Fabrication of Alumina Free-standing Objects by Electrophoretic Deposition

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Abstract

The coating of conductive polypyrrole (Ppy) on nonconductive ceramic substrates was performed by polymerization of pyrrole (Py) in an aqueous solution. The Ppy film was characterized by scanning electron microscopy and conductivity measurements. Electrophoretic deposition of bimodal alumina suspension prepared with a phosphate ester was performed using Ppy film as a cathode. Fabrication of alumina ceramics with irregular shapes or complicated patterns were also attempted by sintering the deposits together with the Ppy coated substrates in air.

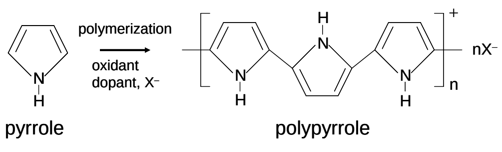
Keywords : electrophoretic deposition, electrode, polypyrrole, alumina, free-standing film

1. Introduction

Electrophoretic deposition (EPD) is a colloidal process wherein ceramic bodies are directly shaped from a stable colloidal suspension by a dc electric field.[1-3] Metals or graphite have been commonly used as substrates since electrically conductive materials must be used as the electrodes. Graphite electrodes, which can be burned out during sintering with deposits in air, are often used to fabricate free-standing ceramic objects with irregular shapes or complicated patterns. However, the adhesion of deposits to the graphite surface is relatively weak because of its chemically inert property; it sometimes leads to the thick deposits peeling off or cracking during drying. In this study, polypyrrole (Ppy), which is one of conductive polymers, films were coated on non-conductive ceramic materials so that they can be used as the substrates for EPD. Fabrication of alumina free standing objects was attempted using the Ppy films as cathodes of the EPD processing.

2. Experimental and Results

The coating of conductive polypyrrole (Ppy) on nonconductive ceramic substrates was performed by polymerization of pyrrole (Py) in an aqueous solution according to the following reaction;



(1)

The polymerization was allowed to proceed for 15-18h at 0°C. Ammonium peroxodisulfate $(NH_4)_2S_2O_8$ and 2,6-naphthalenedisulfonic acid disodium salt $C_{10}H_6(SO_3Na)_2$ were used as an oxidant and a doping agent, respectively. The SEM photograph of the Ppy film prepared on a glass slide is shown in Fig. 1. The Ppy film had slightly rough surface, fairly uniform thickness and dense microstructure. The average thickness of the Ppy films directly formed on ceramic substrates was c.a. 0.5 μm. The average electric conductivity of the Ppy films was 5.88 S/cm. The Ppy film had enough high conductivity as an electrode for the EPD processing.

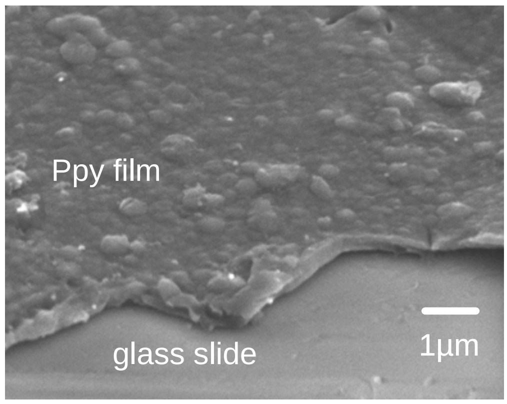


Fig. 1. Micrograph of a typical Ppy film prepared on a glass slide.

The EPD was carried out using a bimodal alumina suspension to enhance the green density. A mixture of alumina powders with average particle sizes of 0.6 μm and 30 nm was dispersed in ethanol, and a 7 vol% suspension was prepared. Butoxyethyl acid phosphate (BAP), and poly vinyl butyral (PVB) were used as dispersant and binder, respectively. The EPD was carried out at a constant voltage of 50 V. The alumina deposits were sintered together with the substrate materials in air.

Fig. 2 shows the relative green densities of the compacts as a function of the nanopowder content, determined by our preliminary experiments. The mixture of the submicron- and nano- powder in the ratio of 9:1 showed the highest relative green density of ~68 %. Adhesion of the deposits to the Ppy surfaces was fairly good. Thick, crack-free deposits were formed on the Ppy-coated ceramic substrates by EPD from of the bimodal suspension. Fig. 3 shows the SEM microstructure of the outermost surface of an alumina green deposit. The void spaces between the larger particles are filled wit small nano particles. This bimodal microstructure probably contributes to the high green density and effectively suppresses large shrinkage and cracking of the compacts during drying. The EPD at higher voltages of 100-300 V could speed up the deposition but caused the generation of ‘warts’ and vertical stripes on the deposit surface.

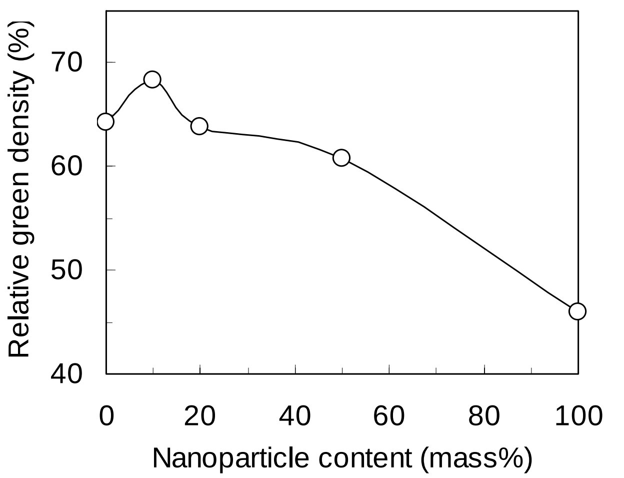


Fig. 2. The relative green densities of the bimodala lumina compacts.

Alumina ceramics with complex shapes and patterns were obtained by sintering the deposits together with the Ppy-coated substrates in air. These examples are shown in Fig. 4. The sintered alumina ceramics could be detached from the substrates without cracking by optimizing the sintering conditions. This technique will be utilized for fabricating various shape-controlled ceramics by EPD.

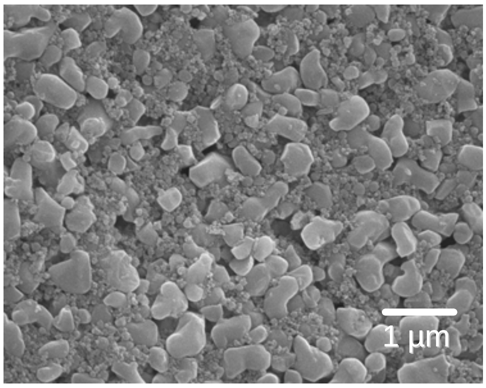


Fig. 3. SEM microstructure of the outermost surface of an alumina green deposit.

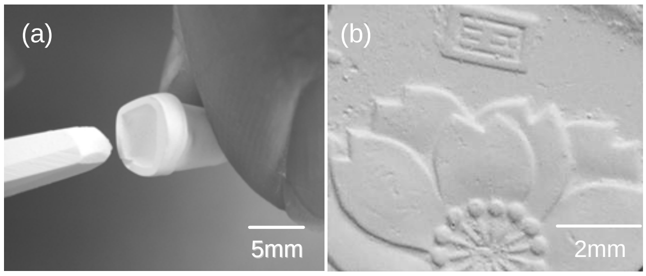


Fig. 4. Alumina free-standing objects with irregular shapes or complex patterns: (a) cap, (b) free-standing film with the transcribed pattern of a ¥100 coin.

3. Summary

Thick, crack-free alumina deposits were formed on the Ppy-coated ceramic substrates by electrophoretic deposition. Adhesion of the deposits to the Ppy surfaces was fairly good. Alumina ceramics with irregular shapes or complex patterns were obtained by sintering the deposits together with the substrates in air.

4. References

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