

NANOSTRUCTURED ZRO2 AS AN ANTI CONTAMINATION COATING

FOR HV INSULATOR – AN EXPERIMENTAL ANALYSIS

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ABSTRACT

Porcelain electric insulators act as a primary support device for transmission lines. Being installed in the outer atmosphere these can exhibit flashover due to expositing to pollutant environments such as industrial areas, deserts and sea air, containing carbon, sand and salt. There has to be a new innovative method to mitigate the above problem and one such problem is coating these insulators with the nanostructured thin film. The present investigation aims to develop and characterize dielectric and hydrophobic zirconium oxide thin films, deposited on porcelain insulators surfaces, using a direct current (DC) magnetron sputtering. A zirconium target with 99.999% purity was used as precursor and argon gas with 99.999% purity was employed. Oxygen was used as a reactive gas. Sputtering power (30, 40, 50 and 60W) has been varied in order to investigate the optical and hydrophobic property. Optical and hydrophobic property was performed using UV-Nis-NIR spectrophotometer and contact angle goniometer (CAG), respectively. Surface roughness was also calculated using atomic force microscopy (AFM). Maximum contact angle was found to be 108.2^{0} which show that the film was hydrophobic in nature. Dielectric constant shows an increasing trend up to 60W power. It attains a maximum value of ~24.6 at 60W power which is very close to the bulk value.

KEYWORDS: Insulators, Flashover, Optical, Dielectric & Hydrophobic

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INTRODUCTION

The outside environment where electrical insulators are uncovered endorse dirt accumulation, results in degradation and a drastically reduce dielectric properties. In addition, if a dirt layer is accumulated, the flashover results in terrible failure in the device (Su et al., Reddy et al., and Farzaneh et al). In order to lessen these harms, the electrical insulators are manually or automatically washed with water jets; a high-cost and frequently required process, that also exposes the system operator to a small safety situation (Portella et al). Hafnium oxide (HfO), sulfur-oxygen bonds (S–O) and titanium oxide films are pointed out in literature as suitable to increase dielectric and hydrophobic properties of electrical insulators (Dave et al, Tu et al. and Castano et al).

Wettability is an imperative property of the material surface, as the facility of a surface to be damp or not, categorized as hydrophilic or hydrophobic. The hydrophobic property defines surface self-cleaning. The contact angle measurement (θ) between the solid/liquid interfaces is used to classify the surfaces. According to ASTM D7334-08, contact angles < 45° are hydrophilic, and > 45° are hydrophobic (ASTM D7334-08). Contact angles < 10° are super-hydrophilic and super-hydrophobic > 150° (Bhushan et al and Ma et al). Thereby, hydrophobicity is the surface facility to stay dirt free and dry, as the water slides down the surface, moving away dirts (Ma et al). The present research aims to develop and characterize ZrO₂ thin films deposited on porcelain (glass) insulators, to

guarantee dielectric properties as well as self-cleaning hydrophobic surfaces, using DC magnetron sputtering.

METHODS

Substrate Preparation

The ZrO_2 films were deposited by DC-magnetron sputtering on glass substrates in a custom designed 12-in. diameter chamber (Excel Instruments, India) at room temperature. A 2-inch diameter and 5-mm thick zirconium target of 99.99% purity were used for the sputtering. The substrates were thoroughly cleaned by rinsing in an ultrasonic bath of acetone and dried for two minutes. The chamber was initially evacuated by a turbo molecular pump backed by a rotary pump. During each deposition, the base pressure kept at 5×10^{-6} Torr and the distance between the substrate and the target was kept at 41 mm. The sputtering was carried out in an argon (99.999 % pure) atmosphere while oxygen was used as a reactive gas. The flow rate of oxygen and argon in the chamber was 10 sccm and 40 sccm respectively. The gas pressure was kept 15 mTorr for all depositions. The ZrO_2 coating was fabricated by the sputtering parameter that is power. The sputtering power was varied from 30 to 60 W keeping deposition time 90 minutes. Before starting the actual experiment, the target was pre-sputtered for 15 min with a shutter located in between the target and the substrate. This shutter was used to control the deposition time.

RESULTS

X-ray diffraction analysis was carried out to determine the preferred orientations and structural transformation of ZrO₂ films on a glass substrate at different sputtering power. Figure 1 shows the XRD pattern of the nanocrystalline films of zirconium oxide deposited at varying DC power. The XRD curve clearly reveals that all deposited films are monocrystalline in nature and the dominant peak occurs at $20 \approx 28^{0}$ which corresponds to (-111) orientation of monoclinic phase of ZrO₂ (Pengtao et al.). The other less dominant peak belongs to (011 and (-221) orientation. While varying sputtering power, it is observed that initially, the ZrO₂ film exhibit (-111) preferred orientation but with increasing power (111) becomes the preferred orientation at $20 \approx 31.468^{0}$. The XRD results of the ZrO₂ films with varying sputtering power may be interpreted on the basis of stress and strain evolution mechanism (Chawla et al). The compressive stress induced in the films contributes to the development of (-111) orientation and it may have relaxed to tensile mode at higher thickness favoring the (111) preferred orientation. Table 1 shows the influence of varying sputtering power and crystallite size as shown in table 5.11. It may be mentioned that with an increase in power, the deposition rate increases, which in turn increases the thickness of the deposited film (Jeyachandran et al).



Figure 1: XRD Patterns of ZrO2 Films Deposited at the Different Sputtering Power

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The surface morphology of the ZrO_2 films was studied using AFM. The three dimensional AFM images obtained for ZrO_2 films deposited at various DC power at 15mTorr pressure are shown in figure 2. The AFM micrographs correspond to a scan area of $2\mu m \times 2\mu m$. It can be seen that the zirconium oxide films have same morphologies. They all are constituted with a large number of nanoscale asperities. Each asperity consists of surface nanograins that are used to form a surface peak. However, the size and roughness of these varies with sputtering power. Table 1 shows rms roughness of ZrO_2 samples at different sputtering power. It is clear that as sputtering power is increased from 30 to 60W, roughness value also increases from 8.05 nm to 62.8 nm and then it decreases with a further increment of power. This behavior of roughness may be correlated to the particle size. The increment in power up to 60W increases the particle size which in turn enhances the roughness while decrement in particle size beyond 60W power reduces the roughness. The O/Zr ratio is approximately two for the film deposited at 50 and 60 W power while for all other deposited samples, it is less than two. The O/Zr ratio less than two is clearly indicative that zirconium was not fully oxidized for the samples deposited at sputtering power other than 50 and 60W. This can be associated with the fact that the increased kinetic energy due to increment in DC power enhanced the activity of the sputtered particles and thus, facilitated the formation of the Zr-O bond. Therefore, the oxidation of Zr atoms is increased and the oxygen vacancy in the thin films is decreased with increasing DC power.



Figure 2: AFM Micrographs of Film Deposited at Different Sputtering Power

The wettability of ZrO_2 coatings was evaluated by measuring the water contact angle using sessile drop method at ambient temperature. The distilled deionized water droplets (about 3 µl) were dropped on the deposited coating surfaces using a microsyringe. The average value of the water contact angle was determined by experimental drop profiles at five different positions for the same sample. The variation of water contact angle and rms roughness of ZrO_2 films as a function of sputtering power is shown in figure 3. All the deposited samples were found to be hydrophobic and the contact angle lies in the range $103.23^0 - 108.2^0$. The contact angle and surface roughness bear a linear relationship, thus satisfying the Wenzel model. The maximum contact angle (108.2^0) occurs at a sputtering power of 60 W where roughness is also high (62.813 nm).

Sputtering Power (Watt)	Crystallite Size (XRD) (nm)	RMS Roughness (AFM) (nm)	wt%Zr	wt%O
30	35.63	8.05179	35.27	64.73
40	48.49	31.0886	33.18	66.82
50	58.01	47.5282	31.85	68.15
60	78.17	62.813	31.05	68.95

Table 1: Influence of Deposition Parameter (Sputtering Power) onCrystallite Size, Surface Roughness of ZrO2 Thin Films



Figure 3: Contact Angle and Roughness of Deposited ZrO₂ Films as a Function of Power

The dielectric constant of the film was calculated by forming a stack of ITO/ZrO₂/Ag. The top electrode of Ag was fabricated using shadow mask through sputtering. Figure 4 shows the plot of the dielectric constant of a ZrO₂ film as a function of frequency in the range of 48.5 - 52.5 Hz. This low-frequency range is selected because an outdoor insulator operates in this zone only. It is clear from the figure that the dielectric constant of the deposited sample at a particular sputtering power practically remains constant within the prescribed frequency range. However, dielectric constant shows an increasing trend up to 60W power. It attains a maximum value of ~24.6 at 60W power which is very close to the bulk value (Robertson). Further increment in power i.e. at 70W power, a reduction in dielectric constant was observed. This behavior of dielectric constant with increasing power for ZrO_2 is readily correlated to the thickness of the films. Natori et al. theoretically concluded that as thickness decreases, dielectric constant also decreases (Natori et al). Hwang et al. also showed through experiments that the dielectric properties of high dielectric thin films are critically dependent upon the film thickness (Hwang et al). Figure 3 shows the variation in thickness with DC power. The dielectric constant is maximum ($\varepsilon_{max} = 24.6$) for 595 nm thick film which is also highest thickness among deposited samples.

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Figure 4: Sputter Power Effect on Dielectric Constant and Thickness of ZrO₂

CONCLUSIONS

The nanostructured zirconium oxide films were developed on glass insulator at different DC sputtering power. The microstructural characterization shows that all the samples were monocrystalline in nature with adequate surface roughness to have high hydrophobicity. High dielectric constant ($\varepsilon_{max} = 24.6$) of the deposited film gives an indication that film can bear high electric field applied to outdoor insulators. Thus, parameters of the hydrophobic ZrO₂ film deposited at 60W power found to be optimum for the glass insulator.

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