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Ceramics International 36 (2010) 1989–1992
CERAMICS INTERNATIONAL
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Short communication

Preparation and characterization of Ni-coated Cr_3C_2 powder by room temperature ultrasonic-assisted electroless plating

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Received 6 October 2009; received in revised form 26 November 2009; accepted 7 March 2010
Available online 24 April 2010

Abstract

Ni-coated Cr_3C_2 powder was prepared by ultrasonic-assisted electroless plating process without conventional sensitization and activation treatments at room temperature. The growth mechanism of Ni layers and the effects of Cr_3C_2 powder concentration on plating rate and coating quality were studied. Surface morphologies and composition of initial Cr_3C_2 powder, pre-treatment Cr_3C_2 powder and Ni-coated Cr_3C_2 powder were characterized by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The results show that the Ni layers on the Cr_3C_2 powder had cell structure with dense and uniform distribution. The growth mechanism of Ni layers appears as follows: the pretreated surfaces of Cr_3C_2 powder act as activated sites, nucleation and the growth of nickel grains take place on the powder surfaces, and the process repeats continuously on the lath particles with reticulate structure on the as-coated surfaces of previously deposited Ni-cells, finally Ni cells grow up and merge into a layer. With the increase of Cr_3C_2 powder concentration, the plating time becomes shorter accordingly and the optimal powder concentration is 40 g L^{-1}.

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Keywords: Ni-coated Cr_3C_2 powder; Electroless plating; Scanning electron microscopy

1. Introduction

Metal-coated ceramics powders refer to the composite powders with ceramics core and metallic shell. Ceramic-matrix composites with metal particles have been developed to improve the mechanical and functional properties of ceramic materials, such as particular electrical, magnetic and chemical properties including the wetting property between metal and ceramics materials [1–4]. Electroless plating plays an important role as a metal-coated technique on ceramic substrates [5]. Electroless deposition process has undergone numerous modifications to meet the challenging needs of a variety of industrial applications since Brenner and Riddell invented the process in 1946 [6]. This technique provides distinct advantages such as uniformity of deposits on complex shapes, lower cost with simple processing. In recent years, the electroless Ni–P composite coatings have been successfully prepared on many ceramics powders such as Al_2O_3, ZrO_2, Si_3N_4, SiC and diamond powder [1,7–11]. Above studies related to the plating of Ni–P onto ceramic substrates, have usually employed the sensitization treatment with HCl + $SnCl_2$ and activation treatment with HCl + $PdCl_2$ prior to plating and the temperature of the nickel plating bath needed to maintain at 40–95 °C.

The aim of this work was to investigate the deposition of Ni onto Cr_3C_2 particle surfaces without conventional sensitization and activation steps by ultrasonic-assisted electroless plating at room temperature. The surface morphologies and composition of Cr_3C_2 powder at different stages were investigated. The effects of Cr_3C_2 powder concentration on the preparation of Ni-coated Cr_3C_2 powder were studied.

2. Experimental

2.1. Pre-treatment of Cr_3C_2 powder

The Cr_3C_2 particle size is 150–200 μm. The powder was first coarsening by immersing into an aqueous solution of 50 mL L^{-1} hydrofluoric acid (HF), 80 mL L^{-1} nitric acid (HNO_3) and 2 g L^{-1} ammonium fluoride (NH_4F). The process

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0272-8842/$36.00. Published by Elsevier Ltd and Techna Group S.r.l.
doi:10.1016/j.ceramint.2010.03.003

was carried out with reinforcement of ultrasonic wave for 25 min at room temperature to obtain a micro-rough surface, to increase the specific surface area of the particles and hydrophilic property. Afterwards, the powder was cleaned with de-ionized water for several times. The surface morphologies of initial Cr_3C_2 powder and pre-treatment Cr_3C_2 powder were examined by SEM.

2.2. Preparation of Ni-coated Cr_3C_2 powder

The pretreated Cr_3C_2 powder was put into a plating beaker immersed in the water bath with a CCW-50W ultrasonic generator, shown in Fig. 1. The bath composition is given in Table 1. Nickel sulphate is a main salt, sodium hypophosphite is the reducing agent, sodium citrate is used as a complexing agent and boric acid acts as a stabilizing agent to control pH of the bath during plating process. The chemicals were all of analytical reagent grade. Plating processing was carried out at room temperature and pH value of the bath was adjusted to 9.0–10.0 by sodium hydroxide (NaOH). After 8–15 min incubation period, the reaction started and became stronger with the solution full of bubbles. The entire reaction approximately lasted 30–55 min.

The composite powder was cleaned with de-ionized water for several times and dried in a vacuum oven at 180–200 °C for 3 h. The surface morphologies of Ni-coated Cr_3C_2 powder were observed by SEM, and the chemical composition of the Ni on the Cr_3C_2 powder was analyzed using EDS.

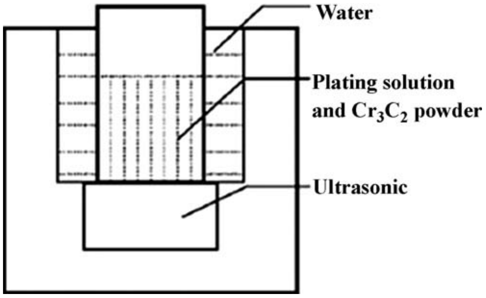


Fig. 1. Schematic diagram of the ultrasonic aided plating device.

Table 1
Composition of the electroless nickel plating bath.

Chemical	Formula	Concentration (g L^{-1})
Nickel sulphate	$NiSO_4 \cdot 6H_2O$	25–30
Sodium hypophosphite	$NaH_2PO_2 \cdot H_2O$	23–40
Sodium citrate	$Na_3C_6H_5O_7 \cdot 2H_2O$	46–63
Boric acid	H_3BO_3	26–42

3. Results and discussion

3.1. Surface morphology analysis

The Cr_3C_2 powder was subjected to a coarsening treatment prior to plating. The surface morphologies of initial Cr_3C_2 powder and as-pretreated Cr_3C_2 powder were examined by SEM in order to compare the change of morphology by coarsening treatment, shown in Fig. 2, when Cr_3C_2 powder concentration is

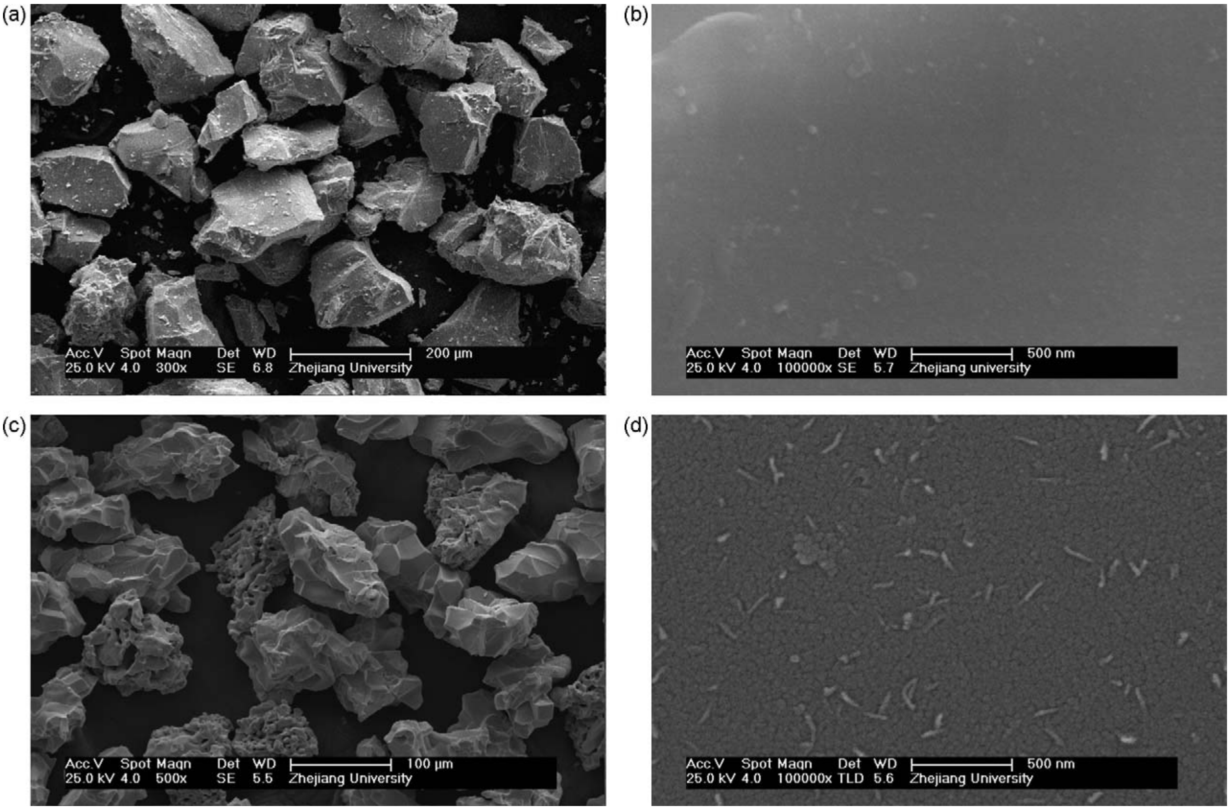
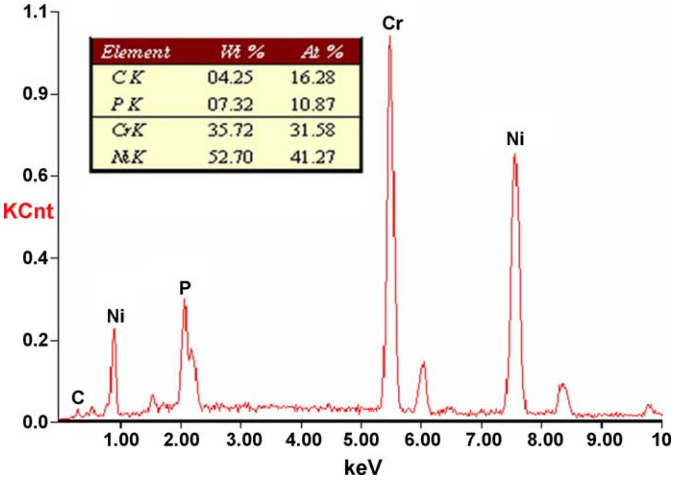


Fig. 2. The morphologies of the powders (a) raw powders, (b) high magnification image of (a), (c) as-pretreated powders, (d) high magnification image of (c).

$40\ g\ L^{-1}$ (the optimal Cr_3C_2 powder concentration in the nickel plating bath is $40\ g\ L^{-1}$). The Cr_3C_2 powder changed into the angular powder after coarsening treatment, therefore, the specific surface area of Cr_3C_2 powder obviously increased. The attached debris and impurities of the Cr_3C_2 powder were cleaned completely by coarsening treatment.

The high magnification morphologies of initial Cr_3C_2 powder and as-pretreated Cr_3C_2 powder are shown in Fig. 2(b) and (d). It is found that the surface of initial Cr_3C_2 particle is clean and smooth, while the surface of Cr_3C_2 powder after coarsening consists of nanometer spherical and lath particles. The spherical and lath particles are densely mosaic forming surface layer. The existence of nanometer spherical and lath particles obviously increases the specific surface area of Cr_3C_2 powder compared with that of original powder, which is beneficial to the adsorption and nucleation as well as the growth of nickel particles during electroless plating.

The morphologies of Ni-coated Cr_3C_2 powders are shown in Fig. 3, when Cr_3C_2 powder concentration is $40\ g\ L^{-1}$. From Fig. 3(a), it is seen that the coating shows cell structure and characteristics of dense, uniform distribution with some free formed Ni particles. SEM-EDS analysis reveals that the composition of the coating on the surface, as shown in Fig. 4, consists of Ni and P elements, the latter come from the reducing agent, sodium hypophosphite.



Element	Wt %	At %
CK	04.25	16.28
PK	07.32	10.87
CrK	35.72	31.58
NiK	52.70	41.27

Fig. 4. EDS result of Ni-coated Cr_3C_2 powder (arrow shown in Fig. 3(a)).

As shown in Fig. 3(b), with the increase of plating time, cells grow up and merge into mosaic layer. As shown in Fig. 3(c), the lath particles with reticulate structure on the as-coated surfaces act as active sites for further deposition forming upper layers. Some free particles seem to adhere on the surfaces. At last, the Cr_3C_2 powder is plated by a certain amount of Ni layers.

Ni–P grains grow on the surface of the Cr_3C_2 powder by a lateral growth mechanism suggested by Homma et al. [12]. Some researchers suggest that a rough surface promote this lateral growth mechanism of the Ni–P deposition. The NiO pretreatment and the surface roughness significantly promote the uniform deposition of Ni–P islands [13]. After coarsening treatment, the increased surface roughness and specific surface

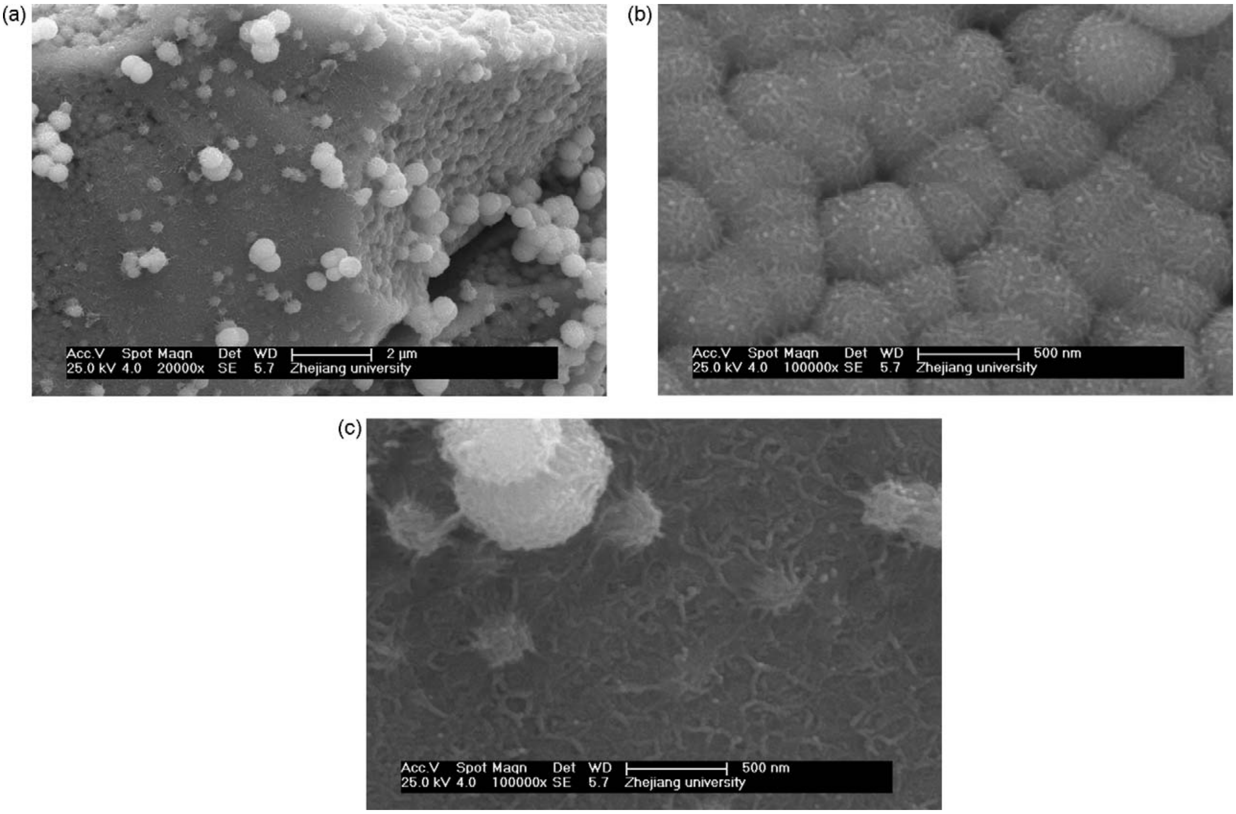


Fig. 3. (a–c) The morphologies of Ni-coated Cr_3C_2 powder.

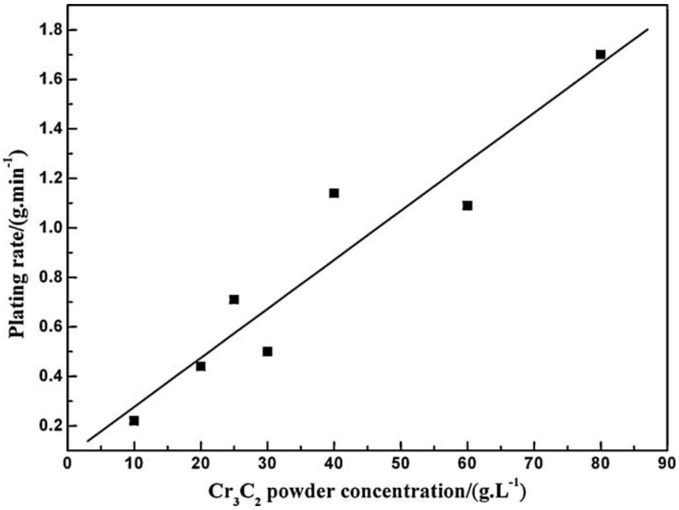


Fig. 5. Effect of Cr_3C_2 powder concentration in the plating bath on plating rate.

area of Cr_3C_2 particle, Fig. 2(d), significantly enhance the efficiency of the plating process. After the Ni–P cell structure grow up, the fresh-formed surfaces also act as catalytic sites for further deposition (Fig. 3(c)), and new Ni–P grains form and grow up on Ni-coated surfaces by the lath particles. The free-state nucleated Ni–P particles are then adsorbed on the Ni-coated surfaces by the lath particles.

3.2. Effect of Cr_3C_2 powder concentration on electroless plating rate

Powder electroless plating is a complex chemical reaction, which is affected not only by the composition of plating solution, but also temperature, pH and powder concentration [14]. The optimal content of $NiSO_4$ (25 g L^{-1}) and NaH_2PO_2 (27.6 g L^{-1}) were fixed up, and pH value and temperature of the bath were adjusted to 9.0 by NaOH and room temperature respectively, then the plating rate can be changed by controlling the Cr_3C_2 powder concentration. Fig. 5 shows the effect of Cr_3C_2 powder concentration on plating rate. With the increase of Cr_3C_2 powder concentration, the reaction time will become shorter accordingly. The reasons are that the Cr_3C_2 particles which participate in response increase in the plating solution, greatly increasing the surface area of plated particles. The nucleation rate and the rate of free-state nucleated Ni–P which adsorbed on the Ni-coated surfaces increase. Therefore, the plating rate increases, and the reaction time shortens. However, when the Cr_3C_2 powder concentration was higher than 40 wt.%, the Ni-coated Cr_3C_2 powder becomes incomplete and uneven, and some Cr_3C_2 powders could not be coated. Therefore, the optimal Cr_3C_2 powder concentration in the nickel plating bath is 40 g L^{-1}.

4. Conclusion

(1) Ni-coated Cr_3C_2 powder was prepared by ultrasonic-assisted electroless plating process without conventional sensitization and activation treatments at room temperature. The Ni layers on the Cr_3C_2 powder had cell structure with dense, uniform distribution.

(2) The generation of nanometer spherical and lath particles after coarsening obviously increased the specific surface area of Cr_3C_2 powder, which is beneficial to the nucleation and growth as well as the adsorption of nickel grains, during nickel plating.

(3) With the increase of Cr_3C_2 powder concentration, the reaction time will become shorter accordingly. The optimal Cr_3C_2 powder concentration in the nickel plating bath is 40 g L^{-1}.

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