Ceramics International 42 (2016) 12092–12096

Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

Preparation of hierarchical leaf-like cobalt and enhanced magnetic properties by a new low-temperature synthesis method

Xing Zhou [a,b,*], Wei Shen [a], Feng Cao [a], Jihang Li [a], Jiali Liu [a], Zongyuan Liu [a]

[a] School of Chemistry, Biology and Material Engineering, Suzhou University of Science and Technology, Suzhou 215009, China
[b] Shanxi Province Key Laboratory of Functional Nanocomposites, North University of China, Taiyuan 030051, China

ARTICLE INFO

Article history:
Received 2 April 2016
Received in revised form
22 April 2016
Accepted 23 April 2016
Available online 25 April 2016

Keywords:
Magnetic properties
Powders: Chemical preparation
Microstructure
Cobalt

ABSTRACT

Hierarchical leaf-like cobalt materials have been synthesized by a simple method at relatively low temperature. The product was characterized by means of XRD, SEM, EDS, and VSM techniques. The effects of temperature and cobalt acetate amount on the final Co were investigated by a series of experiments. It was found that the temperature played an important role in the formation of such novel leaf-like cobalt. When the reaction temperature of the mixture was as low as 40–65 °C, the morphology of final products can be changed from fluffy like to leaf like hierarchical structures. The leaf-like cobalt possessed hexagonal close-packed (HCP) phase structure. The hierarchical leaf-like cobalt exhibited high saturation magnetization (M_s) of 151.6 emu/g and coercivity (H_c) of 158.5 Oe. The low temperature chemical reduction method is quite simple, it will provide possibility for large scale preparation of such leaf-like cobalt. Due to the specific structure and magnetic properties, these cobalt leafs are expected to have potential applications as candidates for microwave absorption and sensors.

1. Introduction

Over the past few years, three-dimensional (3D) hierarchical nanostructure materials have attracted considerable research interest due to their unique structural, suggesting potential application in magnetic, electronics, and other areas [1–4]. One of the most important challenges in three-dimensional hierarchical nanostructure is the synthesis and control of the micro-structures. For example, nanoparticle-, nanowire-, nanocone-, and nanorod-based hierarchical materials have been produced [5–8]. Thus, micro–nano structures were commonly reported in previous studies.

Magnetic materials are very attractive for a range of applications that include magnetic information storage, biomedical research, solar energy transformers and microwave absorptions. Besides that, there are also potential applications in catalysis and medical diagnostics [9–18].

As a typical magnetic metallic material, cobalt has unique physical properties which are crucial for many applications. Cobalt possesses high saturation magnetization, and high permeability could therefore be achieved. Moreover, cobalt has high Curie temperature, which results in cobalt used as candidate for microwave absorption and magnetic memory devices. Several methods have been developed so far to prepare Co with different shapes, such as mushroom like, sword-like, nanoparticles, hollow porous structures, flower-like, and so on [19–22]. However, most of these methods have some drawbacks including long-time reaction, complicated synthetic steps, and high temperature preparation. Hence, it is necessary to develop a simple and inexpensive approach to the synthesis of Co 3D hierarchical materials at a relatively low temperature.

Herein, we report a new hierarchical leaf-like cobalt material, which were synthesized under a relatively low temperature condition using hydrazine hydrate as reducing agent in the absence of any hard templates or ultrasonic equipment. The length and morphology of the Co can be controlled by the reaction temperature and concentration of initial cobalt salt. The leaf-like cobalt possesses high saturation magnetization (M_s) of 151.6 emu/g. The current synthesis method and the structures of the cobalt reported in this paper will benefit the material synthesis as well as the applications.

2. Experimental procedure

2.1. Materials and methods

All reagents were analytical grade and used without further purification. In a typical synthesis, 0.996 g of $Co(CH_3COO)_2 \cdot 4H_2O$ and 4 mL of $C_2H_8N_2$ were dissolved in 50 mL of deionized water

* Corresponding author at: School of Chemistry, Biology and Material Engineering, Suzhou University of Science and Technology, Suzhou 215009, China.
E-mail address: xzhou@mail.usts.edu.cn (X. Zhou).

http://dx.doi.org/10.1016/j.ceramint.2016.04.139
0272-8842/

with intensive stirring to form a homogeneous solution. Then 25 mL of an aqueous solution with 3 g NaOH was added dropwise into above solution at room temperature. The mixture was heated to 65 °C for 30 min under mechanical stirring. Finally, 20 mL $N_2H_4 \cdot H_2O$ (80 wt%) was added to above solution and reacted 1 h. After the reaction was completed, the resulting dark gray products were collected, rinsed three times with distilled water and EtOH, and then vacuum-dried at 50 °C for further characterization.

2.2. Characterizations

XRD pattern was recorded on a Bruker D8 Advance diffractometer with Cu Kα radiation ($\lambda = 1.5406$ Å). The scanning electron microscopy (SEM) images and energy dispersive X-ray (EDS) spectrum were obtained using a Hitachi S3400N microscope and EMAX Horiba, respectively. Magnetic measurement was carried out at room temperature with a VSM (HH-10) by saturating the sample powder in a maximum magnetic field of 10 kOe.

3. Results and discussion

Fig. 1 shows the typical X-ray diffraction (XRD) patterns of the products which are prepared at 25, 40, and 65 °C in 1 h. From the XRD results (Fig. 1(c)), it is confirmed that pure cobalt was formed at the reaction temperature of 65 °C. All the diffraction peaks can be indexed to (100), (002), (101), and (110) crystalline planes corresponding to the hexagonal close-packed (HCP) phase of cobalt (JCPDS no. 05-0727). No other impurities including CoO or Co_3O_4 were detected, indicating that the product was rather pure [20]. It should be noted that the (101) reflections is much broader, which may be attributed to the anisotropic growth of cobalt crystals. The existence of $Co(OH)_2$ (JCPDS no. 74-1057) phase when the reaction temperature was 25 and 40 °C (Fig. 1(a) and (b)) may be due to the incomplete reaction resulted by the low reaction rate.

Fig. 2 display a representative SEM image of the sample with a panoramic view, from which to leaf-like Co. The magnified SEM image (Fig. 2(b)) of select area of Fig. 2(a) demonstrate that the Co possessed hierarchical leaf-like structure, and the leaf length ranged from 6 to 9 μm. The EDS pattern shown in Fig. 2(c) confirms that the final products are Co metal. The EDS results were well consistent with the XRD conclusions.

In the present system, we find that the reaction temperature is vital in the growth of cobalt crystallites, and further determines the final morphology of the leaf-like products. Isolated irregular particles with size of millimeter level were produced when the temperature was 25 °C (Fig. 3(a)). When the reaction temperature

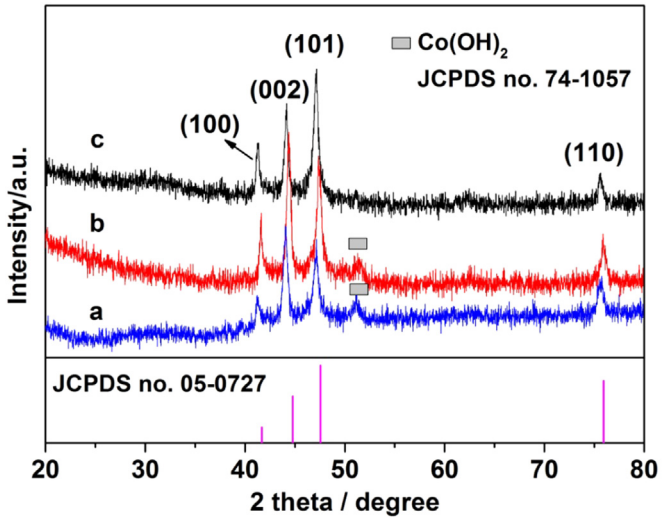

Fig. 1. XRD patterns of the products prepared at (a) 25, (b) 40, and (c) 65 °C with comparison of the standard patterns of cobalt (JCPDS card no. 05-0727).

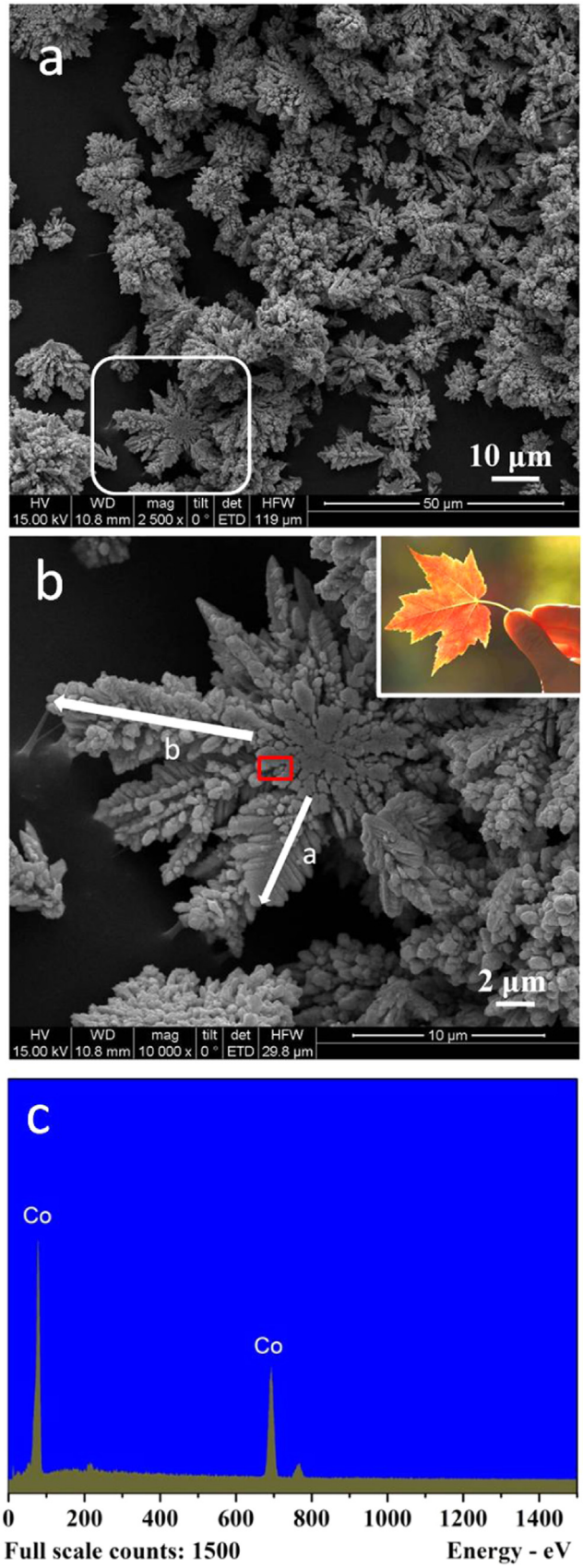

Fig. 2. SEM images (a and b), and EDS pattern (c) of the as-prepared leaf-like Co.