



## Synthesis and studies of sputter deposited ZnO films

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### Abstract

*This paper is aimed to synthesize reactive sputtered ZnO coatings at altered RF powers of 50W, 75W, 100W and 125W and study it's, optical structural and wettability properties. For characterization of ZnO coatings, X-ray Diffractometer (XRD) technique was used. XRD graphs indicate evolution of (002) peak and (100) peak for ZnO thin films with an increment of RF power from 50W to 125W. ZnO thin films become well crystalline with rising the RF power and preferred orientation of ZnO along (002) texture is observed. Surface energy and contact angle of ZnO films were determined by contact angle goniometer. UV-Vis-NIR spectrophotometer was utilized to differentiate optical properties of zinc oxide films.*

**Keywords:** ZnO, Sputtering, XRD, Wettability, Contact Angle.

### Introduction

Wettability can be defined by the contact angle formed amongst a solid surface and liquid. The ability to control contact angle is important for many chemical, electronic, and biological applications<sup>1,2</sup>. Wettability property is also affected by chemical content of surface as well as its topographical features<sup>3-7</sup>. An inspiring self-cleaning phenomenon is observed in nature referred as lotus leaf effect<sup>8</sup>. In the last decade research is done with interest and specific focus on controlling wettability of the surface that is affected by surface roughness as well as its surface energy<sup>9</sup>.

As their electronic and optical properties are excellent they have several potential applications in nano scale sensors, UV laser diodes, and transistors, crystalline nanostructures of ZnO are gaining peculiar interest among the semiconductor oxides<sup>10,11</sup>. ZnO is widely investigated as a conductive and nano-structured material due to its usual properties like a wide band gap (3.3eV) and transmittance in visible span and high-electrochemical stability<sup>12,13</sup> which crystallize as wurtzite hexagonal structure<sup>14</sup>.

The nano-structured ZnO thin film was deposited as nano needles, nano nails, nano clusters, nano rod arrays, etc. Lately, wettability properties of nano structured ZnO thin film has found a lot of interest in exploring its good perspectives as an industrial application<sup>15,16</sup>. Super hydrophobic surface is becoming popular amongst industrialists and academician because of its exceptional properties like self-cleaning, de-icing, anti-sticking, and anti-contamination<sup>17</sup>. Nowadays, zinc oxide thin films are explored for its applications in sensors and transducers<sup>18</sup>. It has excellent piezoelectric properties and electromechanical coupling coefficient, they have been used to fabricate surface acoustic wave devices used for communication<sup>19</sup>.

In this present paper, zinc oxide nanocrystalline thin films are synthesized at various values of RF power by radio frequency (RF) reactive sputtering technique. ZnO films of different thickness were developed at different RF power values and its effect on its various properties are reported in this paper.

### Materials and methods

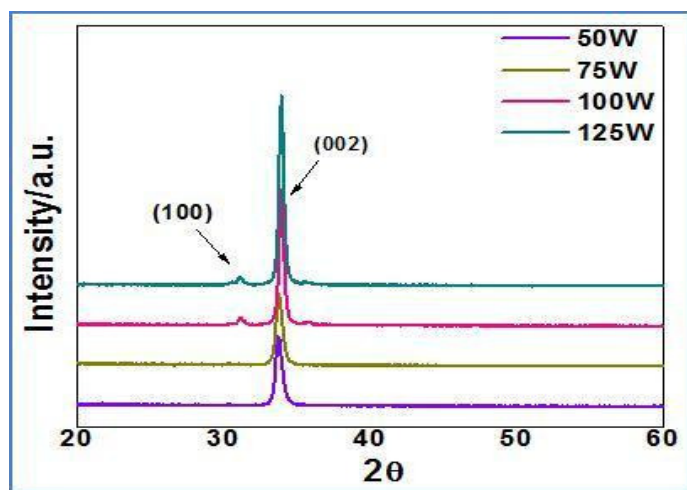
Nanocrystalline ZnO coatings were reactively sputtered in cylindrical compartment (Excel Instruments, India). The target of zinc had 50.8mm diameter and 99.99% purity which was used for sputtering process. Argon was used as inert gas and oxygen was used as a reactive gas to develop ZnO films. The flow of gases was controlled by MFC (ALICAT instruments, USA). Distance between substrate and target was fixed at 50mm for all cases, gas pressure was also constant at 1.0Pa. For different RF power of 50W, 75W, 100W and 125W, the sputtering was done at fixed deposition time of 60minutes and deposition temperature of 500°C.

Zinc oxide thin films were examined for structural properties by XRD (Bruker D2 phaser, advance diffractometer). The contact angle goniometer (Rame-Hart model 290) was utilized to determine contact angle between water and ZnO films. For measuring the optical absorption and transmission of ZnO films surface, UV-Vis-NIR spectrophotometer (Shimadzu, model UV3600 plus) was used.

### Results and discussion

XRD pattern for ZnO coatings developed at different RF powers of 50W, 75W, 100W, and 125W respectively is shown in Figure-1. XRD pattern shows (002) and (100) diffraction peaks of ZnO coatings. The intensity for (002) peak rises with an increase in power from 50W to 125W. Similarly, the evolution

of weakly crystalline (100) peak is observed at RF power values of 100W and 125 W.S. Flickyngerova *et al.*<sup>20</sup> had prepared ZnO coatings at altered RF power and voltage. They found very high intense ZnO (002) peak with rise in power, the intensity of (100) peak decreases with voltage bias. They concluded that with less negative bias voltage along with high RF power, ZnO thin film gets wurtzite structure having (002) and (100) peaks with good crystalline structure. They had also found that upsurge in RF power reveals a rise in natural intensity for its XRD peak and improves crystallinity of films. R. Ondo-Ndong *et al.*<sup>21</sup> had formed ZnO thin film on different substrates by RF sputtering process to study deposition rate at different temperature and a different rate of distance between substrate and target material. They observed (002) peak with high intensity, good homogeneity, and high crystallinity. The intensity of (002) peak increased as the substrate temperature was increased and found improvement in the films crystallinity. So the evolution of (100) and (002) textures of ZnO coatings developed at altered RF power are consistent with the above cited literature.

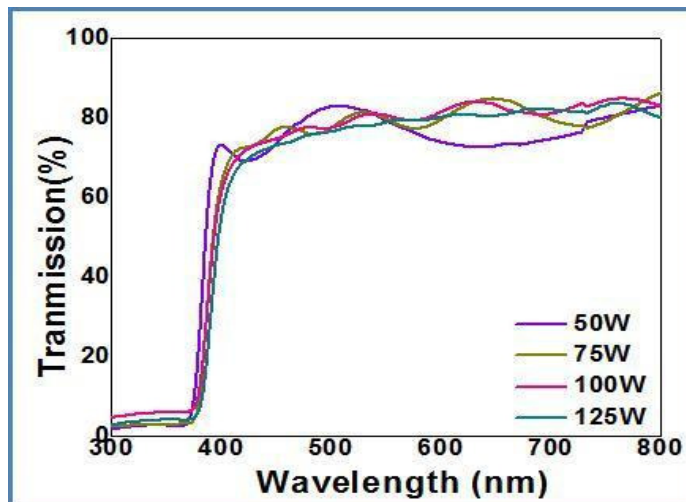


**Figure-1:** XRD graphs of ZnO coatings developed at diverse RF power.

In Table-1, the calculation of average grain size ( $d$ ) of ZnO coatings found from Scherrer formula<sup>22</sup> is mentioned. The average grain size of ZnO coatings increase from 18 to 25nm with increasing power from 50W to 125W. The (002) diffraction peak of ZnO coatings becomes narrow with high intensity due to rise in power which results in larger grain size.

**Table-1:** The computed values for ZnO coatings.

RF Power (in Watt)	Name of Sample	Avg. d(XRD) (nm)	Refractive Index (n)	Thickness from %T data
50	50W	18	1.60	779
75	75W	20	1.62	854
100	100W	24	1.65	1078
125	125W	25	1.70	1260



**Figure-2:** Transmittance of ZnO coatings developed at diverse RF power.

Figure-2 shows the spectral transmittance of zinc oxide thin films developed at several RF power. Thickness of zinc oxide coatings was computed from the transmittance as reported in literature<sup>23</sup>. Thickness of zinc oxide coatings developed at diverse RF power varies between 779nm to 1260nm as given in Table-1. The transmission spectra were utilized to determine reflective index of ZnO films by formula suggested by J.C. Manificier *et al.*<sup>24</sup>. The calculated refractive index is given in Table-1. Refractive indices of ZnO thin films is in between 1 to 2, subjected to thickness of coatings and its structure<sup>25</sup>. Refractive index of deposited zinc oxide thin films increases from 1.60 to 1.70 with the rise in power and thickness.

Figure-3 shows the surface energy and contact angle measurements of ZnO coatings. The contact angle of ZnO coatings increases while its surface energy decreases with rising in the power. When power is 50W, contact angle noted is 94.4°. The maximum contact angle for ZnO films is observed at RF power of 125W which is 103.2°. A reduction in surface energy is observed at RF power of 75W. At RF power of 100W and at 125W, the (100) intensity peak is observed in deposited zinc oxide thin film whereas (002) peak is found in all samples. The intensity of (002) peak increases with RF power, giving larger grain size, thicker films resulting in lower surface energy and higher contact angle.

AFM images of zinc oxide coatings developed at 50W Power and 125W RF power are represented by Figure-4. The average grain size of zinc oxide films surges with the rise in power from 50W to 125W as evident from Figure-4 and confirms the results of XRD graphs. J. Kim *et al.*<sup>26</sup> developed ZnO coatings and achieved hydrophobic surface by doing a surface modification of FAS on ZnO thin films. They examined that with rising in power, roughness of zinc oxide films increases. They found ZnO thin films with rod diameters of 50-150 nm showed a relatively high RMS roughness. A. Ismail and M.J. Abdullah<sup>27</sup>

primed zinc oxide films on silicon at diverse RF power. They found 14.38nm surface roughness at 250W of RF power and concluded that with a rise in ZnO coating thickness its surface roughness also increases. W.J. Khudhayer *et al.*<sup>28</sup> noted that with rise in ZnO coating thickness, the surface roughness is also increased and with that contact angle also increases. So the roughness of surface and contact angle exhibit a direct relationship.

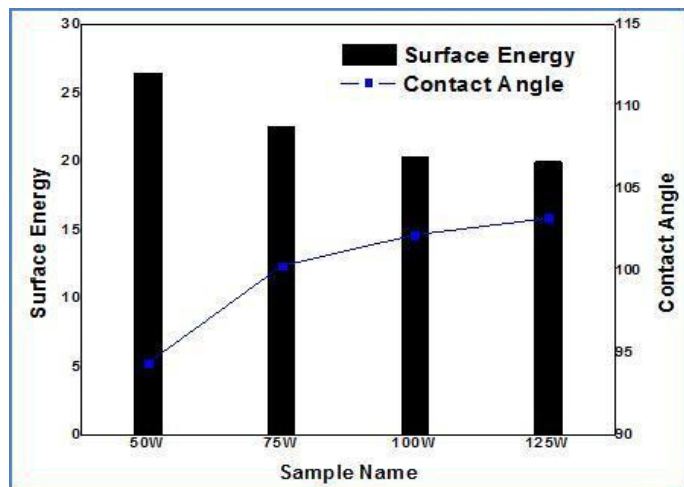


Figure-3: Contact angle and surface energy of ZnO coatings developed at diverse RF power.

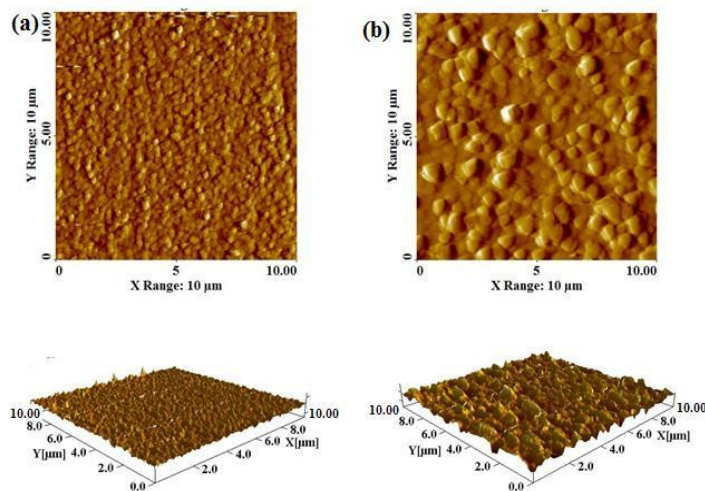


Figure-4: AFM images of ZnO coatings developed at RF power of (a) 50W and (b) 125W.

The relation between contact angle and the surface roughness is represented by Figure-5. Contact angle rises as surface roughness increases which is consistent with above-cited literature. At low RF power of 50W, surface roughness and thickness of zinc oxide films is lower. With the rise in power, thickness of ZnO films increases and with the rise in thickness, surface roughness is increased thereby imparting higher contact angle values. The lowest surface roughness about 13nm is found at 50W RF power. The highest contact angle is found 103.2° at surface roughness value of 28nm at 50W RF power.

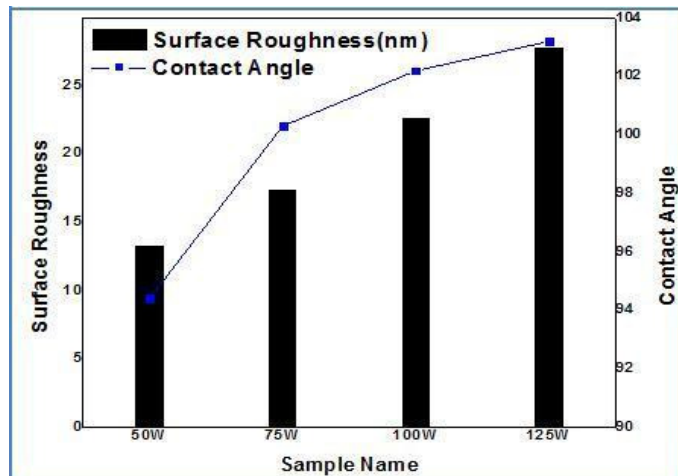


Figure-5: Contact angle and surface roughness of ZnO coatings developed at diverse RF power.

### Conclusion

ZnO thin films have a very intense (002) peak and (100) peak is observed at higher Rf power values of 100W and 125W. The intensity of (002) peak increases with rise in the power from 50W to 125W. Average grain size of ZnO coatings rises from 18nm to 25nm with increasing the RF power from 50W to 125W. Thickness of ZnO coatings rises from 779nm to 1260nm with rise in RF power. The ZnO thin films surface roughness rises from 13nm to 28nm as a consequence contact angle rises from 94.4° to 103.2° which shows the hydrophobic nature of deposited ZnO thin films.

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### References

- Gau H., Herminghaus S., Lenz P. and Lipowsky R. (1999). Liquid Morphologies on Structured Surfaces: From Microchannels to Microchips. *Science*, 283(5398), 46-49.
- Feng X. and Jiang L. (2006). Design and creation of superwetting/antiwetting surfaces. *Adv. Mater.*, 18(23), 3063-3078.
- Lu J., Huang K., Chen X., Zhu J., Meng F. and Sun Z. (2010). Reversible wettability of nanostructured ZnO thin films by sol-gel method. *Applied Surface Science*, 256(14), 4720-4723.
- Abbott N.L., Folkers J.P., Whitesides G.M. and Drive N.F. (1992). Manipulation of the Wettability of surfaces on the 0.1 to 1 micrometer scale through micromachining and molecular self-assembly. Department of chemistry, Harvard University, Cambridge.

5. Li M., Zhai J., Liu H., Song Y., Jiang L. and Zhu D. (2003). Electrochemical Deposition of Conductive Superhydrophobic Zinc Oxide Thin Films. *J.Phys. Chem. B*, 107(37), 9954-9957.
6. Abbott S., Ralston J., Reynolds G. and Hayes R. (1999). Reversible wettability of photoresponsive pyrimidine-coated surfaces. *Langmuir*, 15(26), 8923-8928.
7. Chen W., Fadeev A.Y., Hsieh M.C., Youngblood J., Mccarthy T.J., Chen W., Fadeev A.Y., Hsieh M.C., Didem O., Youngblood J. and Mccarthy T.J. (1999). Ultrahydrophobic and Ultralyophobic Surfaces: Some Comments and Examples. *Langmuir*, 15(10), 3395-3399.
8. Mao-Gang G., Xiao-Liang X., Zhou Y., Yan-Song L. and Ling L. (2010). Superhydrophobic surfaces via controlling the morphology of ZnO micro/nano complex structure. *Chinese Physics B*, 19(5), 1-6.
9. Subedi D., Madhup D., Sharma A., Joshi U. and Huczko A. (2012). Study of the wettability of ZnO nanofilms., *Int. Nano Lett.*, 1(2), 117-122.
10. Yin L.W., Sen Li M., Bando Y., Golberg D., Yuan X. and Sekiguchi T. (2007). Tailoring the optical properties of epitaxially grown biaxial ZnO/Ge, and coaxial ZnO/Ge/ZnO and Ge/ZnO/Ge heterostructures. *Adv. Funct. Mater.*, 17(2), 270-276.
11. Richters J.P., Dev A., Muller S., Niepelt R., Borschel C. and Voss T. (2009). Influence of metallic coatings on the photoluminescence properties of ZnO nanowires. *physica status solidi (RRL)-Rapid Research Letters*, 3(5), 166-168.
12. Huang M.H., Wu Y., Feick H., Weber E. and Yang P. (2001). Catalytic Growth of Zinc Oxide Nanowires by Vapor Transport. *Advanced Materials*, 13(2), 113-116.
13. Yang Z.X., Zhu F., Zhou W.M. and Zhang Y.F. (2005). Novel nanostructures of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> synthesized by thermal evaporation. *Phys. E Low-Dimensional Syst. Nanostructures*, 30(1), 93-95.
14. Wu J.J., Wu J.J., Liu S.C. and Liu S.C. (2012). Low-temperature growth of well-aligned ZnO nanorods by chemical vapor deposition. *Advanced materials*, 21(5), 1-3.
15. Feng X., Feng L., Jin M., Zhai J., Jiang L. and Zhu D. (2004). Reversible super-hydrophobicity to super-hydrophilicity transition of aligned ZnO nanorod films., *J. Am. Chem. Soc.*, 126(1), 62-63.
16. Pan Z.W., Dai Z.R. and Wang Z.L. (2001). Nanobelts of Semiconducting Oxides. *Science*, 291(5510), 1947-1949.
17. Srivastava M., Basu B.B.J. and Rajam K.S. (2011). Improving the hydrophobicity of ZnO by PTFE incorporation. *J. Nanotechnol*, 1-6. doi:10.1155/2011/392754
18. Pearton S.J., Norton D.P., K. Ip, Heo Y.W. and Steiner T. (2004). Recent advances in processing of ZnO. *AVS*, 22(3), 932-948.
19. Wenzel S.W. and White R.M. (1988). A Multisensor Employing an Ultrasonic Lamb-Wave Oscillator. *IEEE Trans. Electron Devices*, 35(6), 735-743.
20. Flickyngerova S., Shtereva K., Stenova V., Hasko D., Novotny I., Tvarozek V., Sutta P. and Vavrinsky E. (2008). Structural and optical properties of sputtered ZnO thin films. *Appl. Surf. Sci.*, 254(12), 3643-3647.
21. Ondo-Ndong R., Pascal-Delannoy F., Boyer A., Giani A. and Foucaran A. (2003). Structural properties of zinc oxide thin films prepared by R.F. magnetron sputtering. *Mater. Sci. Eng. B*, 97(1), 68-73.
22. Rosa A.M., da Silva E.P., Amorim E., Chaves M., Catto A.C., Lisboa-Filho P.N. and Bortoleto J.R.R. (2012). Growth evolution of ZnO thin films deposited by RF magnetron sputtering. *J. Phys. Conf. Ser.*, 370(1), 012020.
23. Wu K.R., Wang J.J., Liu W.C., Chen Z.S. and Wu J.K. (2006). Deposition of graded TiO<sub>2</sub> films featured both hydrophobic and photo-induced hydrophilic properties., *Appl. Surf. Sci.*, 252(16), 5829-5838. doi:10.1088/1742-6596/370/1/012020
24. Manificier J.C., Gasiot J. and Fillard J.P. (1976). Simple Method for the Determination of the Optical Constants. *Journal of Physics E: Scientific Instruments*, 9(11), 1002-1004.
25. Ekem N., Korkmaz S., Pat S., Balbag M.Z., Cetin E.N. and Ozmumcu M. (2009). Some physical properties of ZnO thin films prepared by RF Magnetron sputtering techniques. *Int. J. Hydrogen Energy*, 34(12), 5218-5222.
26. Kim J.H., Lee M., Lim T.Y., Hwang J.H., Kim E. and Kim S.H. (2010). Fabrication of transparent superhydrophobic ZnO thin films by a wet process. *J. Ceram. Process. Res.*, 11(2), 259-262.
27. Ismail and Abdullah M.J. (2013). The structural and optical properties of ZnO thin films prepared at different RF sputtering power. *J. King Saud Univ.-Sci.*, 25(3), 209-215.
28. Khudhayer W.J., Sharma R. and Karabacak T. (2009). Hydrophobic metallic nanorods with Teflon nanopatches. *Nanotechnology*, 20(27), 275302.