

Alumina Coating on Stainless Steel Wire Net by Electrophoretic Deposition and its Oxidation Resistance

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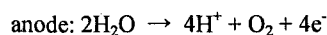
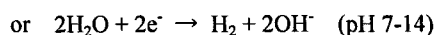
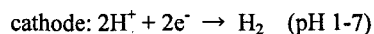
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Alumina coating on stainless steel wire net was examined by electrophoretic deposition (EPD). The starting powder used was α -alumina (AKP-50, Sumitomo Chemicals; average particle size $0.3\mu\text{m}$). The experimental conditions of EPD were as follows: applied voltage 5V, EPD time 15-300s, electrode distance 10 mm, suspension concentration 10 mass%. Nonionic surfactant (Pluronic F-127, BASF Co. America.) was used as a dispersant stabilized the suspension. The pH of the suspension was changed from 6.8 to 3 to increase dispersibility of alumina particle and also to investigate its effect on EPD. At pH 6.8, gas evolution from an electrode was not observed and the obtained deposits were homogenous. The deposits showed no cracks when dried under relative humidity of 60%. Little change was observed in the microstructure of the deposit even after firing at 1000°C 5h in air. This α -alumina coating by EPD was found to be effective to suppress oxidation of the stainless wire net.

Key words: electrophoretic deposition, alumina, oxidation resistance

1. INTRODUCTION

Electrophoretic deposition (EPD) is an effective technique for forming ceramics films, by assisting the movement of charged particles in liquid suspension. When a voltage is applied to the suspension, the charged particles move toward the oppositely charged electrode, depositing a ceramic film on the electrode. Therefore, this technique directly prepares membranes from the suspension, and has some advantages; 1) the equipment is simple and the cost is low, 2) the process is rapid, 3) the film thickness can be controlled by changing the applied voltage and deposition time, and 4) there is little restriction on the shape of products [1], it is, therefore, widely used to prepare ceramic films and coatings such as alumina [1-8], PZT [9] and apatite [10]. Arato *et al.* [2,3] reported the formation of an alumina coating on tungsten double helical heater whose structure was complicated by EPD using a H_2O -ethanol suspension. Many researches have been carried out using organic (non-aqueous) solvents such as alcohols in the EPD process to avoid gas evolution from the electrode by electrolysis of H_2O in aqueous solvent [4] as follows;



In non-aqueous solvents, it is necessary to add an electrolyte such as $\text{Al}(\text{NO}_3)_3$, $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ [2,3] or iodine [5] to enhance the low electric conductivity of the solvent. However, use of a non-aqueous solvent for EPD also has the disadvantages of high cost and environmental effects. It is, therefore, appropriate to use an aqueous solvent for EPD rather than a non-aqueous solvent. A few studies have been reported using aqueous solvents for EPD [1,4,6,7]. In particular, Tang *et al.* [6] used an interesting technique to avoid the obstruction of electrodeposition by H_2 gas evolution. They succeeded in forming dense γ -alumina deposits using palladium, known as a hydrogen adsorption material as the cathode.

In this study, we prepared alumina films on complicated forms such as stainless wire net by EPD using an aqueous suspension. As post of the study of the alumina coating, we examined the oxidation resistance of the coated stainless surface.

2. EXPERIMENTAL

The starting material was α -alumina (AKP-50, Sumitomo Chemicals, Japan). This powder was dispersed in distilled water at a concentration of 10 mass% using an ultrasonic bath (2510J-DTH, Branson, USA) for 10 min. In order to stabilize the alumina suspension, a dispersant

was added and the pH was adjusted. The dispersant was a nonionic-type surfactant (Pluronic F-127, BASF Co., USA). M. Sakar *et al.* [11] reported that this surfactant combined with ultrasonic treatment is capable of producing well stabilized α -alumina suspensions. The dispersant concentration of Pluronic F-127 was 1.0 mass%. The pH of the suspension was adjusted to various values between 6.8 and 3 using 10% HCl (no HCl was required at pH 6.8). The resulting suspension was used for EPD. The EPD experiments were performed at a constant voltage condition (GP0250-1, Takasago, Japan), the current during EPD being measured using a digital multi meter (6452A, Advantest, Japan). Titanium metal plates and stainless steel wire nets (P-60, Nippon Filcon, Japan) were used as the anode and cathode, respectively. The electrodes were 50×35 mm in size with a distance between anode and cathode of 10 mm. The stainless steel wire was 213 μm in diameter with an opening 60 meshes. Before starting the EPD, the titanium plate was washed with acetone and the stainless wire net was treated with 1M NaOH and washed with distilled water and acetone. The EPD times were from 15 to 300s at an applied voltage of 5V.

The EPD samples were dried at room temperature (RT) under ambient conditions or a relative humidity (RH) of 60%. The deposited weight was determined from the difference in weight before and after the EPD process. The dried deposits were fired at 1000°C for 5 h in air at heating rates of 10°C/min (RT-400°C-1000°C) and a cooling rate of 10°C/min. The heating cycle contained a pause at 400°C for 4h to burn out the surfactant. The crystalline phases of the samples were measured by X-ray diffractometry (LabX XRD-6100, Shimadzu, Japan). The microstructures of the samples before and after heating were observed using SEM (JSM-5310, JEOL, Japan).

3. RESULTS AND DISCUSSION

The deposits obtained by EPD depended strongly on the pH of the suspension. Fig.1 shows the relationship between the deposition weight and the pH of suspension for an EPD time of 300s. At pH 6.8, the maximum deposition weight was obtained and the deposit product was homogeneous. By contrast, the deposits obtained at pH 3, 4 and 5 were inhomogeneous. The electric current during EPD increased with decreasing pH reflecting an increase in the concentration H^+ . Tang *et al.* [6] fabricated

γ -alumina deposits by EPD using an aqueous γ -alumina suspension and found that the deposition weight of the γ -alumina depended on the suspension pH. The change of deposition weight of γ -alumina indicates differences in the transported H^+ and γ -alumina particles. When the number of H^+ ions in alumina suspension was increased by pH adjustment, the transport number of the H^+ during EPD became higher than that of the α -alumina particles. Consequently, the deposits obtained at pH < 6.8 were inhomogeneous. Moreover, gas evolution was observed from the electrode at pH < 6.8, showing that the best pH condition is 6.8 (not at a high ζ -potential since the isoelectric point of α -alumina is about 9.2). When the

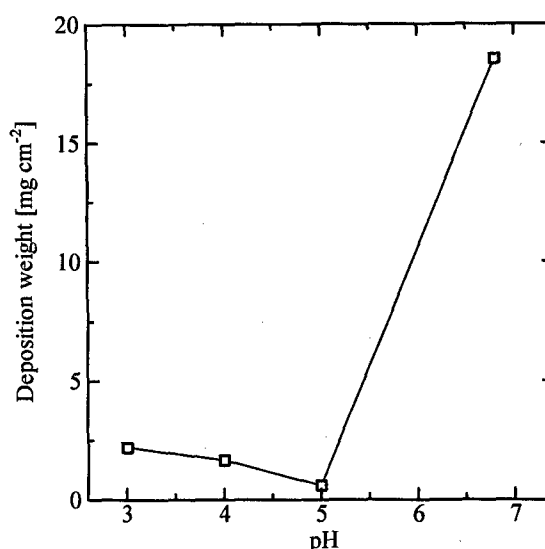


Fig.1. The relationship between the deposition weight and pH of suspension.

suspension pH is close to the isoelectric point, the dispersed alumina particles tend to agglomerate. It is thought that the agglomerated particles exist mainly at pH 6.8 and that deposition of the agglomerated particles leads to a large deposition weight.

Fig.2 shows SEM photographs of the EPD samples dried under atmospheric pressure or RH = 60%. These were obtained by EPD at pH 6.8 and an EPD time of 30s. The alumina film follows the undulating shape of stainless wire in both samples. Cracks were observed in the sample dried under atmospheric pressure (marked by an arrow in Fig.2a) but no cracks were seen in the sample dried under RH = 60% (Fig.2b). The stress developed by shrinkage of the alumina film during drying was suppressed by controlled drying at RH = 60%. Thus,

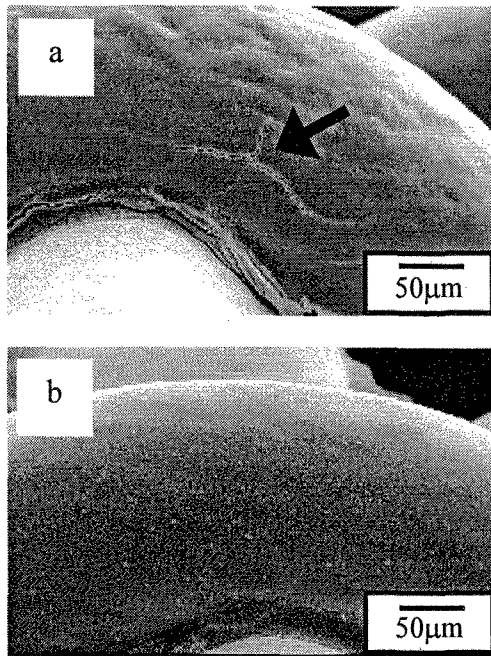


Fig.2 SEM photographs of the dried bodies.
a; under atmospheric pressure. b; under RH = 60%.

crack-free alumina films can be obtained by controlling the drying conditions. The microstructure of the crack-free alumina film was observed to be porous. This microstructure was essentially maintained even after firing at 1000°C 5h in air, the surface of the stainless steel wire remaining well covered with alumina film. It was therefore considered that this alumina film would exert a protective effect on the stainless surface during firing.

The X-ray diffraction (XRD) patterns of the various samples, A - D, are shown in Fig.3. The XRD peaks in the stainless steel wire net before firing corresponded to austenitic steel (Fig.3 A). After firing (Fig.3 B), peaks resulting from Fe_2O_3 and Fe_3O_4 were observed, arising from oxidation of the stainless surface by firing at high temperature in air. On the other hand, even after firing, only XRD peaks belonging to $\alpha\text{-Al}_2\text{O}_3$ appeared in the sample coated with α -alumina by EPD (Fig.3 C). When alumina coating was removed from stainless steel wire after firing (Fig.3 D), the XRD pattern showed little change from the pattern before firing. The stainless surfaces of samples B and D were observed by SEM are shown in Fig.4. The surface roughness of sample D resulting from oxidation at high temperature was clearly suppressed compared with sample B.

The X-ray diffraction results and the observation by

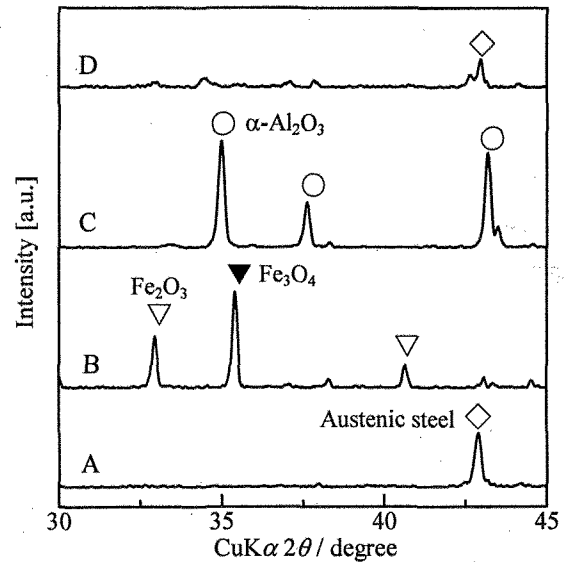


Fig.3. The X-ray diffraction patterns of the surfaces of various samples. (A); before firing without coating, (B); after firing without coating, (C); after firing with EPD alumina coating, (D); sample with the alumina coating removed from stainless steel wire.

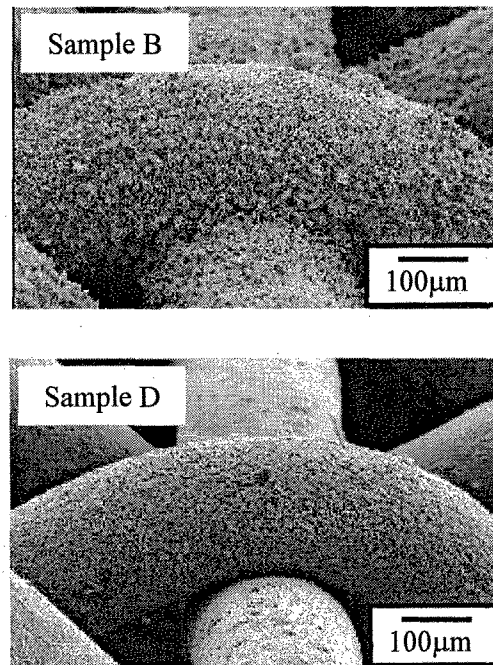


Fig.4 SEM photographs of stainless surfaces of sample B and sample D.

SEM showed that alumina coating by EPD is effective in suppressing oxidation of the stainless wire net at high temperature.

4. CONCLUSION

The deposition weight obtained by EPD depended

strongly on the suspension pH. At pH < 6.8, the transport number of H⁺ increased during EPD by pH adjustment and became higher than that of α -alumina particles. Gas evolution occurred from the electrode at these pH conditions. The best pH was therefore 6.8, without pH adjustment. The alumina deposit obtained at pH 6.8 and an EPD time of 30s showed deposition along the undulating shape of the stainless steel wire. Cracks can be suppressed by drying at RH = 60% to decrease the stress developed by shrinkage of the alumina film during drying. The microstructure of the deposits was maintained even after transformation into α -alumina had occurred on firing at 1000°C 5h in air. Good thermal oxidation resistance was provided by the EPD alumina coating on the stainless steel wire net.

5. ACKNOWLEDGEMENT

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6. REFERENCES

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