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PREPARATION AND CHARACTERISTICS OF NEW TYPE NANOCOMPOSITE POWDER

LING Guo-ping(凌国平), LI Jian(郦　剑), WANG Shang-jun(王尚军)
(*Department of Materials Engineering, Zhejiang University, Hangzhou 310027, China*)
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Abstract: This new type nanocomposite powder was prepared with electroless plating enhanced by ultrasonic wave. High-resolution transmission electron microscopy (HRTEM) showed that As-plated particles have nanometer scale average size. X-ray diffraction (XRD) analysis showed the presence of a noncrystalline structure in the plating layer on the nano-Al_2O_3 matrix. The content of the plating layer was analyzed by energy dispersive X-ray spectroscopy (EDX) technology. The plating layer content was adjustable by changing the solution concentration.

Key words: nanocomposite powder, Al_2O_3 nanoparticles, electroless plating enhanced with ultrasonic wave
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INTRODUCTION

The author's past researches showed that electroless-plated metal-ceramic composite powder could retard ceramic gain growth during the sinter process, evenly disperse the metallic phase on the ceramic matrix and, therefore, increase the fracture strength, toughness and wear resistance of the sintered bulk sample (Li et al., 1997; Mao et al., 1997; Mao et al., 1998). It was found that when the particles volume decreases to nanometer scale the above properties appeared. These findings could be used for preparing new generations of advanced ceramics with superior performance in different aspects of application. Researchers are now interested in how to keep the grain size in nanoscale during sintering process, which can possibly lead to increased density, strength, and toughness. Grain growth is much easier for ceramic powder in nanoscale (Li et al., 1995; Chen et al., 1996; Wu et al., 1996). Researchers had developed some technologies for preparing nanocomposite powder, for example, in ceramic-ceramic, polymer-metal system (Ni et al., 2000). But research for nanoceramic-metal composite powder has not been reported.

Based on former work on composite powder in microscale, this work employed electroless plating process enhanced with ultrasonic wave technology to prepare the nanocomposite Ni-Al_2O_3 powder. The powders were characterized by HRTEM, XRD, EDX, etc..

EXAMINATION PROCEDURE

The average size of the raw Al_2O_3 particles provided by the Weiwei nanomaterials Co. was 10 - 20 nm. The ingredients of the electroless plating solution, were of analysis-grade purity. The raw Al_2O_3 nanoparticles were pretreated to produce catalytic activity in the electroless plating process. The pre-treatments consisted of sensitization and activation in different solutions. Plating process parameters are shown in Table 1. Plating procedures were as follows: sensitization → rinse → activation → rinse → drying → electroless nickel plating → rinse → drying at 80℃ in air. After preliminary treatment, the powder was put into a plating beaker immersed in the water bath of the type CCW - 50W ultrasonic generator shown in Fig.1. Deposition occurred about 20 - 30 minutes later with H_2 bubbles evaporating out of the solution. During the process, the solution remained transparent, did not show signs of self-dissolution and/or precipitation. The particle morphology was observed with HRTEM before and after plating; the phase composition was analyzed with XRD technology;

and the sintered sample was measured with EDX technology.

Table 1 Bath composition and plating conditions

Compound	Condition
$NiSO_4 \cdot 6H_2O$	30 g/L
$NaH_2PO_2 \cdot H_2O$	3 – 25 g/L
Complexing agent	32 g/L
Stabilizer	$0 - 10^{-3}$ g/L
pH	4.0 – 5.5
Temperature	25 – 35 ℃

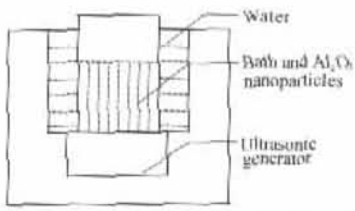

Fig.1 Schema of ultrasonic electroless nickel plating

RESULTS AND DISCUSSION

Nanocomposite powder properties

The As-plated powder was black and electrically conductive. Under room temperature the microscale or larger particle size powder that appeared there was no reduction reaction and thus, deposition did not occur. Deposition on the nano-Al_2O_3 particle surface was stimulated and enhanced by ultrasonic energy.

Generally, during electroless nickel plating, self-catalysis induces reduction reaction on the sample surface chemically described as follows:

$$Ni^{2+} + H_2PO_2^- + H_2O = Ni + H_2PO_3^- + 2H^+$$

The reaction process needs to absorb energy from the surroundings. Commonly heating to 65 – 95℃ facilitates deposition on the microscale or larger powder.

The nanoparticle has high surface energy and, after stimulation by ultrasonic wave, the deposition on its surface normally occurs under room temperature (25 – 35 ℃). It was reported (Zhang et al., 1993; Li et al., 1998) that ultrasonic waves could retard static electricity force and Vander Waals force, which cause nanoparticles to aggregate and form a number of grain clusters, and disperse the clustered nanoparticles. In this way ultrasonic waves can accelerate the deposition process at reduced plating temperature.

The morphology of the Al_2O_3 powder

The original Al_2O_3 particles with average size of 10 – 20 nm are shown in Fig. 2a. Fig. 2b shows that the pretreated particles differed little from the original particles in size or shape, but are more dispersed. As-plated powder in Fig.3 has average size of 50 – 60 nm. The originally granular form of the particles has changed into rugby-like shape. The average coating thickness of As-plated Al_2O_3 particles is evaluated as 20 – 25 nm.

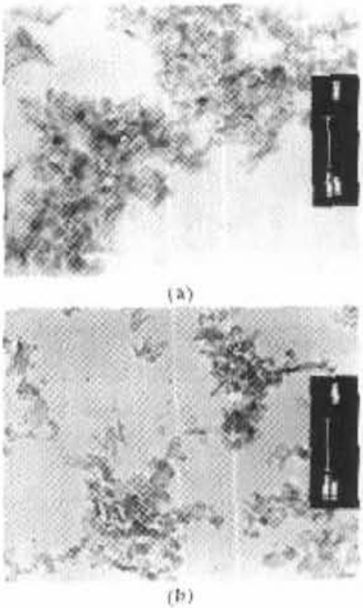

Fig.2 TEM images of nano-Al_2O_3 powder
(a) original nano-Al_2O_3 powder;
(b) powder after pretreatment

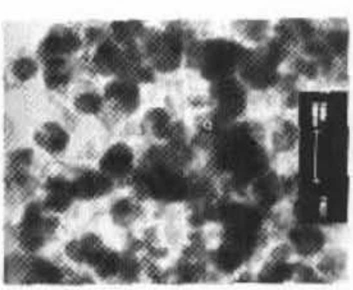

Fig.3 TEM image of Ni-Al_2O_3 nanocomposite powder

XRD examination result

Fig.4 shows the X-ray diffraction spectrum of plated composite powder with some sharp diffraction peaks of α-Al_2O_3 and a broad peak of about 45° diffraction angle. The changes of $NaH_2PO_2 \cdot H_2O$ content had little influence on the diffraction patterns. After heat treatment (400 ℃, 1 hr in air) the Ni_2P sharp diffraction peak appeared as shown in Fig.5. It could be deduced that the deposited layer on the α-Al_2O_3 particles was noncrystalline as commonly is the case with electroless Ni plating layer. The layer was as thin as 20 – 25 nm so X-ray could pass it and the α-Al_2O_3 diffraction peaks appeared.

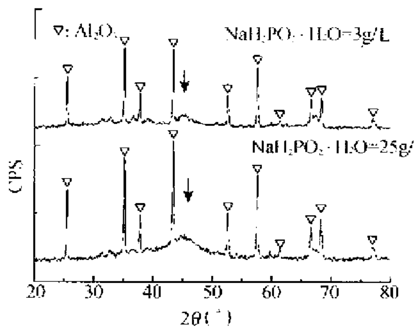

Fig.4 X-ray diffraction patterns of Ni-Al_2O_3 nanocomposite powder As-plated

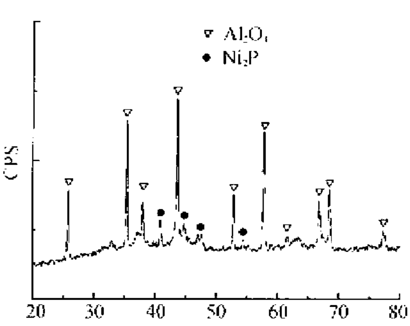

Fig.5 X-ray diffraction pattern of Ni-Al_2O_3 nanocomposite powder after heat treatment

EDX results of plating layer

The plated powder was compressed into a bulk sample, and EDX technology used to determine the content in the plating layer showed that the average content of nickel was about 85.6% wt and that of phosphorus was about 14.4 % wt of the plating with 25 g/L $NaH_2PO_2 \cdot H_2O$ added into the solution. The phosphorous content was adjustable. For example, when $NaH_2PO_2 \cdot H_2O$ added into the solution was 3 g/L, 10 g/L, the phosphorous content in the As-plated layer was 4.3% wt, 8.8% wt correspondingly.

CONCLUSIONS

1. The Al_2O_3 nanoparticles 10 – 20 nm size were successfully plated with an electroless nickel layer. Ultrasonic wave at 34 ± 1.5 kHz frequency was used to facilitate the plating process.

2. The deposition layer on the Al_2O_3 nanoparticle surface was a noncrystallic Ni-P alloy with nanoparticle size of 20 – 25 nm.

3. The chemical content of the plating layer was adjustable by changing the solution concentration.

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