Preparation of nano α-alumina powder and wear resistance of nanoparticles reinforced composite coating

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Caifeng Chen *, Zhidan Ding, Qi Tan, Henghua Qi, Yonghui He

School of Materials Science and Engineering, Jiangsu University, Zhenjiang, Jiangsu 212013, China

ABSTRACT

Nanometer α-alumina powder was synthesized using the pore rich activated carbon as the reaction place, and the α-alumina nanoparticle reinforced nickel based composite coating was prepared by flash thermal spray method. The composite coating was abraded on abrasive wear-machine to study its wear resistance property. The results showed that a large number of uniform size spherical-shaped precursor particles were in the activated carbon pores, which could be long-term stable in a spherical shape and adsorbed on the pore wall. In addition, activated carbon pores could prevent the alumina particles from growing up and reuniting effectively during the precursor particles calcination process. And the activated carbon had completely burned at high temperature as well. Finally, nano α-alumina powder with good dispersion was obtained. The wear resistance of the nickel based composite coating was reinforced by the nanometer α-alumina powder. With 2% volume fraction addition of nanometer α-alumina, the coating has the best abrasive wear resistance property.

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1. Introduction

The thermal spraying technique has been widely adopted in many industries due to its flexibility and cost effectiveness. It is normally used to apply coatings to components to protect against wear, heat and/or corrosion [1–3]. Nickel based composite coating prepared by thermal spraying shows a very good wear resistance property. The composite coating consists of a metallic phase and a ceramic phase. The metallic phase, nickel in the study, acts as matrix, while the ceramic phase, nano α-alumina powder, reinforced the matrix. Nanometer α-alumina powder has good mechanical characteristic property, good oxidation and corrosion resistance and can be easily produced. Therefore, it's widely used in the fields of ceramic coating industries for thermal and special coatings [4–6].

There are lots of methods for the preparation of nanometer α-alumina, such as mechanical synthesis, vapor phase reaction, precipitation, combustion and sol–gel methods [7–9]. The liquid phase precipitation method always gains the priority, and the technology also become more and more mature. But the liquid phase precipitation method appears the phenomenon of serious powder reunion and unequal particle size distribution [10,11]. Although many methods have been adopted to prevent agglomeration and to control particle size, it's still difficult to prepare mono-dispersed and narrow size distributed nanometer α-alumina powder [12–14].

It's well known that activated carbon contains rich pores. Take the large and medium size holes as the liquid reaction place, a large number of precursor particles can generate simultaneously and begin to grow synchronously as well. Moreover, the activated carbon can be removed easily at high temperature. But up to now, there are few related reports about utilizing activated carbon for the preparation of nanometer oxide powder.

In the work, mono-dispersed nanometer α-alumina particles were synthesized using activated carbon as reaction template. The nucleation and growth process of nano particles in the template had been studied. Nickel based composite coating was prepared using thermal spraying. The addition quantity of nanometer α-alumina powder on the resistance properties of the composite coating had been investigated.

2. Experimental methods and materials

Activated carbon was prepared using charcoal block and zinc chloride as raw materials. Equal quality of charcoal and zinc chloride was mixed, and the mixture was dipped in the wet state for an hour. Then it was put into a furnace at 500 °C for 90 min. After that, the sample was boiled with 0.1 mol/L HCl solution for 20 min, and it was washed carefully with distilled water until not any chloride ion were picked out from the raffinate. Finally the activated carbon was obtained after dried at 120 °C for 12 h.

The right amount of activated carbon block prepared above was impregnated with a certain concentration of mixed urea and $Al(NO_3)_3$ solution at room temperature. It was got out after 12 h and put into the oven at 75 °C for a certain period of time. Then it was dried at 150 °C for 8 h and calcined at 1200 °C for an hour, and the nanometer α-Al_2O_3 powder was acquired.

* Corresponding author. Tel.: +86 13861399050.
E-mail address: chenjsust@163.com (C. Chen).

The α-Al$_2$O$_3$ powder and nickel base automatic molten alloy powder were mixed in ball mill grinding for half an hour. The nano α-alumina/Ni composite coating on the surface of Q235 steel was prepared by the method of oxygen–acetylene flame thermal spraying.

Microstructures of the precursor particles and powder were characterized with X-ray Diffraction (XRD, D/max2500PC), Scanning Electron Microscopy (SEM, JSM-7001F) and Transmission Electron Microscope (TEM, JEM-2100) techniques. The process of removing template as temperature changing was recorded by Thermo-Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC, STA 449C) at a heating rate of 5 °C·min^{-1} with temperature ranging from 20 °C to 1200 °C.

The wear resistance property of the composite coating was measured by the MLS-23 wet sand rubber wheel abrasion tester. The hardness of rubber wheel for shore hardness is 75, and the speed is 240 r/min. Ordinary river sand was taken as grind grain, whose main ingredient is SiO$_2$. Wear resistance of the composite coating is measured by relative abrasion resistance E, E = W$_1$/W$_2$, W$_1$ is the abrasion loss of the standard sample, while W$_2$ is the abrasion loss of the sample.

3. Results and discussion

3.1. The microstructure of the activated carbon

The micro-topography of activated carbon is shown in Fig. 1(a). From the picture, it's evident that the activated carbon contains a great number of large holes. The diameter of the holes rang from 10 μm to 30 μm. Most of them are run through holes in the longitudinal direction. There're little sediments falling on the inside and outside surfaces. A mass of nanometer pores exists on the hole wall between big holes and the inner wall of pores, just as shown in Fig. 1(a) and
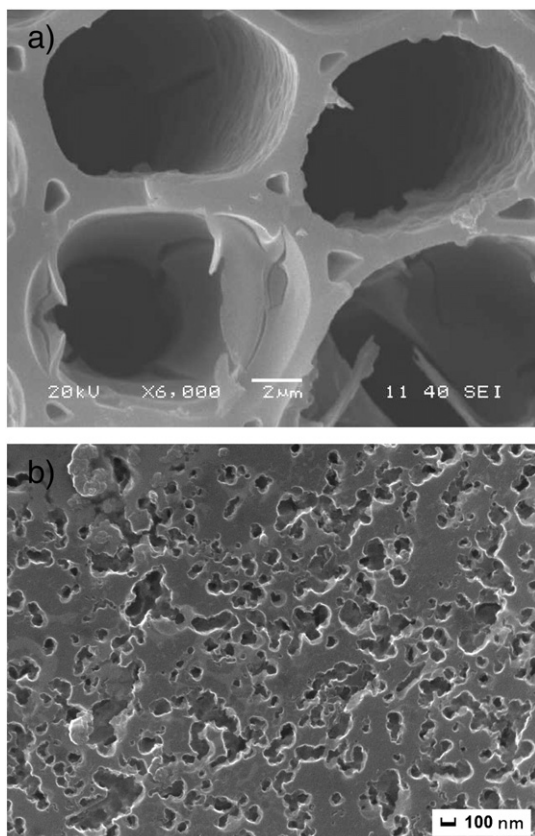

Fig. 1. SEM images of activated carbon (a: the longitudinal section and b: the microporous).

(b). All these pores offer good places for chemical reaction to prepare alumina precursor particles.

3.2. The influence of concentration of Al(NO$_3$)$_3$ on growth of precursor particles in the activated carbon pores

The activated carbon was immersed in the mixed solution of urea and Al(NO$_3$)$_3$ at room temperature. Urea and Al(NO$_3$)$_3$ do not react usually at normal temperature, but kept it at 75 °C for a certain period of time, and the abundant pores in the activated carbon acted as wonderful places for chemical reaction, the reaction processes were as follows:

$$CO(NH_2)_2 + 3H_2O \rightarrow 2NH_4OH + CO_2 \tag{1}$$

$$NH_4OH \rightarrow NH_4^+ + OH^- \tag{2}$$

$$CO_2 + 2OH^- \rightarrow CO_3^{2-} + H_2O \tag{3}$$

$$2Al^{3+} + 3CO_3^{2-} \rightarrow Al_2(CO_3)_3 \tag{4}$$

$$Al^{3+} + 3OH^- \rightarrow Al(OH)_3. \tag{5}$$

The morphology of precursor particles in the activated carbon pores under different liquid concentrations is shown in Fig. 2(a) and (b). The solution concentrations of Al(NO$_3$)$_3$ were 0.03 mol/L (in Fig. 2(a)) and 1.5 mol/L (in Fig. 2(b)). The urea concentration was 1 mol/L. The Energy Dispersive X-ray (EDX) pattern of the precursor particles was shown in Fig. 2(c).

As shown in Fig. 2(a), the shape of single particle was spherical and dispersed well when the concentration of the Al(NO$_3$)$_3$ solution was 0.03 mol/L. The particle size distribution was uniformed and highly dispersed. Most of the particles attached to or scattered on the holes wall. But when Al(NO$_3$)$_3$ solution concentration increased to 1.5 mol/L, just like Fig. 2(b) showed, in such a high concentration, a large number of precursors were generated in the pores. Except a small amount of precursor body absorbed on the wall, other precursors with massive shape had filled the entire duct. As the spectrum analysis from Fig. 2(c), it can be seen that elements Al, O, and C are in accordance with the precursor product in chemical composition.

3.3. The influence of reaction time on the growth of precursor particles

The urea occurred homogeneous decomposition and hydrolysis inside the activated carbon pores when it was at the temperature of 75 °C, the generated NH$_3$ and CO$_3^{2-}$ were distributed in the solution between pores. When the concentration of CO$_3^{2-}$ reached the critical super-saturation, and the precursor nucleated explosively. Following, the crystal nucleus captured the remnants of OH$^-$ and CO$_3^{2-}$ in the solution to grow into nanoscale primary particles. The generated primary particles would be adsorbed on the activated carbon pore wall and grew up into spherical particles by surface reaction.

As the reaction process in the activated carbon pore continuing, part of the unabsorbed, nanometer sized precursor particles collided with each other or with the ones being adsorbed on the hole wall. The particles adhered, aggregated, formed more big spherical particles and were adsorbed on the pore wall again. Fig. 3 is the micromorphology of precursor particles after the reaction lasted for 10 h. At the moment, part of the precursor particles was agglomerated, but they were still adsorbed on the pore wall.