Fig. 1. (a) SEM and (b) TEM images of NiNWs. The inset in (b) is the local magnification of NiNW from the red rectangular area. (c) Digital photograph of as-synthesized NiNWs. (d) XRD and SAED pattern of NiNWs. (e) TGA curves of NiNWs synthesized with PVP and without PVP. (f) XPS spectrum of as-synthesized NiNWs. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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