

Research Article

Electrophoretic Deposition of Aluminum Nitride from Its Suspension in Acetylacetone Using Iodine as an Additive

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We have studied electrophoretic deposition of AlN from its suspension in acetylacetone with I₂ as an additive. AlN powder with particle size <10 μm is dispersed to produce a positive charge and deposited on the cathode by applying fields greater than 10 V/cm between the electrodes. X-ray diffraction and FTIR studies indicate that the AlN before and after deposition has the same composition and structure. An increase in the amount of AlN in the suspension, the deposition potential, and the deposition time results in a linear increase in the weight of the AlN deposited. Electrophoretic deposition from 10 g/L AlN suspension shows an initial increase in the weight of AlN deposited with the concentration of I₂, and the weight of AlN decreases after reaching a maximum at 0.20 g/L I₂.

1. Introduction

In this paper, we describe a simple method of coating aluminum nitride, AlN, using an electrophoretic deposition technique in nonaqueous medium. We performed the coating from a suspension of AlN in acetylacetone using I₂ as an additive on the Al cathode. We provide material characterization data such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier transform infrared (FTIR) spectra of the coated film. We also discuss the effect of various parameters such as potential, deposition time, and the amount of I₂ and AlN present in the suspension on the amount of AlN deposited.

AlN coating has a wide range of practical application. It is used as an electronic packaging material due to its high thermal conductivity, 320 W/(mK), and electrical resistivity, >10¹³ Ωcm [1–3]. It has also applications in the optical industry for dielectric and protective coatings, thin film transducers, and surface acoustic wave (SAW) devices [4–7].

Various physical and chemical methods are used to prepare AlN films. These include chemical vapor deposition,

reactive sputtering and evaporation, and ion beam nitridation [8–16]. Electrophoretic deposition is an alternative method of coating. In electrophoretic deposition, charged particles migrate independently of one another in a suspension under the influence of an electric field and are deposited onto an electrode. The appealing features of this coating method are that the coating can be performed within a short period, it does not require expensive apparatus, it is well suited for coating irregularly shaped objects, and it is suitable for mass production. The theoretical and its general advantages and applications for ceramic coatings have been well documented in a review by Sarker and Nicholson [17] and van der Biest and Vandeperre [18]. The theoretical and experimental development of the technique has also been reviewed by Heavens [19]. Various reports have appeared in the literature on the application of the technique for ceramic coatings [18–37].

Electrophoretic deposition from aqueous or nonaqueous suspensions containing water has problems due to the electrolysis of water that takes place together with the deposition. The production of H₂ and/or O₂ gases during the electrolysis

prevents the formation of a well-adhered and uniform film. The electrolysis of water also lowers the current efficiency of the electrophoresis process. A longer period of coating is thus required for the deposition of powdered layer of significant thickness.

These problems of electrophoretic deposition in the presence of water are entirely eliminated using nonaqueous organic media such as benzene or ketones. The oxidation-reduction potentials of these organic solvents are extremely high, thus preventing the formation of gases due to electrolysis. This improves the quality of the coated film and also results in high current efficiency. Electrophoretic coating from organic solvents, however, requires several hundred volts of potential since the charges adhered to the particles in the suspension are extremely low due to the small amount of free ions present in the pure solvents. This problem has been solved by using I_2 as an additive in acetylacetone as the suspension media [20]. The reaction of acetylacetone and iodine produces free protons that will be adsorbed on the particles of the suspension increasing their surface charges.

Electrophoretic deposition of AlN ceramic coating has been reported by various workers from its suspension in ethanol using various additives [21–26]. In the work of Mortiz and Reetz [21], poly(acrylic acid)-coated AlN powder emulsified in ethanol was deposited on Pt resulting in an AlN coating. Vandeperre et al. [22] deposited AlN on a cathode from ethanol in the presence of acetic acid. Mortiz and Müller [23] electrophoretically deposited AlN from ethanol using polyacrylic acid and triethylamine as additives. Jian-Feng et al. [24] used polyacrylic acid as a dispersant for EPD of AlN from its suspension in ethanol. Abdoli et al. [25] used ethanol in the presence of iodine as its suspension medium for the coating of AlN. Zhang and coworkers [26] conducted electrophoretic deposition of AlN from its suspension in ethanol in the presence of triethylamine.

Wade and Crooks [27, 28] electrophoretically deposited AlN polymer precursor on n-Si cathode from a suspension in CH_3CN and calcined the deposited film at a temperature of 1100 C in flowing NH_3 to AlN ceramic coating. Our work [29, 30] has shown that electrophoretic deposition can be performed from the suspension of AlN in acetylacetone using iodine as an additive

2. Experimental

AlN powder (98% pure and particle size $<10\ \mu m$) from Aldrich was used with no further treatment. The suspension was prepared in a solution of I_2 (EM Science, 99.8% pure) in acetylacetone (Aldrich). Unless otherwise noted, 10 g/L AlN suspension in 0.2 g/L I_2 /acetylacetone was used for all electrophoretic deposition works.

Electrophoretic deposition was carried out in a cylindrical cell of about 3.0 cm diameter and a capacity of 50 mL. The cathode and the anode used were 1×3 cm Al (Johnson-Mathey, 98.5% pure, and 1.00 mm thick), and they were approximately positioned 1 cm apart parallel to each other. 20 mL suspension was used for all works. The Al cathode was cleaned with soap water, dipped in 1 M H_2SO_4 acid (heated

to a temperature of about 75 C), rinsed with deionized water and ethanol, and finally dried under a stream of N_2 . A potential of 10 to 50 V was applied with a DC power supply (Sorensen Model DCR 150-3B produced by Raytheon Co. or BK Precision Model 1602 High Voltage Power Supply). For all potentials used, a well-adhered electrophoretically deposited film was observed on the cathode covering the entire electrode. Our adherence test of the films involved withstanding of a light wiping with a lab wipe followed by washing with ethanol and N_2 drying. The weight of the deposit was obtained by taking the weight difference of the Al cathode before and after electrophoretic deposition.

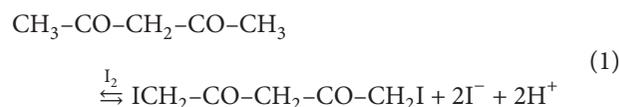
Prior to the electrophoretic deposition, the suspension was ultrasonically stirred for about 1 h to break up the AlN powder particles. In order to prevent settling of AlN powder, the suspension was stirred gently and continuously with a magnetic stirrer by the time the deposition was carried out. Conductivity and pH of the I_2 /acetylacetone solution before and after adding AlN were measured at room temperature 5 min after the electrodes were immersed in the suspension.

To identify the structure of the coated film, powder X-ray diffraction (XRD) was conducted on both a scraped powder and the deposited film using a Philips/Norelco computer-controlled diffractometer using $Cu\ K\alpha$ radiation at 35 kV and 20 mA in Bragg-Brentano parafocusing geometry. The detector stepped from 10 to 90 degrees 2θ using a step size 0.05 degree/step and counting time of 4 s/step. To study the surface morphology of the deposited film, scanning electron microscopy (SEM) study was conducted using an Amray 1810 microscope with Oxford Instruments INCA frame-grabbing software.

FTIR analysis of the original powder of AlN before deposition and a film scraped from the Al cathode was performed using Nicolet MAGNA-IR 550 Series II spectrometer. The IR spectrum of the original powder of AlN was compared with the spectrum of the scraped film to see if any chemical species were adsorbed or incorporated in the film by the time the coating was performed. The samples for the FTIR study were prepared as KBr pellets.

3. Results and Discussion

3.1. Electrophoretic Deposition Study. Table 1(a) shows that the conductivity of I_2 /acetylacetone increases with an increase in I_2 . It has been reported that this increase in conductance with an increase in I_2 concentration is due to the production of free protons from the reaction of acetylacetone and I_2 , equation (1) [20]. This is in agreement with the pH results shown in Table 1(b). Consider



We observed a decrease in conductivity and an increase in pH upon addition of AlN to I_2 /acetylacetone, Tables 1(a) and 1(b). This suggests the reduction of the free protons by the AlN particles. We believe that this is due to the adsorption of protons on the surface of the AlN particles. This is in

TABLE 1: (a) Effect of AlN on the conductivity (κ) of I_2 /acetylacetone. (b) Effect of AlN on the pH of I_2 /acetylacetone.

(a)		
Amount of I_2 (g/L)	κ ($\mu\text{S/cm}$) of I_2 /acetylacetone with no AlN	κ ($\mu\text{S/cm}$) of I_2 /acetylacetone with 10 g/L AlN
0	0.7	0.8
0.1	10.9	9.4
0.2	21.8	19.7
0.3	35.4	32.7
0.4	47.8	44.6
0.6	73.1	70.2
0.8	99.4	96.9

(b)		
Amount of I_2 (g/L)	pH of I_2 /acetylacetone with no AlN	pH of I_2 /acetylacetone with 10 g/L AlN
0	5.01	5.04
0.1	4.59	4.65
0.2	4.29	4.56
0.3	3.92	4.29
0.4	0.91	1.89
0.6	0.05	0.76

agreement with the electrophoretic deposition result, where the positively charged AlN particles undergo electrophoretic migration in an electric field and selectively coated on the cathode.

The weight of the coated film in the electrophoretic deposition is a function of various factors. These are the amount of the particles in the suspension (C), the permittivity of vacuum (ϵ_0), the relative permittivity of the dispersing medium (ϵ_r), the zeta potential of the particles (ζ), the viscosity of the solvent (η), the applied potential (E), the distance between the electrodes (L), and the deposition time (t). In the initial period of the deposition, ignoring the charge carried by the free ions, the weight of the charged particles deposited per unit area (w) is expressed using (2) [20, 31] as follows:

$$w = 2/3 [C\epsilon_0\epsilon_r\zeta\eta^{-1}EL^{-1}t]. \quad (2)$$

From (2), it can be seen that the deposition yield increases with any of the parameters except the viscosity of the solvent and the distance between the electrodes.

To investigate the effect of I_2 concentration on the amount of AlN deposited, we conducted electrophoretic deposition studies at 20 V for 5 min on 10 g/L of AlN suspension in various concentrations of I_2 /acetylacetone, and the result is shown in Figure 1. Figure 1 illustrates an initial increase in the weight of AlN deposited with the concentration of I_2 and a decrease after reaching a maximum at 0.20 g/L I_2 . Ishiria and coworkers observed and explained similar phenomenon for electrophoretic deposition of Y_2O_3 -stabilized ZrO_2 film [20].

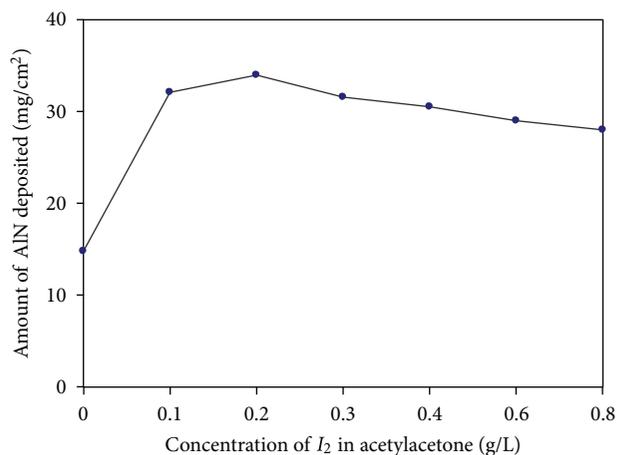


FIGURE 1: The effect of I_2 concentration on the weight of AlN deposited for the electrophoretic deposition of AlN in I_2 /acetylacetone. Deposition potential and time are 20 V and 5 min, respectively. The amount of AlN in the suspension is 10 g/L.

An initial increase in the amount of AlN deposited is due to the adsorbed proton that increases the surface charge of AlN with a subsequent increase in the zeta potential (ζ) and the amount of AlN deposited, equation (2). Further addition of I_2 results in the formation of free protons in the suspension. Since the mobility of the protons is much higher than the AlN particles, most of the charges will be carried by the protons, thus decreasing the amount of AlN deposited. Figure 1 also demonstrates that the highest deposition rate of AlN took place in 0.2 g/L I_2 /acetylacetone, in which the maximum weight of the coated AlN was obtained. We conducted all the subsequent studies using AlN deposited in this medium of I_2 /acetylacetone.

To examine the effect of deposition time on the weight of AlN deposited, we conducted electrophoretic deposition at 20 V for a time range of 0 to 5 min. Figure 2 shows that the weight of the coated AlN increases linearly with the deposition time according to (2). We observed a deviation from this linearity at longer duration of electrophoresis. This is due to the smaller value of the mobility of the positively charged AlN particles than the theoretical value used in the derivation of (2) [20]. As the deposition time becomes longer, the initially coated film and ions accumulated at the electrode surface shield the electric field applied to the suspension. This decreases the mobility of the AlN particles from the theoretical value.

Figures 3 and 4 show the effect of deposition potential and the amount of AlN in the suspension on the weight of the coated AlN film, respectively. In both cases, there is a linear increase of the deposited weight of AlN in agreement with (2).

3.2. Characterization of the Deposited Film. The surface morphology of the Al substrate and the AlN film on Al substrate is shown in the SEM micrographs of Figures 5(a) and 5(b). Figure 5(b) shows the SEM micrograph of AlN film deposited from 10 g/L AlN suspension in 0.2 g/L I_2 /acetylacetone at

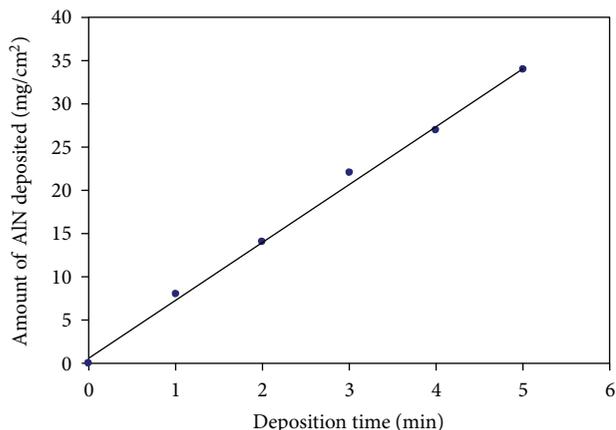


FIGURE 2: The effect of deposition time on the weight of AlN deposited for electrophoretic deposition of AlN in 0.2 g/L I_2 /acetylacetone. Deposition potential and the amount of AlN in the suspension are 20 V and 10 g/L, respectively.

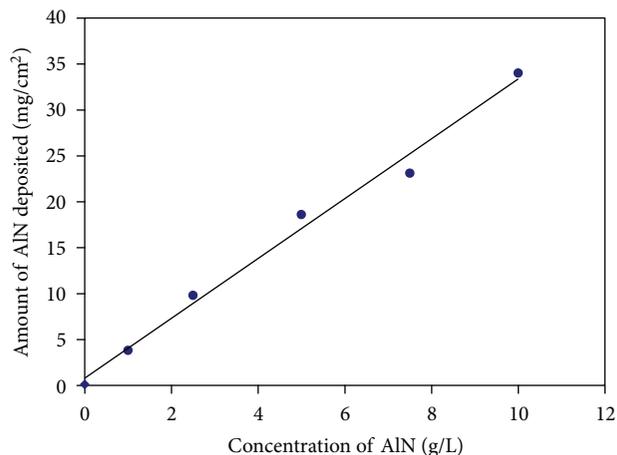


FIGURE 4: The effect of the amount of AlN in the suspension on the weight of AlN deposited for electrophoretic deposition of AlN in 0.2 g/L I_2 /acetylacetone. Deposition potential and time are 20 V and 5 min, respectively.

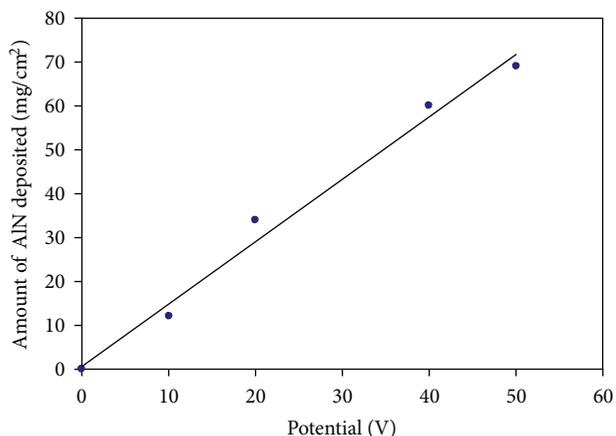


FIGURE 3: The effect of potential on the weight of AlN deposited for electrophoretic deposition of AlN in 0.2 g/L in I_2 /acetylacetone. Deposition time and the amount of AlN in the suspension are 5 min and 10 g/L, respectively.

a potential of 20 V for 5 minutes. The deposited material was continuous and well adhered.

The broad X-ray diffraction patterns of the Al foil, the AlN powder, the coated foil, and the coating scraped from the Al foil after deposition are shown in Figure 6(a).

Figure 6(b) shows the detail of the AlN coated Al foil sample. Table 2 lists the observed XRD reflections of AlN powder, coated Al foil, and the scraped coating, along with the d-spacings for Al and AlN standards. All of the observed peaks can be adequately indexed using the ICDD standard patterns for cubic Al (ICDD no. 4-787) and hexagonal AlN (ICDD no. 25-1133), indicating that the AlN starting powder and deposited AlN coatings are single phase. The 2θ measurement precision is ± 0.05 deg, which yields d-spacing precision of ± 0.003 Å at $2\theta = 40$ deg and ± 0.0003 Å at $2\theta = 80$ deg. The measured d-spacings are in reasonable agreement with the standard values within this measurement precision. The AlN powder, the AlN coating, and the scraped AlN coating

all exhibit random intensity patterns, corresponding closely to the published standard. The XRD peak profiles of Al and AlN peaks do not exhibit anomalous broadening, indicating that the diffracting domains have a narrow lattice parameter distribution, with domain size greater than 100 nm.

FTIR transmission spectra of the original AlN powder before coating and the scraped powder of the deposited film from the cathode are shown in Figure 7. The broad peak at about 800 cm^{-1} in both cases is due to the Al-N stretching vibration. From Figure 7, it is also possible to see that the scraped film powder has almost the same transmission spectrum as the original powder. This shows the absence of chemical species adsorbed or incorporated from the suspension into the film during electrophoretic deposition.

4. Conclusion

In this study, we have shown that electrophoretic deposition of AlN in nonaqueous medium, acetylacetone, leads to the formation of a deposited film on the Al cathode with I_2 used as an additive. The weight of the AlN deposited shows a linear increase with the weight of AlN in the suspension, deposition time, and the potential applied in agreement with the theoretical prediction.

Nonaqueous electrophoretic deposition has an advantage of producing a better quality coating than aqueous suspensions. This is due to the absence of H_2 and/or O_2 gases that will be produced from the electrolysis of water. Its higher current efficiency also makes it suitable for quantitative production of powdered layer in a shorter period of time than aqueous suspension. Compared with other coating techniques of AlN such as chemical or vapor deposition, ion beam nitridation, or reactive sputtering, electrophoretic deposition has some general advantages. These include short period of coating, little restriction on the shape of the substrates, and use of simple deposition apparatus.

TABLE 2: The observed reflections of AlN powder, coated Al foil, the scraped coating, and *d*-spacing for Al and AlN standards.

Al foil	AlN powder	Coated Al foil	Scraped coating	ICDD 4-787 Al	ICDD 25-1133 AlN
	2.696	2.704	2.700		2.695
	2.493	2.499	2.496		2.490
	2.372	2.378	2.375		2.371
2.342		2.348		2.338	
2.028		2.030		2.024	
	1.829	1.833	1.831		1.829
	1.556	1.559	1.557		1.5559
1.433		1.435		1.431	
	1.413		1.414		1.4133
	1.347				1.3475
	1.320	1.322	1.321		1.3194
	1.301				1.3007
1.221		1.223		1.221	

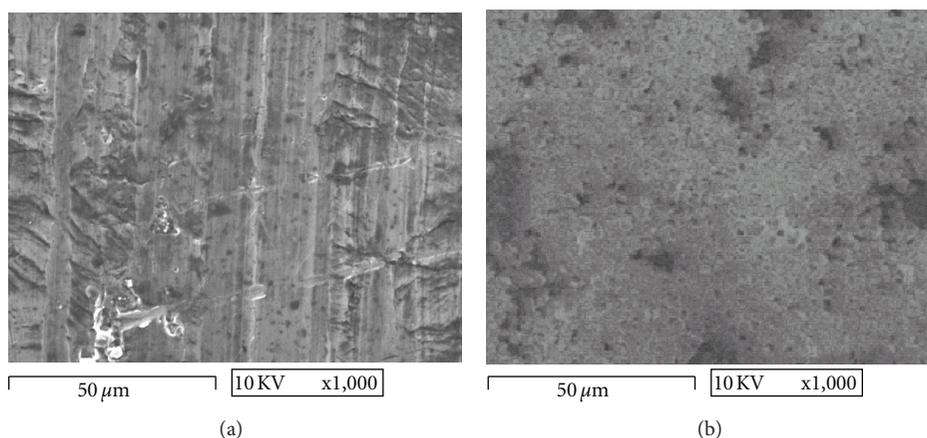


FIGURE 5: (a) SEM micrograph of bare Al substrate. (b) SEM micrograph of an AlN coating on Al cathode for electrophoretic deposition of AlN in 0.2 g/L I_2 /acetylacetone at a potential of 20 V. Deposition time and the amount of AlN in the suspension are 5 min and 10 g/L, respectively.

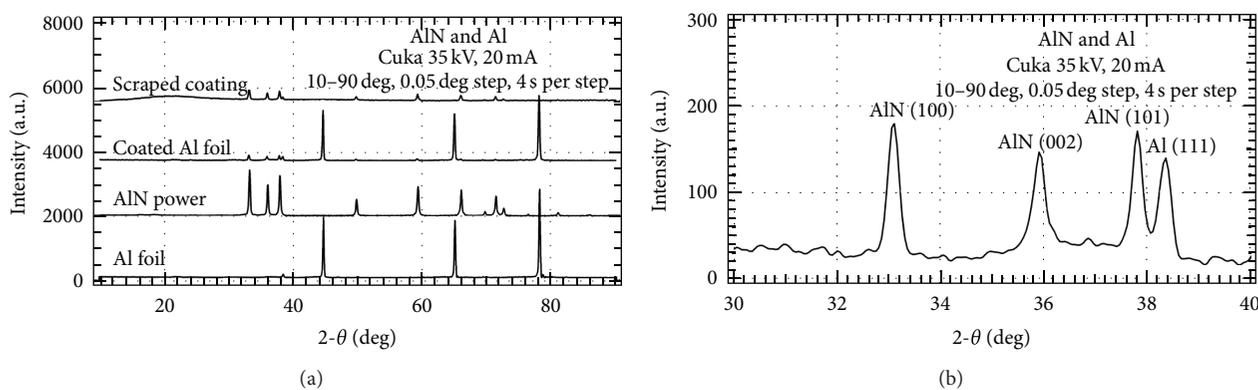


FIGURE 6: XRD diffraction patterns of (a) an Al cathode foil, AlN powder before coating, an Al cathode with AlN, and AlN film scraped from the Al cathode. (b) Detail of the AlN-coated Al cathode sample.

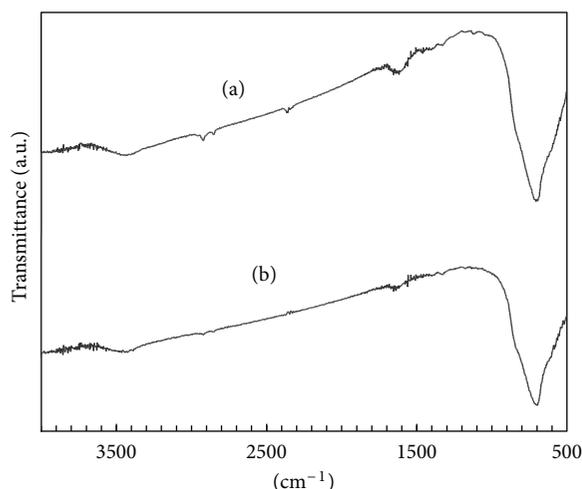


FIGURE 7: Transmission FTIR spectra: (a) The original AlN powder before electrophoretic deposition; (b) The AlN film scraped from the Al cathode.

Conflict of Interests

All the authors of the paper declare that they do not have a direct financial relation with any commercial identity mentioned in the paper that might lead to a conflict of interests for any of the authors.

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