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Solvothermal synthesis of 3D leaf-like α-Fe_2O_3 and its gas-sensing properties research

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ABSTRACT

3D leaf-like Alpha-iron oxide (α-Fe_2O_3) nanostructure and other nanomorphologies were fabricated by adding different ethanol content in ethanol-water mixed solvent under a simple solvothermal route. The structures and morphologies of nanostructures were characterized by X-ray diffraction (XRD), field-emission electron scanning microscopy (FESEM) and transmission electron microscopy (TEM). Their morphologies and gas-sensing properties were mainly studied. The sensor synthesized in 80 vol% ethanol–water as the solvent exhibited lower operating temperature (260 °C), higher response and shorter Response/recovery time (about 8 s and 9 s). The results indicated that 3D leaf-like iron oxide (α-Fe_2O_3) nanostructure was a potential candidate for fabricating effective acetone sensor.

1. Introduction

Among various metal oxide semiconductors, iron oxide (α-Fe_2O_3) with a wide bang gap (2.1 eV) and large exciton binding energy (60 meV) is considered as one of the most stable gas sensing materials at ambient conditions [1]. In recent years, 3D α-Fe_2O_3 nanostructures have attracted much attention because of their unique properties and potential prospects in the field of gas sensing applications. These structures have several advantages such as high specific surface area, open structure feature and less agglomerated configuration, which are effective to enhance the gas sensing performance. Currently, great progress has been made on controllable synthesis α-Fe_2O_3 with three dimensional nanostructures, including nanoflowers [2], nanospheres [3] and nanocubes [4]. Among the various synthesis methods of nanocrystals, hydrothermal and solvothermal synthesis have notable advantages, such as convenience, economy, low processing temperature and so on [5–7]. But fewer by adjusting the proportion of mixed solvent to alter the morphologies of the α-Fe_2O_3 nanostructures. Thus, our study is very significant.

In this work, we report a simple solvent-regulated solvothermal synthesis of α-Fe_2O_3 nanocrystals with various 3D nanostructures morphologies and without any template. The as-prepared 3D leaf-like α-Fe_2O_3 nanostructure sensor exhibit a high response, quick response/recovery characteristics and good selectivity to acetone at 260 °C. These as-prepared products are analyzed in terms of its structures, morphologies and properties by XRD, SEM, TEM and gas-sensing testing device. In addition, the possible formation mechanism is also proposed.

2. Experimental detail

All the chemicals were analytical purity. Experiment details were as follows: 0.336 g ferrocene was added to 30 mL ethanol with four different concentrations (20, 50, 80 and 90 vol%) aqueous solution under stirring at room temperature. Then, 0.212 g Sodium chlorate was added and the whole mixture was stirred to obtain a homogeneous solution at 55 °C for 30 min. Subsequently, the homogeneous solution was transferred into a 50 mL Teflon-lined stainless steel autoclave, sealed and heated in an electric oven at 180 °C for 24 h. After that, the autoclave was cooled to room temperature naturally, the as-prepared sample was washed several times with deionized water and dried in a vacuum oven at 60 °C for 6 h. Finally, the products were annealed at 550 °C for 3 h in a furnace. For simplicity, the samples prepared with 20, 50, 80 and 90 vol% ethanol–water as the solvents were labeled as S1, S2, S3 and S4, respectively.

In this paper, the structures and the morphologies of the as-prepared samples were characterized by X-ray diffraction (XRD, D/

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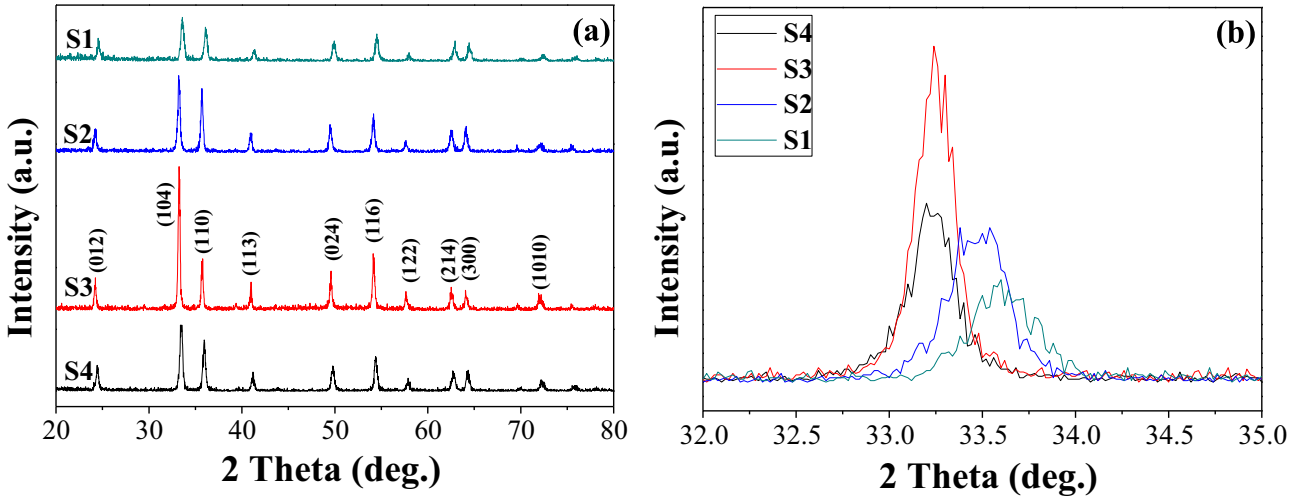

Fig. 1. XRD patterns of S1 (20 vol% ethanol–water mixed solvent), S2 (50 vol% ethanol–water mixed solvent), S3 (80 vol% ethanol–water mixed solvent) and S4 (90 vol% ethanol–water mixed solvent).

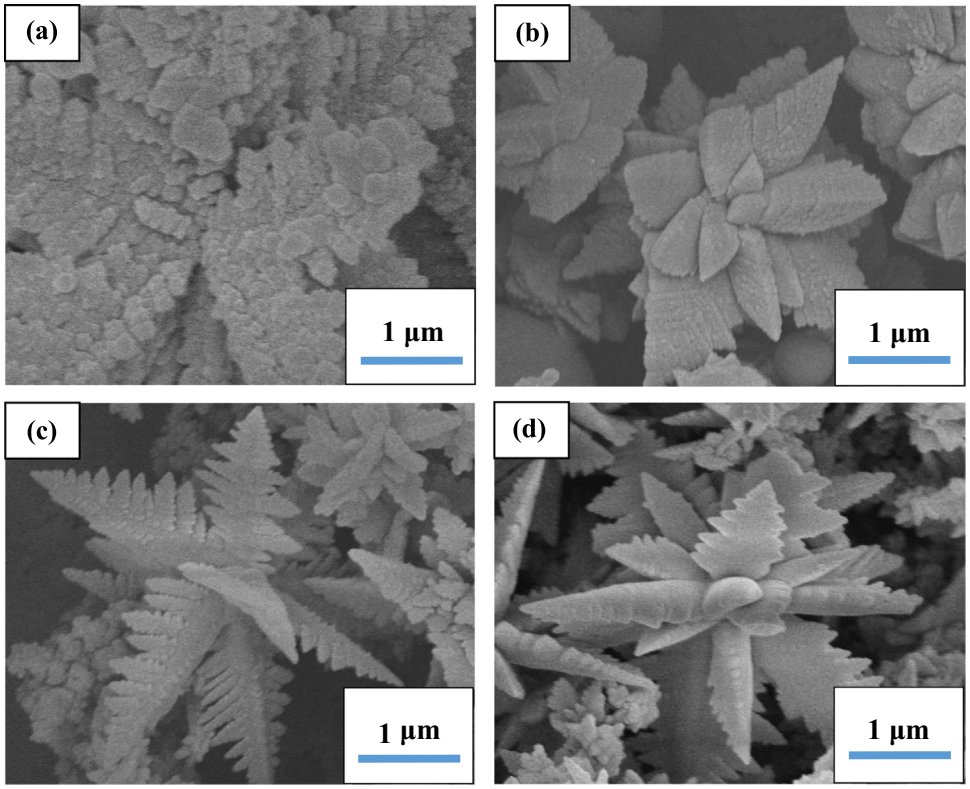

Fig. 2. SEM images of (a) S1, (b) S2, (c) S3 and (d) S4.

Max-2400), scanning electron microscopy (SEM, S-4800) and transmission electron microscopy (TEM, USA FEI TEVNAI G^2 TF20). A sensor preparation and testing system have been presented in details in our preceding papers. The sensor response (R) was defined as Ra/Rg, where Ra and Rg were the resistance of the sensor in air and in test gas, respectively [8].

3. Results and discussion

Crystallinity and phase characteristics of the products are determined by using XRD spectra of the samples, as shown in Fig. 1 (a). The results indicate that all the α-Fe_2O_3 samples are hexagonal crystal structures and these products are high purity. As seen in Fig. 1(b), the diffraction peaks of the S3 are more intensive than that of S1, S2 and S4. These imply that crystallinity degree of the S3 is the highest (in Fig. 1(b)), which may enhance the sensing response.

The SEM images of the samples synthesized at different solvents are shown in Fig. 2(a)–(d). Fig. 2(a) shows the SEM image of S1 that a large number of nanoparticles are formed, and some particles have several flower-like crystals which grow from the center of the particles. Image Fig. 2(b) clearly visible that S2 has a tapered shape along the growth direction, and regular flower-like nanostructures with good geometric symmetry begin to appear. On the condition that the ethanol content in water–ethanol mixed solvent reached 80% and 90%, The SEM images Fig. 2(c) and (d) reveals that S3 and S4 are composed of the leaf-like