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PREPARATION OF Al_2O_3/CU NANOCOMPOSITE POWDER BY ELECTROLESS PLATING AND SUBSEQUENTLY SINTERING

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ABSTRACT

In this study, the Al_2O_3–Cu core-shell nanocomposite, thin metallic films coated ceramic particles, was fabricated by Electroless plating of Cu on Al_2O_3 particles and subsequently conventional sintering. This kind of nanocomposites can be used as the catalysts, electromagnetic wave absorbing materials, electrodes in fuel cells and etc. because of its excellent properties such as good electrical and thermal conductivities, excellent resistance to high temperature annealing, excellent corrosion and wear resistance. At first, a thin metallic film is deposited onto ceramic particles, using the Electroless deposition, in order to elaborate a composite powder. and than the coated particles were cold pressed at 100- 500 MPa and sintering was conducted at different temperature of 900 - 1100 °C with different atmosphere (air and Argon gas). The influence of various parameter such as effects of pretreatments, PH, temperature, time and chemical composition in the plating bath, Al_2O_3 powder concentration and Al_2O_3 powder size (micron and nanosized) on the uniformity, thickness and composition of coated layer were investigated. The results show that it is possible to fabricate Cu/alumina core-shell powder materials by electroless plating technique. Also it was found that the sizes of Cu on Al_2O_3 particles are between several and several hundreds of nanometers. while the amount of coating on the powders as increases as temperature and PH increase.

KEYWORDS: *Coated Al_2O_3, Core shell structure, Composite powder, Electroless plating*

INTRODUCTION

Core shell structural composites, thin metallic films coated ceramic particles, are an attractive kind of ceramic matrix composite due to their combined characteristics of metals and ceramic matrix with improved functional and mechanical properties [1]. This kind of composites are fabricated by solid state methods such as ball milling, liquid state methods such as sol gel, Electroless deposition (ED) and precipitation deposition, and vapor deposition such as PVD and CVD. Among these methods, the electroless coating has been recognized as one of the most effective techniques for preparing metal-coated ceramics powders due to the its advantages such as even dispersion of the metal coating, easy control of the coating thickness and nondependence on the electrical properties of the substrate [2-4].

In this study, the Al_2O_3–Cu core-shell nanocomposite was fabricated by electroless plating of Cu on Al_2O_3 particles and subsequently conventional sintering. This kind of nanocomposites can be used as the catalysts, electromagnetic wave absorbing materials, electrodes in fuel cells, thermal protective coatings of air engines, contact materials in relays, contactors, switches, circuit breaks, electronic packaging, high current brush and etc, because of its excellent properties such as good electrical and thermal conductivities, excellent resistance to high temperature annealing, excellent corrosion and wear resistance, high bonding strength, excellent weldability, and controllable magnetic properties through suitable heat treatment [5-7].

EXPERIMENTAL

Copper was coated on the surface of Al_2O_3 particles through an electroless coating process. In the Electroless deposition, a thin metallic film is deposited onto ceramic particles in order to elaborate a 'composite powder'. Because of chemical inertness of Al_2O_3 particles, at first they were pretreated to obtain catalytic activity. The pretreatment had been performed in these stages:

(i) Washing: In order to improve the adherence of the coatings and remove impurities on the surface. The Al_2O_3 particles were first washed in acetone solution by magnetic stirrer and dried in an air heater at 110 °C for 15 min.

(ii) Coarsening: to obtain a rough surface and increase the specific surface area and hydrophilic property of the particles. the powders were etched by a hydrophilic solution of 100 ml/l HF and 100 ml/l HCl by magnetic stirrer for 5 min and at room temperature. Afterwards, the powder was cleaned with de ionized water for several times.

(iii) Sensibilisation: for chemisorption of Sn on the surface of Al_2O_3 particles. the coarsed powders were submerged in the sensitization hydrochloric acid solution of 10 g/l $SnCl_2$ and 50 ml/l HCl by magnetic stirrer for 30 min and at room temperature. Then the powders were washed in distilled water.

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(iv) Activation: for chemisorption of Pd onto Sn clusters, the powders were submerged in the hydrochloric acid solution of 0.25 g/l $PdCl_2$ and 10 ml/l HCl by magnetic stirrer for 30 min and at room temperature. Then the powders were washed in distilled water.

(v) Drying: The powders were washed with the de-ionized water for several times and then were filtrated and dried in an oven at 80 ºC.

Electroless deposition was performed in a described bath in table1, while magnetic stirrer was used to stir the bath and avoid particle agglomeration.

Table1: composition and properties of Electroless deposition bath

Composition	Concent	PH	Temp (ºc)	Time (min)
Copper sulphate Sodium hydroxide Pottasium sodium tartrate Formaldehyde Pretreated Al_2O_3	5-20g/l 10g/l 50g/l 10g/l 1-10g/l	8-10	50-90	10-120

In electroless deposition it is difficult to obtain uniform metallic coating on the ceramic powders because the process was affected by lots of parameters. However, The influence of various parameter such as effects of pretreatments, PH, temperature, Time and chemical composition in the plating bath, Al_2O_3 powder concentration and Al_2O_3 powder size (micron and nanosized) on the uniformity, thickness and composition of coated layer were investigated.

After that the coated particles were cold pressed at 100- 500 MPa and sintering was conducted at different temperature of 900 - 1100 ºC for 30- 60 min in a tube furnace with different heating rate and different atmosphere (air, vacuum and Argon gas).

The morphology and structure of the starting material and as coated Composite powders (Cu coated alumina micropowders and nanopowders) were characterized by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD).

RESULTS AND DISCUSSIONS

The optimized condition for copper coating of Al2O3 particles by electroless is reported:

- 5 step pretreatment, using 10 g/l $SnCl_2$ and 0.25 g/l $PbCl_2$ as the activating materials.

- Chemical composition of bath: 10 g/l Copper sulphate, 50 g/l Pottasium sodium tartrate, 10 g/l Sodium hydroxide and 10 g/l Formaldehyde.

Most uniform coating was applied at the temperature of bath 60 ^{o}C, PH=10 and 30 min stirring the suspending solution containing 5 g/l of micron sized Al_2O_3 Or 2 g/l of nano sized Al_2O_3.

XRD analysis shows very sharp peaks of Cu, indicated that the as-deposited copper film was crystalline, also peaks of P and Ni_3P appeared in some cases.

The morphology and average thickness of the coatings was measured by SEM. In the optimized condition the thickness of Cu layers were obtained 80 ± 20 nm on the micron sized Al_2O_3 and 25 ± 5 nm on the nano sized Al_2O_3.

The composite powders were sintered successfully at temperature 900^{o}C in vacuum atmosphere. While sintering at air caused to formation of copper oxides.

CONCLUSION

The results show that it is possible to fabricate Cu/alumina core-shell powder materials by electroless plating technique. Also it was found that the sizes of Cu on Al_2O_3 particles are between several and several hundreds of nanometers, while the amount of coating on the powders as increases as temperature and PH increase.

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