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Preparation of Fe₂O₃/Al composite powders by homogeneous precipitation method

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ABSTRACT

The Fe₂O₃/Al composite powders were prepared by homogeneous precipitation method. The influence of the concentration of Fe²⁺ and the molar ratio of raw materials on the preparation of Fe₂O₃/Al composite powders were investigated. X-ray diffractometer, scanning electron microscope, Fourier transform infrared spectroscopy and differential thermal analysis were used to analyze the morphology and structure of the Fe₂O₃/Al composite powders. The results show that the content of iron oxide in the composite powders could be effectively controlled by adjusting the concentration of Fe²⁺ and the molar ratio of raw materials in the plating solution. The surface of Al particle was coated with a layer of thick and dense iron oxide. The core-shell Fe₂O₃/Al composite powders with Fe₂O₃ content of 14.1% were produced, the coating efficiency of Fe₂O₃ reaches more than 77%. The iron oxide, which coated on the surface of the aluminium particle is flower-like cluster structure, each flower-like cluster is constituted by nano-flaky iron oxide.

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1. Introduction

Al-Fe₂O₃ system is a typical energetic material [1–3], it can release enormous heats when the thermite reaction begins [1,4]. The Fe₂O₃/Al mixture has been used widely for the many applications. Liu et al. [5] used in-situ thermite reaction of Fe₂O₃ and Al powder mixtures to synthesize Fe-Al/Al₂O₃ composites. The Fe₂O₃/Al composite powders were used as spray coating material also have been reported [6], FeAl coating [7] and the FeAl₂O₄ coating which consisted of FeAl₂O₄, Al₂O₃, Fe and a little FeAl [8–10] prepared by reactive plasma spraying of an Al/Fe₂O₃ composite powder. Fe-Al intermetallic compounds, which have been widely used due to its excellent physical and mechanical properties [11], known as semi-ceramic materials. It can be used as the buffer layer between metal substrate and alumina ceramic coating [12]. The metallurgical bonding will form between the composite coating and substrate and it will increase the bounding strength of coating layer and substrate.

The Fe₂O₃/Al composite powders are usually prepared by mechanical aggregation method (ultrasonic dispersion method

[13], powder mixing) [14,15], self-assembly [16] and sol-gel method [1], etc. However, those methods cannot obtain the uniform and complete core-shell structure powders. The homogeneous precipitation method by urea hydrolysis is an efficient synthesis method for superfine oxides, such as TiO₂, Al₂O₃, MgAl₂O₄, Fe₂O₃ [17,18] and other composite materials, such as Fe₂O₃/graphene composite anode materials [19], Ag@Fe₂O₃ composite materials [20] and alpha-Fe₂O₃@ATO (antimony doped tin oxide) particles [21]. The compact and uniform core-shell structure powders can be got by the homogeneous precipitation method and the performance of the composite powders is stable [22,23]. Based on the related preparation methods, the homogeneous precipitation method (FeSO₄·7H₂O as raw material and CO(NH₂)₂ as precipitator) was used to prepare the core-shell Fe₂O₃/Al composite powders.

2. Experimental

2.1. Pretreatment process of the Al particles

Since the micron-Al powder can be easily oxidized in air, an oxide layer will form on the surface [24]. Thus, Al particles with an average 50 μm must be cleaned and activated. The raw Al particles were pretreated as follows: 6 g Al powders were added to 0.5% NaOH aqueous solution, sonicated for 5 min. Then the

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powders were cleaned three times with the deionized water. Finally, 0.05 mg sodium dodecyl sulfate was added into the solution to activate the surface of Al powders. The pretreatment method can increase the number of active spots on the surface of Al particles, improve the plating speed, complete the plating coat.

2.2. Preparation of Fe₂O₃/Al composite powders

In order to deposit a thick layer of Fe₂O₃ on the surface of aluminium particle, the effects of the concentration of Fe²⁺ and the proportion of urea and FeSO₄ in the plating solution were investigated. The composition of the solution and the plating conditions are shown in Table 1. The total reaction time was 3 h, the deposition temperature was 80 °C. When the solution in the beaker was heated to 80 °C, adding urea solution to the beaker (the speed was 2 mL/min). After plating, the solution was cooled to room temperature and separating the powder from the solution. After plating, the composite powders were washed with distilled water and then dried in an oven at 80 °C for 2 h. Finally, the composite powders were calcined in muffle at 400 °C for 2 h in air atmosphere to get the Fe₂O₃/Al composite powders. The heating rate was 5 °C/min.

2.3. Characterization

The microstructure and phase identification of the composite powders were carried out with a Rigaku X-ray diffractometer (DMAX-2500PC X-ray diffractometer). Surface morphology and composition analysis of the composite powders were observed by scanning electron microscope (SEM, Hitachi-S4800) and transmission electron microscopy (TEM). The thermal properties of Fe₂O₃/Al composite powders were analyzed by differential thermal analysis (DTA). In the paper, two parameters CE (Efficiency Coating) (Eq. (2-1)) and PC (Percentage of Coating) [25] (Eq. (2-2)) were used to characterize the composite powders.

$$CE = \frac{\text{Fe}_2\text{O}_3 \text{ on Fe}_2\text{O}_3/\text{Al particles(g)}}{\text{Fe}_2\text{O}_3 \text{ produced theoretically(g)}} \quad (2-1)$$

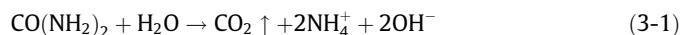
$$PC = \frac{\text{Fe}_2\text{O}_3 \text{ on Fe}_2\text{O}_3/\text{Al particles(g)}}{\text{Fe}_2\text{O}_3/\text{Al particles(g)}} \quad (2-2)$$

3. Results and discussion

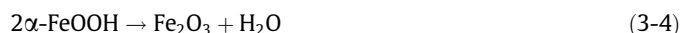
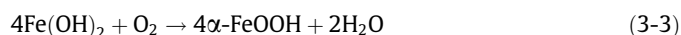
3.1. The mechanism of formation of Fe₂O₃/Al composite powders

In the homogeneous precipitation process, when the urea was slowly hydrolysis in a solution containing Fe²⁺, the generated anions (OH⁻), with metal cations (Fe²⁺), form precipitation nuclei (Fe(OH)₂). The precipitation nuclei (Fe(OH)₂) begin heterogeneous

nucleation on the surface of the Al powders, and grow up. The reactions are described as follows:



The Fe(OH)₂, as an unstable compound, will be oxidized into α-FeOOH. The reaction is described as follows:



After calcination, the α-FeOOH turn to the α-Fe₂O₃. The formation mechanism of Fe₂O₃/Al composite powders is shown in Fig. 1.

3.2. The effect of Fe²⁺ concentration on the CE and PC

Fig. 2 shows the curve of the CE and PC of the Fe₂O₃/Al composite powders under different concentration of Fe²⁺. It indicates that with the increasing of Fe²⁺ concentration, both of the CE and PC show a trend of increase before decrease. When the Fe²⁺ concentration is 0.2 mol/L, the value of CE and PC reach the maximum. The reason of this phenomenon is possibly as follows: when the concentration of Fe²⁺ is 0.1 mol/L, the saturation of the reaction system is very small, which is not conducive to the growth of nucleation. With the gradually increasing of Fe²⁺ concentration, the nucleation [26–28] rate and the crystal growth rate [29,30] increase, and the reaction becomes more easily. The ferrous hydroxide, which deposited on the surface of aluminium particles, will increase too. However, if the concentration of Fe²⁺ in the reaction system is too high, the nucleation rate becomes faster than the crystal growth rate [27], there are more spontaneous nucleation of the ferrous hydroxide rather than the ferrous hydroxide precursor precipitated on the aluminium particles. Therefore, the PC of the composite powders will decrease. Besides, the loss of free iron oxide particles in the experiment may result in the decrease of CE. Thus, the most appropriate concentration is 0.2 mol/L.

3.3. The effect of the proportion of urea and FeSO₄ on the CE and PC

With the increasing of the proportion of urea and FeSO₄, the CE and PE of the composite powders improve steadily, as shown in Fig. 3. But when the proportion of urea and FeSO₄ exceeds 20:1, the growth rate of the CE and PE becomes slow. This is because when the concentration of urea was added to the reaction system is too high, the urea hydrolysis rate [31] becomes faster, the concentration of OH⁻ increases rapidly. It would generate a lot of ferrous hydroxide in the local area, so that the amount of free ferrous hydroxide will increase and that may lead to a decrease of the PC. In addition, it will result in a greater waste of raw material and increases the cost when the ratio continue to increase. The most suitable ratio of urea and FeSO₄ is about 20:1.

3.4. Morphology of the Fe₂O₃/Al composite powders

Fig. 4(a) shows the morphology of original Al powders. It can be seen that these powders have a smooth surface. After coating, these powders' surface turn into rough. A Fe₂O₃ surface coating was formed in the surface of Al particles, as shown in Fig. 4(b).

Fig. 5 is SEM micrographs of partial Fe₂O₃/Al and iron oxide coating layer. As shown in Fig. 5(a), uniform and dense Fe₂O₃ surface coating was formed in the surface of aluminium particle. Fig. 5 (a) and (b) showed that the iron oxide which deposited on the surface of Al powders were flower-like clusters in micro scale, while

Table 1

Composition and operating conditions of the plating solution for Fe₂O₃ coating on Al powders.

Chemical composition of the plating solution and its operating conditions	
Fe ²⁺	0.1–0.3 mol/L
n(CO(NH ₂) ₂):n(FeSO ₄)	5:1, 10:1, 15:1, 20:1, 25:1
SDS	0.05 mg/L
Al	6 g
Deionized water	1 L
Temperature	80 °C
Time	3 h
Stirring speed	150 rpm

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