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Effect of sputtering power and substrate temperature on structural, optical, wettability and anti-icing characteristics of aluminium doped zinc oxide

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Abstract

Thin films of ZnO:Al were synthesized on glass substrates by RF magnetron sputtering. Structural, optical, wettability and anti-icing properties of the thin films are studied as a function of substrate temperature and sputtering power. XRD patterns showed an increase in the intensity of (002) peak when the sputtering power and substrate temperature are increased. The roughness and average grain size also increased with an increment in substrate temperature and sputtering power. Transmittance and band gap energy observed in the wavelength range of 350–800 showed the average transmittance was in the range of 90 to 76% and 3.12–2.88 eV. The contact angle and anti-icing properties observed during the investigation demonstrated that the synthesized coatings are hydrophobic and the formation of ice was delayed when compared to uncoated substrates.

1. Introduction

Nanotechnology has proven to be an effective method with a wide range of advantages. It has been found that nanoparticles have better surface properties across several applications. Transparent conducting oxide (TCO) AZO/ZnO:Al has been utilized for decades and has been studied and used extensively [1].

One of the most promising alternatives to indium tin oxide (ITO) is zinc oxide (ZnO) since the elements are easily available and that are also safe and reliable throughout the process of designing. Pulsed laser deposition [2], atomic layer deposition [3], magnetron sputtering [4, 5], chemical vapour deposition [6], sol-gel dip coating [7] and sol-gel spin coating [8] are only some of the methods that can be used to synthesize it. Zinc oxide (ZnO) is an n-type semiconductor with a broad direct band gap (3.37 eV) in the near-UV spectral region and great chemical stability; nevertheless, its high resistivity prevents it from being widely used in thermochromic elements (TCEs). Doping with anionic or cationic is an effective way to modify the physical properties of these materials, allowing for finer control over how they perform in a variety of applications [7]. Extrinsic doping of ZnO is possible with several different dopants, including Al, In, Ga, Cu, and Cd. Conductive ZnO thin films can be prepared most efficiently by doping with Al, as reported by Lassar *et al* [9] and Dai *et al* [10].

Ice buildup on cold surfaces has the potential to cause numerous malfunctions in high voltage electrical grids, windmills, and commercial planes. Many incidents in electrical networks have occurred in recent decades as a result of ice, such as conductor galloping and tower collapses, which result in power outages. However, most old technologies, such as thermal, mechanical, or electrical procedures, have a number of drawbacks, including inefficiency, energy loss, and even equipment damage. As a result, research into effective anti-icing methods was critical. Many hydrophobic surfaces have been manufactured by researchers for prospective uses such as self-cleaning, anti-icing, anti-frosting, and anticorrosion all encouraged by the lotus effect [11].

Wettability has been shown to be a significant feature of solid surfaces, prompting an expansion in the study in recent years. The structure and chemistry of any substrate may be used to alter its wetting qualities. Depending on the application, we can vary the wettability of the surface to shift the performance from hydrophilicity to

Table 1. Sputtering conditions for thin film deposition.

Target	Aluminium doped zinc oxide
Substrate	Corning glass
Sputtering Power	170 W, 190 W, 210 W and 230 W (Temperature: 350 °C)
Substrate temperature	150 °C, 200 °C, 250 °C and 300 °C (RF power: 250 W)
Working Pressure	2.5 Pa
Deposition time	60 min

hydrophobicity. Translucent hydrophobic coatings have many industrial uses, including anti-fogging, anti-ice adhesion, anti-wetting, anti-corrosion, and moderating friction resistance [12].

The wettability of aluminium doped zinc oxide coatings is limited in the literature, hence this is an area of interest. To achieve hydrophobic/ice phobic and transparent ZnO:Al thin films, RF magnetron sputtering was used in conjunction with helium and argon as inert gases. The influence of changing the substrate temperature and power on wettability, optical and structural characteristics of synthesized coatings were explored.

2. Methods

Aluminium-doped zinc oxide thin films were deposited by RF magnetron on glass substrates. For synthesizing thin films, the distance between the substrate and Aluminium doped Zinc Oxide target was 50 mm. The mass flow controller was utilized to monitor the gas flow rate of helium and argon gases. During the deposition process, the sputtering pressure was 2.5 Pa and the deposition time was 60 min for all the samples. The substrate temperature was 350 °C and the power was varied from 170 W to 230 W, whereas during the change in substrate temperature from 150 °C to 300 °C the sputtering power was 250 W respectively. Table 1 provides information regarding the deposition conditions:

The structural characteristics of the deposited thin films were studied using x-ray Diffractometer (XRD) (Bruker D2 phaser) using Cu-K radiation of wavelength 1.54. Surface morphology and the elemental analysis were done using an energy dispersive x-ray analysis (Emitech, Evo-18). An atomic force microscope (Bruker, Multimode Nanoscope-IV) was used to examine the surface topography. UV-vis-NIR spectrophotometer (Shimadzu 3600) was used to examine the optical properties of aluminium doped zinc oxide coatings. To determine the wettability properties, a contact angle goniometer was used (Rame Hart 290).

3. Results and discussion

The structural properties of the synthesized aluminium doped zinc oxide were studied using XRD. The XRD patterns of thin films deposited at sputtering power of 170 W, 190 W, 210 W, and 230 W and the substrate temperature was 350 °C are shown in figure 1(a). The intensity of the (002) peak at 170 W is low as seen in the graph. K Patel and S Rawal (2016) developed thin films with sputtering power ranging from 60 to 180 watts. They noticed a similar pattern of (002) ZnO peak. At 60 W, there was absolutely no peak, but as the power was increased, the peak of (002) ZnO, as well as the peaks of (100) and (101) ZnO increased [12]. A similar trend of (002) ZnO peak was observed by P Misra *et al* (2017) [13]. When the power was elevated from 190 W to 210 W the peak intensity of (002) ZnO increased. The peak intensity (002) of ZnO was maximum at 230 W. The impact of the bombardment of high-energy electrons on the evolving coating rises as the intensity of sputtered atoms approaching the substrate increases, with the increase in sputtering power. These two components contribute thermal energy to the deposited atoms, allowing them to move more freely on the substrate and so improve the crystalline structure of the deposited AZO layer.

The XRD patterns of thin films deposited at substrate temperature 150 °C, 200 °C, 250 °C, and 300 °C is shown in figure 1(b). When the temperature was increased, the energy of the atoms increases, which increases mobility. Higher mobility sputtered atoms induce larger grains than lower mobility sputtered atoms because they can spread at the surface during development, which promotes grain growth and eliminates imperfections. It was observed that the rise in substrate temperature influenced the intensity of (002) peak, which also increased with the rise in temperature. The findings are consistent with previous literatures [14–17].

To understand the structural properties of the coatings average grain size was calculated using the Scherrer equation [18]:

$$D = \frac{0.94\lambda}{\Delta\omega \cos \theta_B}$$

where, θ_B is Bragg angle, $\Delta\omega$ is the peak's full width and half maximum, λ is wavelength and D is the average crystallite size. The average crystallite size determined using the Scherrer formula grew from 14.13 nm to 23.01

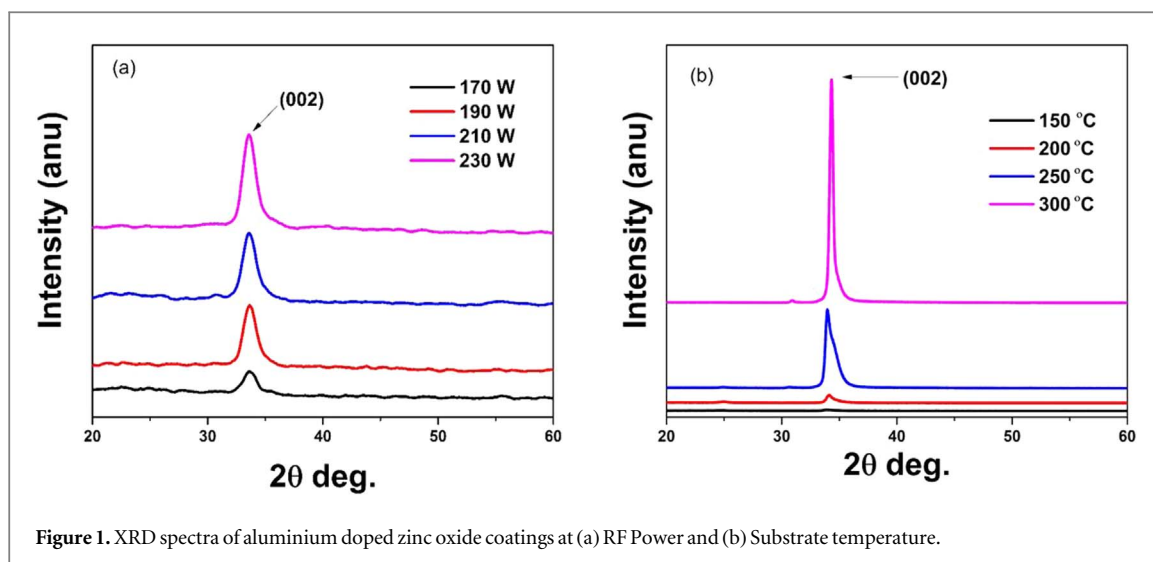


Figure 1. XRD spectra of aluminium doped zinc oxide coatings at (a) RF Power and (b) Substrate temperature.

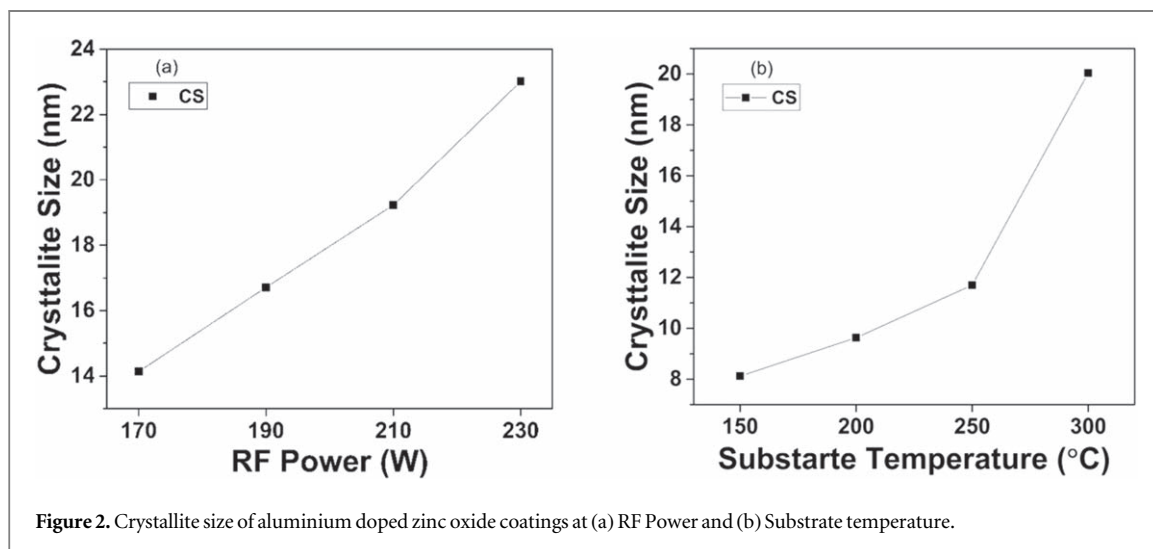


Figure 2. Crystallite size of aluminium doped zinc oxide coatings at (a) RF Power and (b) Substrate temperature.

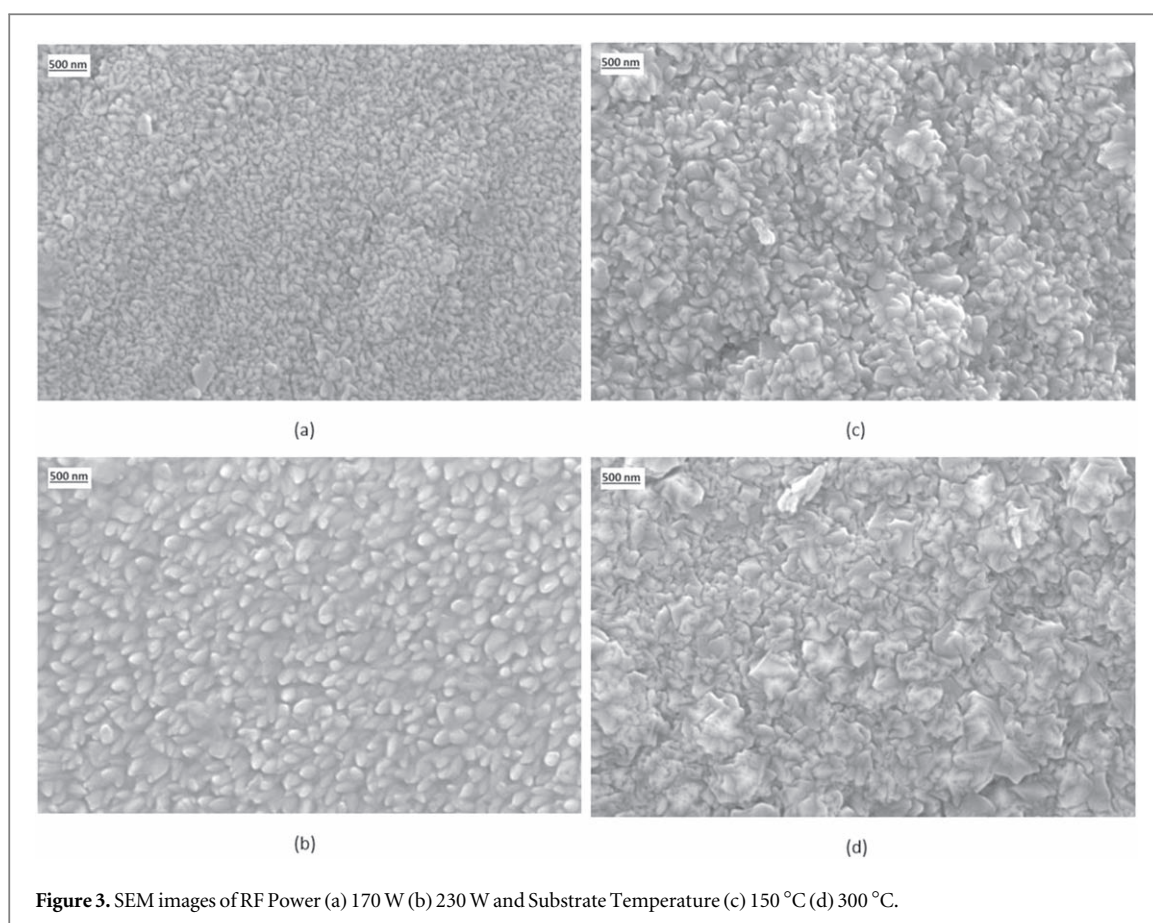
Table 2. Various observed results of deposited Al:ZnO thin films.

Sr no.	RF power (W)	Temperature (°C)	Avg. size (nm)	Thickness (nm) from %T data	O:Al:Zn composition (atomic %)
1	170	350	14.13	957	48.01:4.32:47.67
2	230	350	23.01	1126	44.16:6.5:49.34
3	250	150	8.12	1002	46.24:4.64:49.11
4	250	300	20.04	1215	43.81:3.20:52.99

nm as the power was increased from 170 W to 230 W, and from 8.12 nm to 20.04 nm as the temperature was raised from 150 °C to 300 °C. The crystallite size of the aforementioned thin films is shown in figure 2.

Table 2 displays the elemental and atomic composition of AZO films as measured by energy dispersive x-ray spectroscopy (EDAX). EDAX detected the presence of O, Al, and Zn in AZO thin films. As can be seen in figure 1, the evolution of AZO films may be attributable to an increase in metal content (Al and Zn) caused by an increase in RF power from 170 W to 230 W. A minor reduction in oxygen concentration in AZO films was observed as the temperature was raised from 150 °C to 300 °C. Common causes of XRD pattern shifts include strain and variations in chemical composition. As RF power is increased, more atoms from the AZO target are sputtered into the chamber, and most of these atoms may have high kinetic energy to cause the AZO film to evolve and grow thicker. As the temperature was increased, there was a shift in chemical composition, which was also reflected in structural properties of the deposited thin films.

Figure 3 depicts the influence of sputtering power and substrate temperature on the surface morphology of AZO thin films. Figures 3(a) and (b) demonstrate that increasing the sputtering power would increase the



particle size, which agrees well with the XRD study. If atomic kinetic energy increases, grain size would increase accordingly. Similarly, the surface morphology images at various substrate temperatures are shown in figure 3(c) and (d). It was observed that with the change in substrate temperature the surface of the thin films changes significantly. Furthermore, the grains of the AZO thin films grow rapidly and the grain boundary increases significantly. Thin films deposited at a higher substrate temperature exhibited dense morphology and larger grain size, which was also observed in the XRD analysis.

Figure 4 depicts the topography of the coated thin films at different sputtering conditions. As the sputtering power was raised from 170 W to 230 W, the average grain size of the AZO coating deposited increased in size, and the roughness of the thin films increased as well. For 170 W and 230 W, the roughness of the aforementioned films was 221 nm and 558 nm respectively. It reveals that the surface smoothness decrease at elevated sputtering power due to the bombardment impact on the surface. S. Kuo *et al* (2010) studied the impact of power on various properties of AZO thin films and noticed that the smoothness of the films was reduced from 3.15 nm to 2.06 nm with the increment of power [19]. Similarly, with the rise in substrate temperature the average grain size increased which was also reflected in AFM images. The roughness of AZO thin films were 454 nm and 634 nm at 150 °C and 300 °C respectively.

The optical properties of ZnO:Al coatings developed at varying powers and substrate temperatures were evaluated using a UV-vis-NIR spectrophotometer. The transmittance data of AZO thin films synthesized at 170 W, 190 W, 210 W, and 230 W is depicted in figure 5(a). The transmittance spectra of the films revealed that they were transparent, with transmittance values decreasing from 90% to 76% for thin films deposited at 170 W to 230 W, respectively. Y Xia *et al* (2016) utilized DC magnetron sputtering to deposit AZO ultra-thin films at varied power levels ranging from 50 W to 125 W and observed that with the rise of power level, the optical transparency dropped due to crystal quality degradation [20]. A Spadoni and M Addonizio (2015) reported similar decrease in transmittance as the power was increased from 600 W to 1200 W. They reported that due to free carrier absorption, which increases as the carrier concentration grows, the transmittance in the NIR wavelength region falls as discharge power was increased [21]. A similar decrease in values with increasing power have been reported in the literature [12].

The transmittance values of aluminium doped zinc oxide coatings developed at varying substrate temperature is shown in figure 5(b). It was observed that transmittance was slightly decreased from 84% to 79% with an increase in temperature from 150 °C to 300 °C respectively. S Yang *et al* (2019) studied the influence of

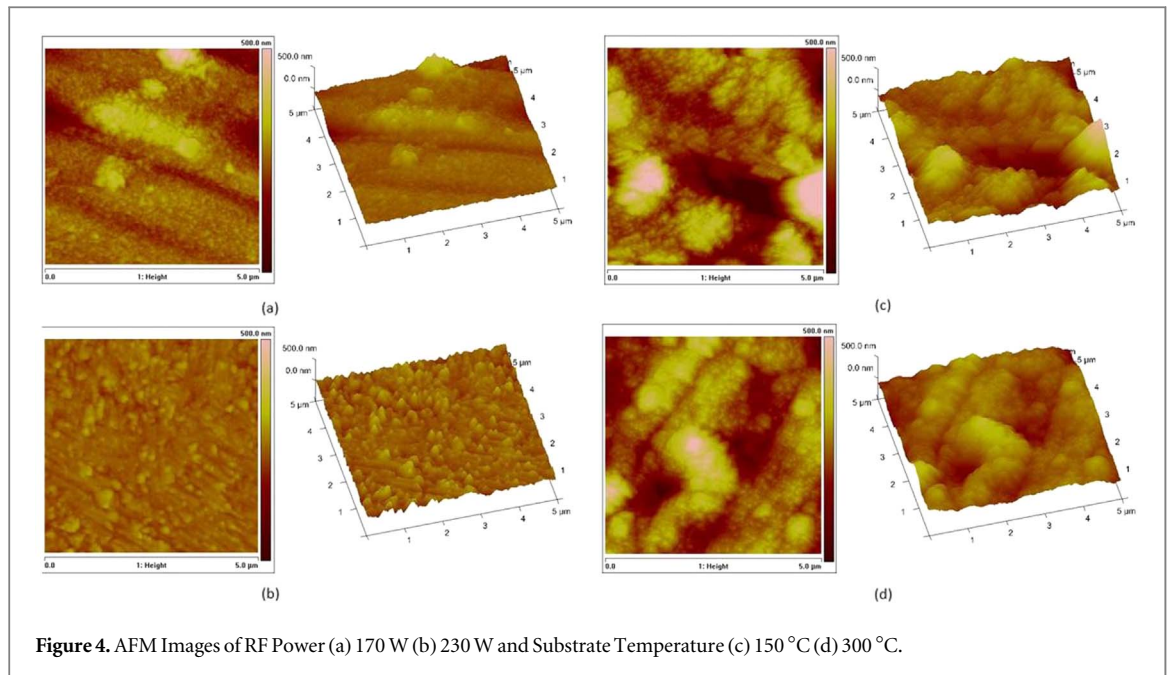


Figure 4. AFM Images of RF Power (a) 170 W (b) 230 W and Substrate Temperature (c) 150 °C (d) 300 °C.

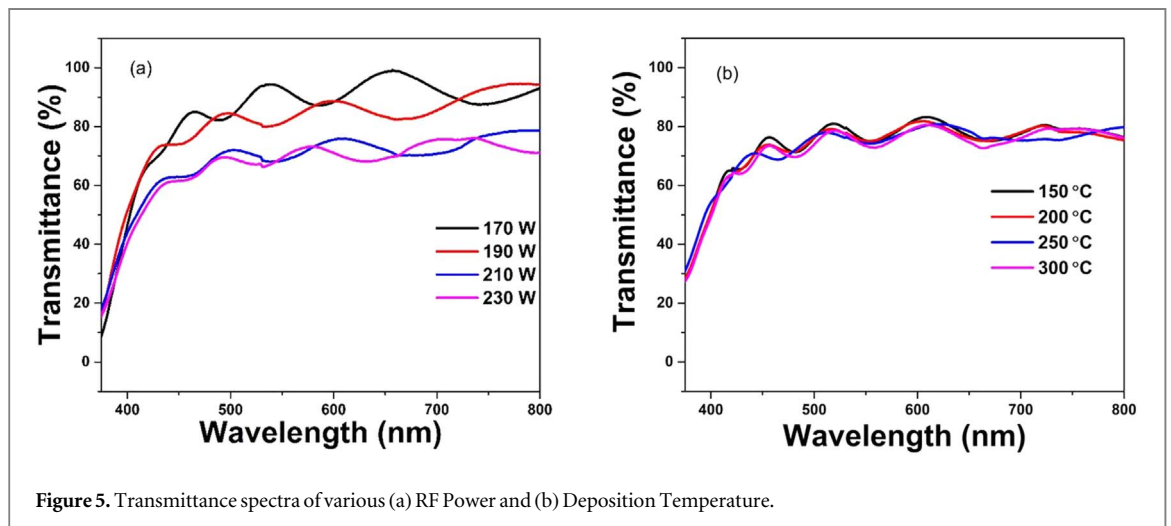


Figure 5. Transmittance spectra of various (a) RF Power and (b) Deposition Temperature.

various deposition temperatures from 100 °C to 400 °C. Sharp absorption was noticed in 300 to 350 wavelength region and the transmittance of the thin films decreased from 91.3% to 88.4% for films synthesis at 100 °C to 400 °C respectively [22]. K Patel and S Rawal (2016) also observed that with the increase in temperature the transmittance decreased [12]. The transmittance of the coatings is affected by the size of the grains and film thickness. Due to higher crystallite size along with the increased surface roughness, the electrons will scatter which will affect the optical transmittance at elevated temperatures and power.

The relation used to calculate the refractive index of deposited aluminium doped zinc oxide was [23]:

$$\text{Refractive index } n = [N + (N^2 - n_0^2 n_1^2)^{1/2}]^{1/2}$$

where

$$N = \frac{n_0^2 + n_1^2}{2} + 2n_0 n_1 \frac{T_{\max} - T_{\min}}{T_{\max} T_{\min}}$$

n_1 and n_0 represents substrate and air's refractive index.

The refractive index of ZnO:Al coatings developed at 170 W to 230 W was in the range of 1.50 to 1.51, while the refractive index of developed coatings at 150 °C to 300 °C was between 1.51 to 1.53 respectively. K. Patel and S Rawal (2016) and P Misra *et al* (2017) noticed that AZO thin films had constant refractive index. The thickness of the deposited coatings was calculated using the below relation [24]:

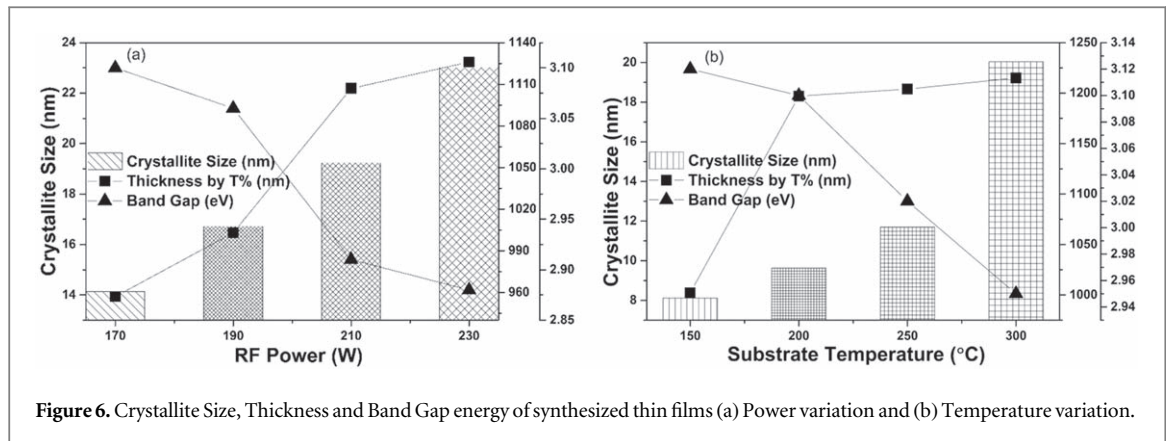


Figure 6. Crystallite Size, Thickness and Band Gap energy of synthesized thin films (a) Power variation and (b) Temperature variation.

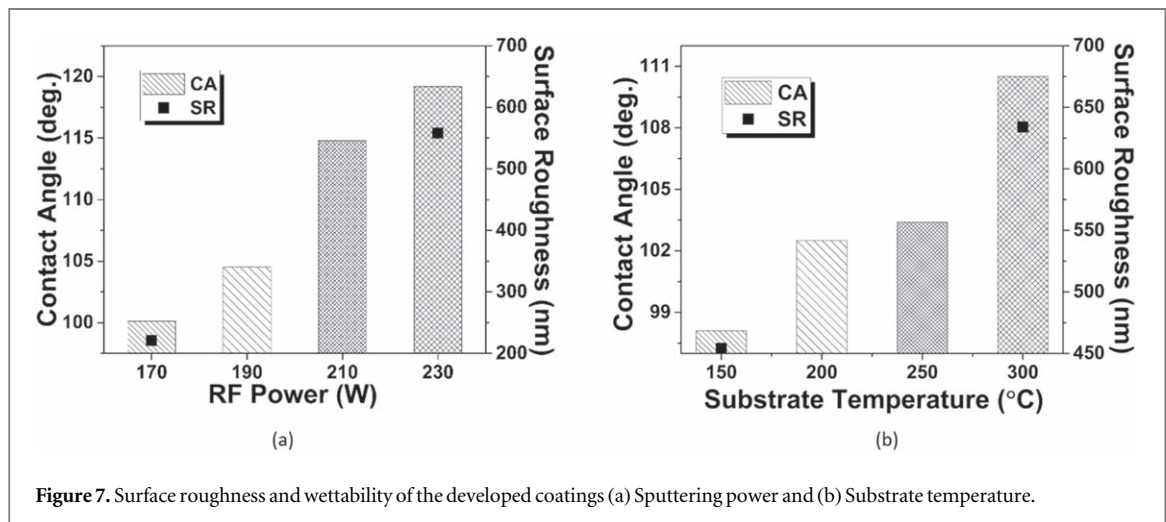


Figure 7. Surface roughness and wettability of the developed coatings (a) Sputtering power and (b) Substrate temperature.

$$t = \frac{M\lambda_1\lambda_2}{2[n(\lambda_1)\lambda_2 - n(\lambda_2)\lambda_1]}$$

where λ_1 , $n(\lambda_1)$, λ_2 and $n(\lambda_2)$ are corresponding wavelength and index of refraction and M is number of oscillations.

The increase in RF power from 170 W to 230 W resulted in increase in film thickness, as assessed by transmittance spectra using the above discussed equation. The thin films deposited at 170 W, 190 W, 210 W, and 230 W had thicknesses of 957, 1003, 1107, and 1126 nm, respectively. Thin films deposited at 150 °C, 200 °C, 250 °C, and 300 °C substrate temperatures had thicknesses of 1002, 1197, 1204, and 1215 nm, respectively.

Band gap energy E_g of the developed thin films are related to absorption coefficient α and was calculated using Tauc relation [25].

$$\alpha = A(h\nu - E_g)^n/h\nu$$

where, A is a constant, $h\nu$ is photon energy and n is the transition index.

The band gap energy and the calculated thickness of the deposited thin films is shown in figure 6. The above relation was used to calculate the band gap, and E_g was found to be in the range of 3.10 to 2.88 eV for RF power 170 W to 230 W, and 3.12 to 2.95 eV for substrate temperatures 150 °C to 300 °C. The findings are consistent with previously published literature on ZnO:Al thin films [26, 27].

The surface roughness and wettability properties of developed ZnO:Al coatings at varying power and substrate temperatures are depicted in figure 7. The hydrophobicity of the developed thin films increased at elevated power and substrate temperature. This was evident from the roughness of the thin films. Surfaces with a balance of micro textures and nano textures are often needed to generate superior hydrophobic surfaces. The contact angle decreases as the surface becomes smoother, and increases as the presence of micro textures or nano textures within the surface increases. To understand the wetting phenomena on rough surfaces, two models were proposed by Cassie-Baxter and Wenzel. The relation between roughness and contact angle is understood by Wenzel's equation [28]:

Table 3. Ice formation delay and water contact angle of thin films deposited at various sputtering conditions.

Sputtering Condition	170W	190W	210W	230W	150 °C	200 °C	250 °C	300 °C
WCA (°)	100.1	104.5	114.8	119.2	98.1	102.5	103.4	110.5
Time Delay (times/folds)	2.5 x	2.83 x	4.18 x	4.29 x	2.5 x	2.58 x	2.75 x	3.58 x

$$\cos \theta_w = A \cos \theta$$

where θ_w denotes WCA with rough surface, θ indicates WCA on the basis of interfacial energy at the point of contact between three phases and A signifies the roughness factor which can be defined by the ratio of actual and apparent surface areas which is greater than 1. So if $\theta_w < \theta$ the surface is hydrophilic and has higher wettability and if $\theta_w > \theta$ the wettability is decreased and the surface is hydrophobic [29].

A maximum water contact angle of 119.2° was observed for ZnO:Al thin films developed at 230 W. The contact angles of the synthesized thin films at 210, 190, and 170 W were 114.8°, 104.5°, and 100.1°, respectively. With increasing RF power, the contact angle and roughness increased. Similarly, the contact angle of the AZO thin films declined from 110.5° to 98.1° as the substrate temperature decreased from 300 to 150 °C. Surface roughness had a beneficial impact on the contact angle, which was in line with the literature reported [30–32]. Additionally, changes in temperature and RF power of deposited thin films are correlated with changes in the chemical composition of the coatings. The development of AZO films is subject to the rate at which sputtered atoms can be retrieved within the sputtering chamber. When the RF power and the temperature are elevated, the atmosphere inside the chamber may change. As can be seen from EDAX data, the chemical composition of the AZO films changes depending on the availability of sputtered atoms, which in turn affects the formation kinetics of the films. In both situations, the rise in hydrophobicity and contact angle of AZO films may have been caused by the changes in surface roughness and chemical composition.

The icing characteristics of thin films produced under various sputtering conditions were investigated using a Peltier cooling setup where the temperature was –15 °C. The icing characteristics of thin films were compared to uncoated samples, revealing that ice formation on coated samples was delayed. Table 3 demonstrates the increase in icing time delay on thin films deposited at various RF power and sputtering temperature.

The AZO thin films synthesized at 230 W RF power had the maximum delay in ice formation, which was 4.29 times higher than the uncoated samples. The development of ice was observed to be delayed when the hydrophobicity of the thin films surged from 100.1° to 119.2° for change in RF power. Similarly, for AZO thin films deposited at varying substrate temperatures, a comparable delay in ice formation was observed. When compared to an uncoated substrate, thin films deposited at 300 °C took 3.58 times longer to develop ice on the surface. Recent scientific research suggests a direct link between ice and water repellency [33]. One of the two key components in improving hydrophobic and anti-icing characteristics is using materials with low surface energy, and another essential component in increasing the water contact angle on surfaces, which gradually increases the anti-icing characteristics of the thin films is surface roughness.

S Noormohammed and D Sarkar (2021) studied the influence of hydrophobic coatings on the adhesion and formation of ice. They suggested that superhydrophobic surfaces regulated by chemistry (low surface energy) and geometry (textured) are acceptable options for icephobic surfaces. They also observed that with an increase in hydrophobicity, the adhesion strength of ice decreases. It was also reported by other researchers that it is necessary to achieve surface roughness and low surface energy to obtain a hydrophobic and in fact, an icephobic surface [34, 35]. G Liu *et al* (2020) investigated the impact of time on the wettability of zinc oxide thin films developed on an aluminium substrate. The deposited thin films were hydrophobic, and frosting and icing were delayed on the substrates that were coated with ZnO thin films. In addition, the ice adhesion strength of coated substrates was lower than that of bare substrates [11].

4. Conclusion

Various sputtering parameters were used to deposit ZnO:Al/AZO thin films. It was observed from XRD patterns that the intensity of the peak (002) and average crystallite size increased as the RF power and substrate temperature were elevated. As the sputtering power and temperature of aluminium doped zinc oxide films are increased, the refractive index increased whereas the optical transmittance and band gap energy reduces. The thickness measured using transmittance data shows the thickness was 957 to 1126 nm for sputtering power 170 W to 230 W and 1002 to 1215 nm for thin films developed at 150 °C to 300 °C substrate temperatures respectively. ZnO:Al thin films developed at 230 W had a maximum contact angle of 119.2° and the ice formation was delayed by 4.29 folds when compared to untreated substrates.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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