

Effect of Voltage and Suspension Medium on Stanum Oxide (SnO₂) Film Prepared By Electrophoretic Deposition (EPD)

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Abstract. The electrophoretic deposition (EPD) technique was applied in order to produce stanum oxide (SnO₂) coating on stainless steel. Voltages of 2V, 4V, 6V and 8V and two types of SnO₂ suspensions; (i) acidic (pH 3) and (ii) alkaline (pH 10) were studied. Depositions were carried out for duration of 10 minutes. It was found that weight of coatings produced using both acidic and alkaline SnO₂ suspensions increased proportionally with increasing voltage. However, the morphology of coatings in acidic SnO₂ was observed to yield better SnO₂ depositions through the EPD technique.

Introduction

Electrophoretic deposition (EPD) is an electrochemical coating method which is attracting increasing interest as a material coating technique [1]. It is a process achieved via the motion of charged particles, dispersed in solvent, towards an electrode under an applied electric field [2]. It has been used for the processing and fabrication of a wide variety of advanced ceramic materials and because of the high versatility of use with different materials and their combinations as well as its cost-effectiveness [3] and its ability to produce films on substrate of complex shapes and large dimensions [4]. EPD offers advantages of simplicity, uniformity of deposits, control of deposit thickness, microstructural homogeneity, and deposition on complex shaped substrates, including the potential to infiltrate porous substrates [2].

EPD processing method is applicable to any solid (metal, polymer and ceramic) available as fine powder (<30 μm) suspended as a colloidal suspension [4]. The colloidal processing technique has been used to fabricate solid oxide fuel cells, micro-laminate and functionally graded materials, ceramic-matrix/ ceramix-fibre composites and porous layers as membranes or thermal barrier coatings [5].

This coating method manipulates on a number of different parameters such as voltage deposition, time, and the type of coating electrodes suspension. Charged particles of SnO₂ suspension will migrate to opposite charged to form coatings when electrical current is supplied. When the suspended particles are positively charged, deposition occurs on the negative electrode (cathode) *via* a specific EPD process known as cathodic EPD. *Vice versa*, deposition of negatively charged particles on positive electrode (anode) is termed as anodic EPD [3,6].

The advantages of EPD are the capability to produce coatings on products with complex geometries, and films of variable thickness to be processed in a low cost and simple setup. Thus, EPD technique is more economical than conventional deposition methods such as sol gel, spraying, painting and other coating method [7].

The present work discloses the effects of voltage and suspension medium on the EPD of SnO₂. Further significant finding lays in the effects of the suspension medium pH to zeta potential values to the SnO₂ film morphologies. Investigations on EPD of SnO₂ in stable suspensions of acids and alkaline suspension have been performed by various researchers [8] but none have directly compared the coatings morphologies formed by using two stable suspensions of different pH.

Experimental Procedure

Aqueous SnO₂ suspension is first prepared by mixing SnO₂ (Aldrich Chemistry, US) powder with distilled water. SnO₂ with solid loadings of 0.5 wt% were used in all aqueous SnO₂ suspension. Afterwards, the pH of the SnO₂ suspensions were adjusted to pH 3 (acidic SnO₂ suspension) and pH 10 (alkaline SnO₂ suspension). pH adjustments were performed using nitric acid and ammonia. The SnO₂ aqueous suspensions were sonicated for five minutes before the EPD process was conducted. After the EPD of SnO₂ was completed, the coated stainless steel were dried at room temperature and sintered at 600°C at 2°C/min for an hour. The weights of the stainless steel prior to EPD and after EPD were recorded as for weight changes measurement with the analytical balance (Gmbh, Germany). Examinations of the coatings surface morphologies was perform by FESEM (JEOL Ltd., Japan) with X20,000 of magnification. The phases formed in the coatings were identified using X-Ray Diffraction (Bruker AXS Gmbh, Germany), (Spectra Services, USA). The SnO₂ coatings was analysed to present phases in the 2θ range 20° - 70°.

Result and Discussion

The phases of all the coatings obtained from acidic and alkaline SnO₂ suspension EPD were identified as SnO₂ cassiterite (JCPDS: 00-041-1445) and Iron Chromium (JCPDS: 00-054-0331). The detection of both coatings material and substrate by the XRD analysis is due to the resultant thin layer deposition of SnO₂ on the stainless steel substrate which caused the penetration of the x-rays into the substrate as well. Figure 1 (a) and (b) shows the XRD phase for SnO₂ coating in acidic and alkaline suspension on stainless steel at 8V.

The XRD analysis shows that the SnO₂ phase was observed in all the coated stainless steel. Thus, this indicates that successful deposition of SnO₂ was obtained in all the samples, regardless of the types of suspension and voltage used.

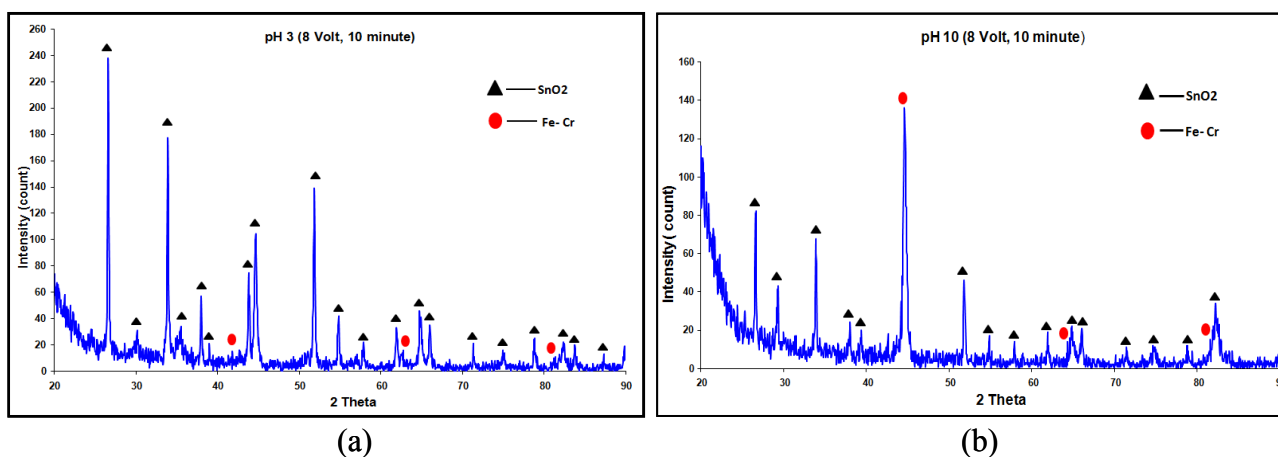


Figure 1: XRD phases for SnO₂ coating in (a) acidic and (b) alkaline suspension at 8V.

Generally, different suspensions *i.e* acidic and alkaline will affect the coating of the EPD process [8]. For both acidic and alkaline SnO₂ suspensions, the depositions occur on negative substrate which implies that the movements of charged SnO₂ particles are from the cathode to anode. The deposition of SnO₂ in both acidic and alkaline suspension on anode is due to the same type of SnO₂ particle charge which is the negative charge. This is supported by the zeta potential analysis result shown in Figure 2, by Edson R. and Caue R.,[9]. It was clearly indicated that the zeta potential values of both pH 3 and pH 10 suspensions are in negative values range. The positive values zeta potential was observed only up to ~pH 2. As the pH increases to pH 4 which is still acidic, the zeta potential value changed to a negative value. Subsequently, as the pH values continue to increase, the zeta potential values increase until it starts to be constant at ~pH9.

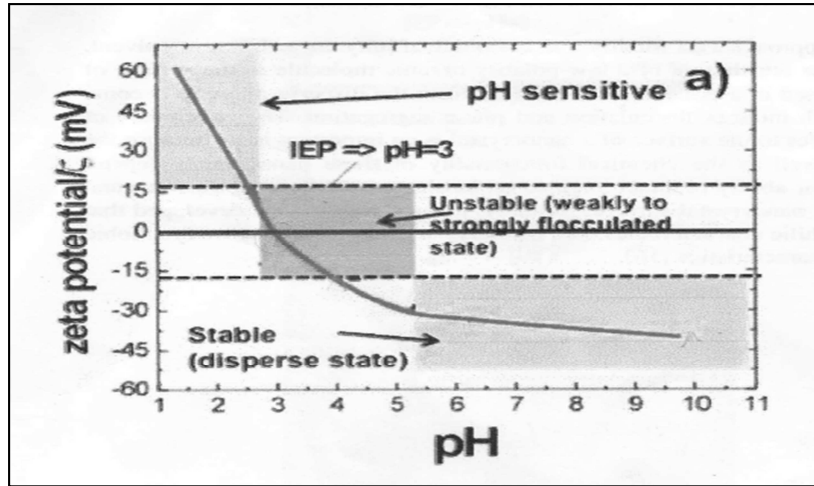


Figure 2: Typical plot of zeta potential as function of ph for SnO₂ nanocrystal colloidal dispersion in water[9].

Morphology of coatings

Overall, the uniformity and homogeneity of the coatings surfaces observed are increased from 2V to 8V in SnO₂ acidic and alkaline suspension. It is clear that the morphology of coatings is more compact and the uniformity of coatings is increased. This condition is supported from work by Radice *et al* [10] which also observed that when the voltage increased, the resultant coating shows increased uniformity and homogeneity.

At 2V and 4V, resultant coatings from both alkaline and acidic suspensions are incompact and inhomogeneous. However, the resultant coating from alkaline suspension is less compact and dense from observation through by FESEM micrograph as compared by coatings from SnO₂ acidic suspension. This can be due to the high zeta potential values of the SnO₂ which lead to highly stable SnO₂ suspension. The strong repulsive force in between the SnO₂ particles would not be overcome by the applied electric field and thus hindering depositions [6]. Meanwhile, gradient of the zeta potential value at the pH 2.5 to pH 5.5 indicated unstable zeta potential values which led to formation of unstable suspension. This situation would have assisted in facilitating the movement of particles in suspension during the application of the electric field [8,11]. Table 1 clearly shows that SnO₂ acidic suspensions of pH 3 is better in forming coatings by EPD due to the obtained compact and uniform SnO₂ coatings.

Table 1: The morphology of SnO₂ coatings in acidic and alkaline suspension at 8V.

Parameters	Acidic	Alkaline
8V, 10 minutes		

Weight of coatings

Generally, the percentage of weight changes of SnO₂ coatings in both acidic and alkaline SnO₂ suspensions increase as amount of voltage increase. Figure 3 (a) and (b) shows the graph effect of voltage of acidic and alkaline SnO₂ suspension.

From figure 3 (a), at the low voltages of 2V and 4V only slight weight changes was obtained because the coating formed on the substrate is too thin. While the weight difference can be seen in the thicker coating formed of the high voltage at 6V and 8V. From figure 3(b) showed that there was no change in weight at 2V because the resulting coating is too thin. However, the weight changes are more increased when the voltage increased at 4V, 6V and 8V.

The percentage of weight changes SnO₂ coating in both suspensions (acidic and alkaline) increases as voltage increase. The high voltage value provides better electric field on the charge particles in suspension compared with the suspension at low voltage [6,10].

(i) Voltage effect

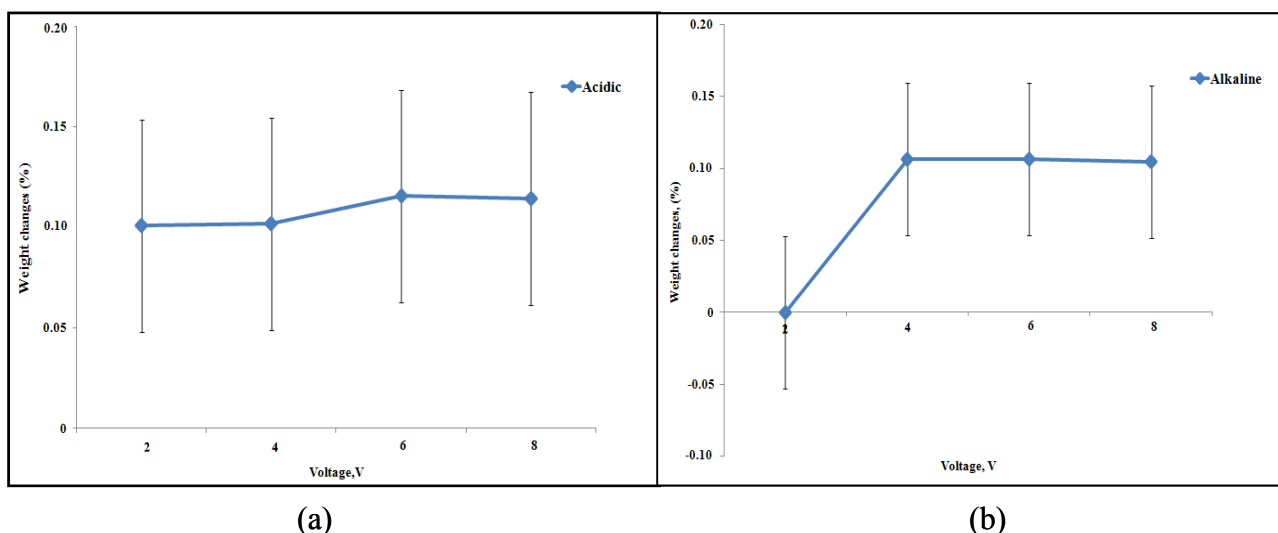


Figure 3: Voltage of effect on percentage of weight changes in (a) acidic and (b) alkaline SnO₂ suspension.

(ii) Suspension effect

As shown in Figure 4, at 2V coatings deposited from alkaline suspensions shows no percentages of weight change which may have resulted from very thin coatings produced. Since very thin coatings were formed, thus the weight changes may be very minimal, which in this charge. Furthermore, this also agrees well with the non-uniform and inhomogeneous (Table 1) coating surface observed.

At 4V, the weight changes observed in alkaline SnO₂ suspension were higher than acidic suspension. This shows the very minimal deposition of SnO₂ particles suspended in acidic suspension caused no weight changes of coated substrate. This is also related well to the morphology of coatings surfaces in which the surface morphology from alkaline is denser than the acidic SnO₂ suspension.

At 6V and 8V, high percentages of weight change of SnO₂ depositions can be clearly seen from SnO₂ suspended in both acidic and alkaline suspension. Moreover, the percentage of weight changes recorded from SnO₂ suspended in acidic suspension are higher than in alkaline suspensions. Thus, this again indicates that the EPD of SnO₂ suspended in acidic suspension is better than alkaline suspension.

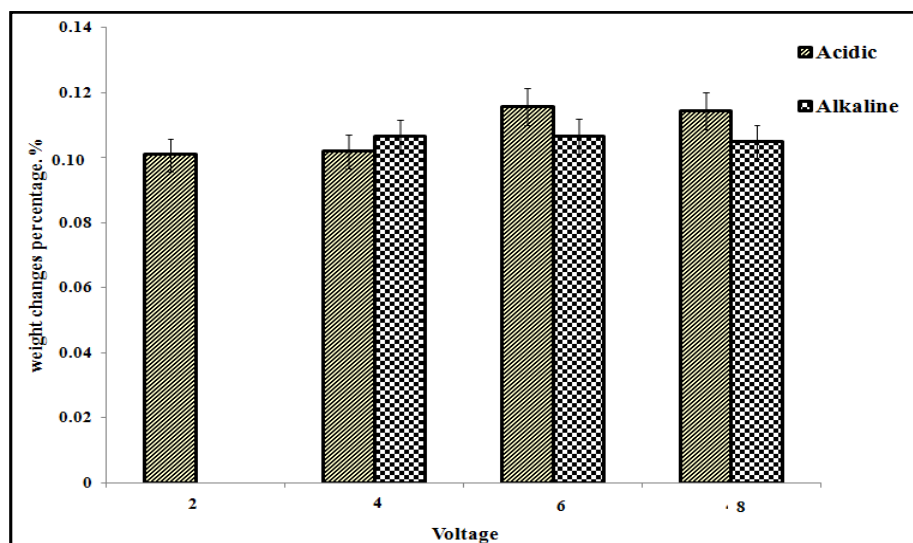


Figure 4: Percentage of weight changes of SnO₂ coatings in acidic and alkaline suspension.

Conclusion

Overall, EPD of SnO₂ in acidic suspension yielded better SnO₂ coatings. It was observed that the morphology of SnO₂ coatings from acidic suspension are more uniform, homogeneous and dense in packing, compared to SnO₂ coatings formed from alkaline SnO₂ suspension. The thicknesses of SnO₂ coatings from acidic suspension are also generally higher than SnO₂ coatings obtained from alkaline suspension. The better quality SnO₂ coatings obtained from acidic suspension should be due to the difference in the suspension stability and zeta potential values. In terms of voltage increment, the increased voltage resulted in a more uniform, homogeneous and dense coating. The thicknesses of coating are also found to be proportional to the applied voltage in which higher voltage produces higher electrical field, thus promotes better mobility and deposition of particles to form coatings.

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