Benzene-thermal Synthesis of Environmental Catalytic Material of Iron Nitride Nanoparticle

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Abstract: Environmental catalytic material of iron nitride nanoparticle was synthesized by Benzene-thermal method. In high-pressure reaction kettle, anhydrous $FeCl_3$ and Li_3N were reacted in benzene with nitrogen-flow at 450 ~ 500℃ and stirred for more than 30 h. XRD showed that Fe_4N was the main product and the average crystalline size was about 10 nm. The above reaction conditions were easy to control.

Key words: iron nitride; benzene-thermal; nanoparticle; synthesis
CLC number: O69; X5 Document code: A Article ID: 0529-6579 (2007) S1-0019-02

Iron nitrides have the properties of hardness, reflectance, abrasive-resistant, durable-refractory and catalytic activity, which are the most promising materials in widening application in magnetic, electronic and catalytic fields[1-2]. Metal nitrides are traditionally synthesized by direct reaction of the metal powder, or metal hydride with nitrogen or ammonia at elevated-temperature ($>$ 1000℃) for extended time period[3]. Now many other synthetic methods were developed, for example, chemical vapor deposition (CVD), solid-state metathesis (SSM) and benzene-thermal reaction[4]. Iron nitride particles were prepared by different methods, such as mechanical alloying[5], nitrifying iron in NH_3 gas flow[6], and chemical synthesis from $Fe(CO)_5$ and a gaseous mixture of NH_3 and H_2[7]. In this paper, nanoparticle iron nitride was prepared by benzene-thermal liquid-solid reaction of $FeCl_3$ and Li_3N at 450 ~ 500℃.

1 Experimental

All of the experimental manipulations were carried out in a nitrogen-flowing glove box. An appropriate amount of anhydrous $FeCl_3$ and Li_3N were put into the high-pressure reaction kettle, and then the kettle was filled with benzene up to 50% of the total volume. The kettle was heated to 450 ~ 500℃ and stirred for about 30 h. After cooled naturally to room temperature, the kettle was washed with benzene to remove organic substances and washed with deoxygenated water to remove soluble impurities for several times, the products were collected and dried in a vacuum drier at 60 ~ 80℃ for 2 h.

Powder X-ray diffraction (XRD) was performed on a Rigakuγ A X-ray diffractometer using Cu K_α radiation (λ = 1.54178 Å). The crystal size was calculated with Scherrer's formula.

2 Results and discussion

The composition and structure of the products were identified by XRD. A typical XRD pattern of the sample was shown in figure 1. The two strong peaks indexed as c and e were Fe_4N, which was the same as found in the literatures[9, 11], the broadening of the peaks indicated that the as-prepared products were composed of very small particles. The four small peaks indexed as a, b, d and f were peaks of impurity $Li_2Fe_3O_5$. The crystalline size of the sample was about 10 nm, as calculated using Scherrer's formula from the full-width at half-maximum (FWHM) of the diffraction peaks.

Synthesis of nanoparticle Fe_4N by benzene-thermal reaction could be a liquid-solid reaction, because anhydrous $FeCl_3$ could be slightly dissolved in benzene:

$$FeCl_3 + Li_3N \rightarrow Fe_4N + LiCl$$

Among the experiments, impurities were found to be amorphous carbon, Fe_2O_3, Fe_3O_4, $FeCl_3$ and $Li_2Fe_3O_5$, etc. When the reaction temperature was low ($<$ 400 ℃), the output and the amount of crystalline Fe_4N decreased. When the reaction temperature was rising, the amount of crystalline Fe_4N increased and

①收稿日期：2007-01-10
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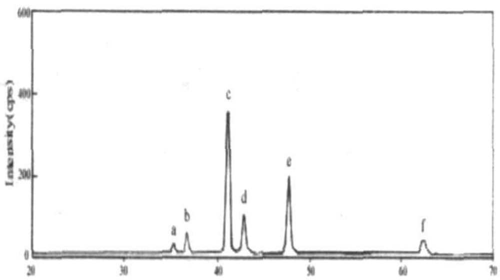

Fig. 1 X-ray diffraction of the product powders

$Li_2Fe_3O_5$ was the primary impurity. However, the amorphous carbon appeared and the size of Fe_4N powders increased along with the increasing of the reaction temperature (> 450 ℃) and reaction time (> 10 h).

3 Conclusions

Nanoparticle Fe_4N was synthesized through benzene-thermal reaction of anhydrous $FeCl_3$ with Li_3N. The optimum condition for the synthesis of nanoparticle Fe_4N was stirred at the temperature range of 450 ~ 500 ℃ for more than 30 h. The average crystalline size of Fe_4N was about 10 nm. The reaction conditions were easy to control.

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苯热法合成环境催化材料纳米氮化铁

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摘 要：采用苯热法合成环境催化材料纳米氮化铁。在高压反应釜内，无水 $FeCl_3$ 与 Li_3N 在通氮气的苯溶液和 450～500 ℃条件下反应 30 h，XRD结果表明主要产物为 Fe_4N，晶体的平均直径约为 10 nm。该反应条件容易控制。

关键词：氮化铁；苯热合成；纳米材料；合成
中图分类号：O69；X5

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V^{5+}、Ho^{3+} 共掺杂 TiO_2 光降解甲基橙

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摘 要：采用溶胶—凝胶法制备了不掺、单掺和共掺杂纳米 TiO_2，对样品进行了 XRD和 UV-Vis吸收光谱分析，并以甲基橙的光脱色反应考察了样品的光催化性能。结果发现 V^{5+} 拓宽了 TiO_2 光谱响应范围，提高了光的利用率，产生更多的电子—空穴对；Ho^{3+} 的掺杂抑制了 TiO_2 粒径的长大，引起了晶格的畸变和膨胀，延缓了光生载流子的复合；V^{5+}、Ho^{3+} 共掺杂时，由于两种掺杂物的共同作用，可进一步提高 TiO_2 的光催化性能。

关键词：二氧化钛；光催化；共掺杂；钒；钬
中图分类号：TQ146