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Electrophoretic Deposition of Alumina on Stainless Steel Wire Net

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Electrophoretic deposition (EPD) of alumina was investigated in aqueous solvent using stainless steel wire net as an electrode. The starting materials were γ-alumina, α-alumina and mixtures of γ-alumina and α-alumina. The experimental conditions of EPD were : applied voltage 5-30 V, EPD time 60-300 s, slurry concentration 10 mass%, electrode distance 10 mm. The weight of deposits increased with increasing applied voltage and EPD time and also changed with the ratio of γ-alumina and α-alumina. The deposition weight started to decrease >80% α-alumina and decreased sharply in 100% α-alumina. The deposits covered the wire net completely and formed a sheet-like body. These deposits showed cracks during drying, especially with increasing content of γ-alumina. The microstructures of these deposits changed little even after firing at 1000°C to form a porous body.

Key words: Electrophoretic deposition, stainless steel wire net, alumina, porous material

1. INTRODUCTION

Electrophoretic deposition (EPD) is an effective technique for forming ceramics, and has been extensively reported [1]. Deposits can be formed easily and speedy on various metals and also on carbon, i.e. electrodes. In aqueous solvents, however, gases are evolved from the electrodes due to electrolysis, degrading the microstructure of the deposits by the presence of many large pores formed by the evolved gases. Clark et al. [2] examined EPD of alumina sol in aqueous solvent using an Al plate as a cathode. They found H_2 evolution at the cathode by applying >2.5 V and the resultant deposit was peeled off from the substrate by EPD at >15 V. To avoid these phenomena, nonaqueous solvents such as alcohols are used. In EPD using nonaqueous solvents, it is necessary to enhance the low electric conductivity of the solvent by addition of reagents such as I_2 which is known to enhance the electric conductivity. Miyazaki et al. [3] succeeded in preparing α-alumina deposits on a Pt wire by increasing the electric conductivity of acetylacetone with a small amount of I_2. Similarly, α-alumina and δ-alumina were deposited by EPD on carbon fibers using iso-propanol [4] and on carbon and stainless steel fibers and tow using ethanol [5].

Use of a nonaqueous solvent for EPD also has disadvantages including increased production costs and environmental effects. It is therefore preferable to perform EPD using aqueous solvents. Ferrari et al. [6] prepared alumina/zirconia layers on carbon fibers in aqueous solvent by EPD. They prepared the deposit under a constant current 2.8 mA/cm^2 and EPD time <10 min. Although they did not discuss the effect of gas evolution, no influence was detected judging from the resultant dense microstructure of the deposits. This result may arise from the use of a carbon electrode, low EPD current and/or short EPD time. By contrast, Tang et al. [7] used an interesting technique to avoid the influence of evolved H_2 gas during EPD in aqueous solvent using a Pd plate as the cathode. As is well known, Pd can adsorb H_2 in its structure. Tang et al succeeded in forming dense γ-alumina deposits using this cathode. They also found that the deposited weight of γ-alumina was strongly dependent on the pH of the solvent and showed a maximum at a pH of about 8 (isovalent electric point=9.5), not at a high ξ-potential. From these papers, it is considered that influence of H_2 evolution in EPD using aqueous solvent may possibly be avoided under appropriate preparation conditions.

In this study, we investigated EPD deposition of alumina in aqueous solvent by using stainless steel wire net as an electrode and examined the microstructure of the resulting deposits after drying and firing.

2. EXPERIMENTAL

The starting materials were γ-alumina (AKP-G015, Sumitomo Chemicals) and α-alumina (AKP-30, 50, Sumitomo Chemicals). Mixtures of these powders were prepared with mass ratios of γ-alumina/α-alumina 50/50, 20/80, 10/90, 5/95. These powders were dispersed in de-ionized water at a concentration of 10 mass% using an ultrasonic bath (2510J-DTH, Bransonic) for 10 min. The samples containing γ-alumina were ground in an attrition mill (MA01SC-X, Mitsui Miike Kakoki) for 3 h before use to improve their dispersability by decreasing their aggregated particle size.

EPD experiments were performed under a constant voltage condition (GP0250-1, Takasago), measuring the current during EPD using a digital multi meter (6452A, Advantest). Ti metal plates and stainless steel wire nets (P-150, Nippon Filcon) were used as the anode and cathode, respectively. The electrodes were 50x35 mm and their separation was 10 mm. The stainless steel (SUS316) wire was 65 μm in diameter with an aperture of 150 mesh, corresponding to an area of about 100x100 μm^2. Before the EPD, the Ti plate was washed with acetone and the stainless wire net was treated with 1M NaOH and washed with de-ionized water and acetone. EPD times of 1 to 5 min and applied voltages of 5-30 V were used.

The EPD samples were dried at room temperature (RT). The deposited weight was evaluated from the difference in weight before and after the EPD. The dried deposits were fired at 1000°C for 5 h under flowing N_2 (2 l/min) at heating rates of 5°C/min (RT-800°C) and 1°C/min (800-1000°C) and a cooling rate of 5°C/min.

The particle sizes of the samples were measured using a Microtrack instrument (7995-30SPA, Nikkiso). N_2 adsorption-desorption isotherms were measured using an Autosorb-1 (Quanta Chrome) at 77K. The samples were pre-heated at 180°C for 3 h in vacuo before the measurements. The specific surface area (S_{BET}) was calculated by the BET method using the data recorded in the partial pressure range 0.05-0.30. The microstructure of the deposits was observed using SEM (JSM-5310, JEOL).

3. RESULTS AND DISCUSSION

From the supplier's specifications, all the particle sizes of the starting materials are <1 μm. The average measured particle sizes (d_{50}: 50% particle size) were 2.7 μm in the γ-alumina, 0.8 μm in the AKP30 (α-alumina) and 1.2 μm in the AKP50 (α-alumina). Since these values are much larger than the supplier's values, the starting materials are thought to be agglomerated, especially the γ-alumina, because the primary particle size should be several tens of nm to be consistent with the S_{BET} value of 170 m^2/g. Therefore, the γ-alumina powder was ground by attrition mill. The d_{50} value gradually decreased with increasing grinding time and became 0.9 μm after grinding for 3 h. The samples containing γ-alumina were therefore ground for 3 h. The resulting d_{50} values ranged from 0.9 to 1.3 μm except the 80% α-alumina sample (1.9 μm). The reason for the larger d_{50} of this sample is uncertain at present. The pH values of the slurries of suspended sample powders showed a gradual decrease from 7.7 to 7.0 with increasing α-alumina content. By contrast, the pH of the 100% α-alumina sample was 5.8, apparently lower than for the samples containing γ-alumina. This may be related to the grinding treatment.

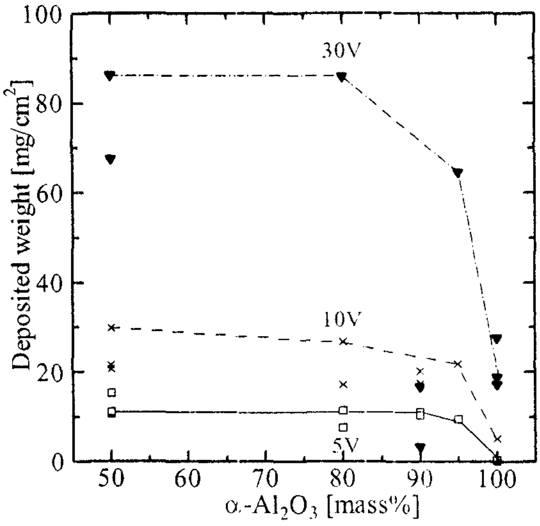

Fig. 1. Deposited weight as a function of α-alumina ratio.

The deposits obtained by EPD differ greatly according to the ratio of α-alumina. The deposit from the 0% α-alumina sample was very thick but broke into pieces during drying. By contrast, the deposit from the 100% α-alumina sample is very thin and light in weight. Therefore, the EPD experiment was performed under same conditions for all the samples. Fig. 1 shows the deposited weight as a function of the ratio of α-alumina. The deposit weights are high in samples containing from 50 to 80% α-alumina but decrease at higher α-alumina contents. The weight is much less in the 100% α-alumina sample. The deposits of 90% α-alumina sample deviate

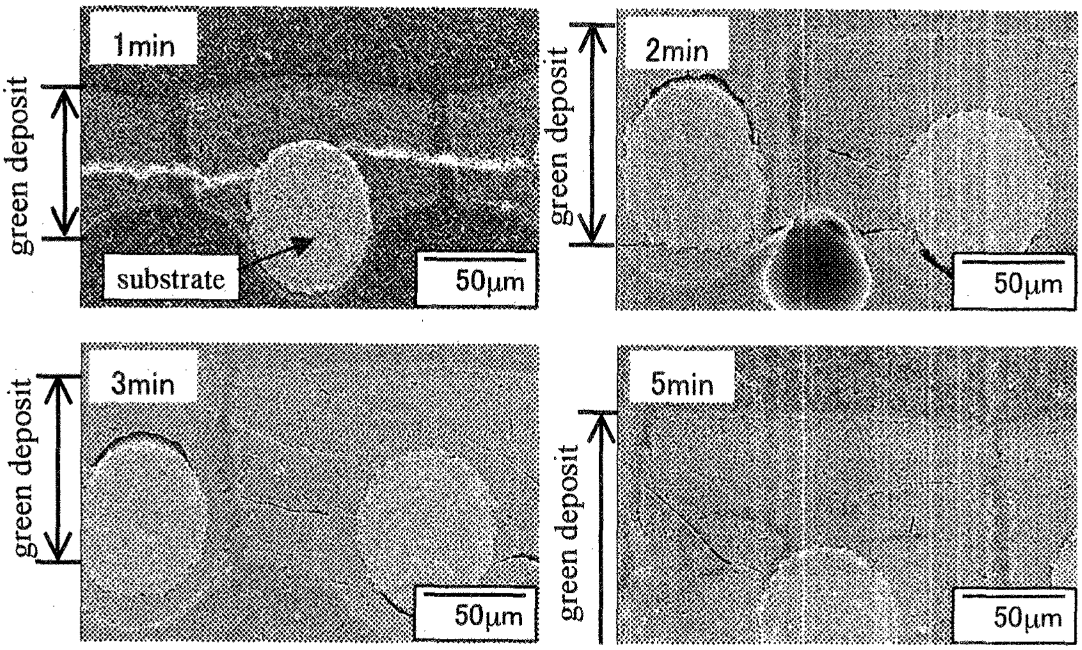

Fig. 2. SEM photographs of cross sections of green deposits (80% α-alumina) EPDed at 5 V for various times.

from the electrode, possibly as a result of the EPD current of this sample being higher than in the other samples. The deposit weights increase with higher applied voltage, showing an almost linear relation up to 30 V.

Fig. 2 shows the cross sections of the green deposits prepared at 5 V for 120-300 s. The top surfaces shown in the photographs correspond to the deposit plane. Deposition is found to occur not only on the deposit plane but also on the opposite plane, forming sheet-like deposits covering the cathode net. The thickness of the deposits increases with increasing EPD time, as shown in Fig. 3. A steep increase in thickness is observed at the beginning of EPD, gradually becoming slower. This is due to the decrease in EPD current by deposition of non-conductive material on the cathode as reported by Sarbar and Nicholson [1] who used a constant voltage condition. All the present deposits show similarly densely packed microstructures (Fig. 2) and no macropores resulting from evolution of H_2 gas are observed even at EPD of 30 V. Differences in the cathode may be responsible for the difference between these results and those of Clark et al.[2]. The green densities of the deposits determined from the thickness and weight are about 50%, similar to those reported by Tang et al.[7].

As shown in Fig. 2, cracks are formed in the green deposits, becoming more apparent with increasing α-alumina content of the starting material which becomes heavier with decreasing α-alumina ratio. Since these cracks are observed mainly in the centers of the openings of net, they evolve by shrinkage of the deposit during drying.

The microstructures of the fired samples are shown in Fig. 4. Little change was observed in the microstructure after firing and the deposits maintained good contact with stainless steel wire. It is therefore important to suppress cracking during drying of the EPD deposit by using

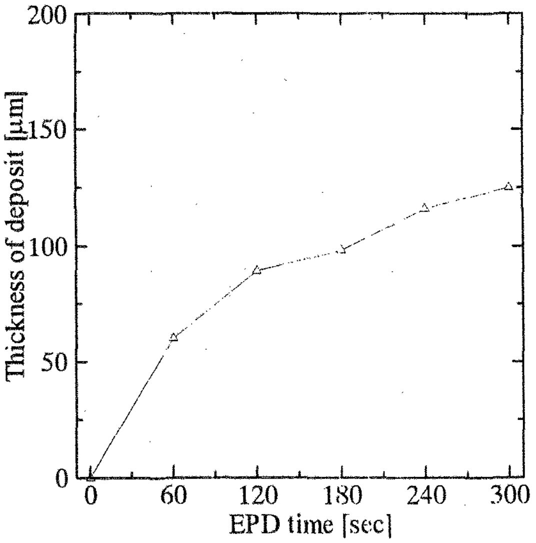

Fig.3. Change of thickness as a function of EPD time.

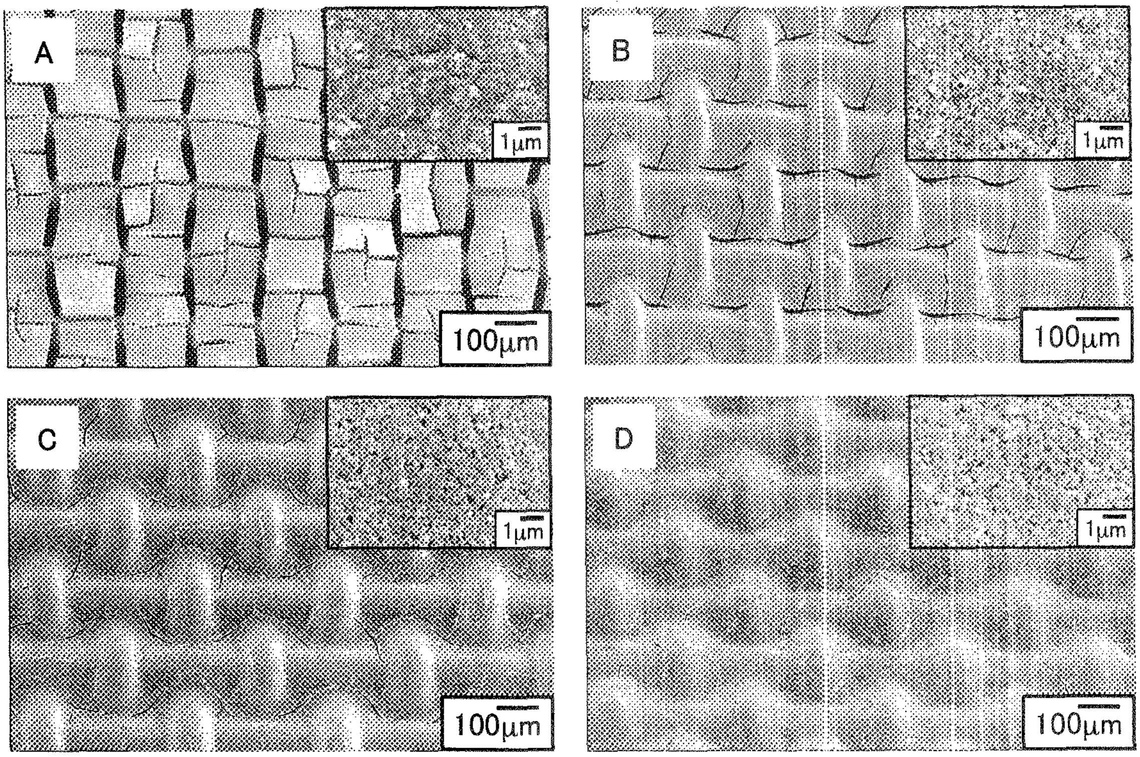

Fig. 4. Microstructures of deposits fired at 1000°C for 5 h. (A): 50% α-alumina, (B): 80% α-alumina, (C): 95% α-alumina and (D): 100% α-alumina.

controlled drying conditions.

4. CONCLUSION

Alumina was electrophoretically deposited on a cathode of stainless steel wire net using an aqueous solvent. The deposit weight was found to increase with increased applied voltage and EPD time but the rate gradually decreased at longer EPD times. The deposit weight also changed with the proportion of α-alumina in the starting materials, decreasing with increasing α-alumina ratio. Although the deposits contained cracks due to shrinkage during drying, they retained their microstructure after firing at 1000°C to form a porous body. Careful drying to suppress cracking in the green body appears to be important to obtain a crackfree fired body.

5. ACKNOWLEDGEMENT

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