

# Wettability assessment of Zirconium oxide by Radio Frequency Magnetron sputtering

Nikhil Soni<sup>1</sup>, Sujal Patel<sup>2</sup>, Krutarth patel<sup>3</sup>, Dharmesh Chauhan<sup>4</sup>, Divyeshkumar Dave<sup>5</sup>  
1,2,3,4,5(Mechanical department, Sardar Patel College of Engineering(SPCE), Vadtal road, Bakrol-388315, Gujarat, India.)

## Abstract:

The aim of this study is to identify the formation of zirconium oxide films by reactive sputtering of chromium target using oxygen and nitrogen as reactive gases along with helium that was used as an inert gas. Zirconium oxide thin films were deposited on glass and silicon substrates by using RF magnetron sputtering process and influence of working pressure was investigated. The structural properties of thin film were characterized by using X-ray diffractometer (XRD). Surface energy and contact angle were explored by using contact angle measuring system. Surface energy and contact angle of thin films increased with increasing pressure from 1.5 to 4.5 Pa and revealed transformation from hydrophilic to hydrophobic behavior of zirconium oxide thin films.

*Keywords* — Magnetron sputtering, Zirconium oxide, Wettability.

## I. INTRODUCTION

Zirconium oxide or Zirconia material characterized by large resistance against oxidation, high refractive index, good ionic conductivity, thermal stability and high melting point in Y-cubic phase [1]. Furthermore, ZrO<sub>2</sub> represent structures and properties of thin films, but it is depending upon the precise deposition and deposition process [2]. Films of zirconium oxide utilized for absolute application such as protective coating, abrasive coating, resisting element sensor [3,4] and detectors, and high heat barrier coating [5,6]. Zirconium films prepared by plasma technique because method is common in work condition [7].

ZrO<sub>2</sub> films has Prefer to control on film composition, minimized target positioning and deposition rate by reactive magnetron sputtering process. Moreover, the thin film generated as crystalline form, so it always easy to used polycrystalline application: microelectronic devices and optical [3].The sputtering coating applied an increasing the efficiency and life of tools used in machine such as cutting, moulding and drilling [8]. The PVD (Physical vapor deposition) technique is widely used for hard coating [9,1].

In the present work, we report the ZrO<sub>2</sub> thin films by RF magnetron sputtering and study the wettability properties of ZrO<sub>2</sub> thin films. The zirconium oxide thin films examine by XRD techniques.

## II. EXPERIMENT DETAILS

The zirconium oxide thin film was deposited on a substrate called Glass & Silicon by radio frequency(RF) magnetron sputtering process. The Zr target diameter 2” and thickness 5mm was used for film deposition by the PVD setup manufactured by excel instruments (INDIA). The substrate to target distance kept constant (50mm). Before deposition the target was sputtered cleaned in argon atmosphere for a period of time. Further, the sputter chamber was pumped down to  $5 \times 10^{-4}$  pa by a molecular turbo pump for reducing epidemic. Then, the oxygen (purity 99.99%) & argon gas at 4sccm and 20sccm respectively were introduced in a chamber. The mass flow controllers were used to controlled the gas ratio. The deposition parameter was such as RF power of 150w, base pressure  $4 \times 10^{-4}$  Pa, deposition time upto 1hour and deposition temperature was 400°C. The structural characterization of the zirconium oxide film was carried out by X-ray diffractometer (XRD). The study of films on the property wettability was made

by finding the contact angle of water with the films and surface energy.

### III. RESULT AND DISCUSSION

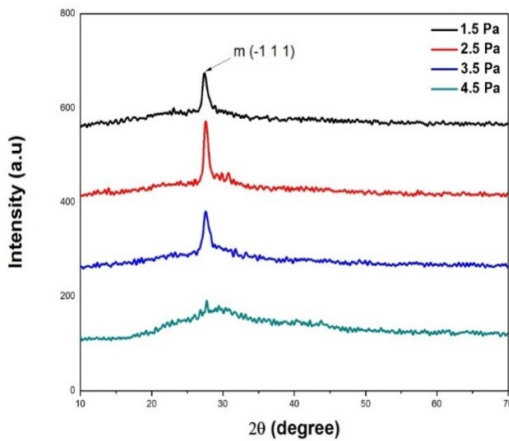


Fig. 1 XRD Patterns of ZrO<sub>2</sub> coatings

The microstructure of the films was investigated by X-ray diffractometer is shown in fig.1, which depicts the coating is uniformly distributed or not and also illustrate at which degree we can find acme intensity. The peak point is shown in fig. 1 at 28° and the plane (-1 1 1) is in monoclinic phase. The crystalline size is calculated from full width at half maximum (FWHM) of the XRD peaks using Scherrer's formula, [7,10,11,13]

$$D = \frac{k\lambda}{\beta \cos \theta}$$

Where, D is the crystallite size, K is the Scherrer constant (0.9), λ is the wavelength of the X-rays used (1.54Å), β is the line broadening at half the maximum intensity (FWHM) (in radians) after subtracting the instrumental line broadening, and θ is the Bragg angle (indegrees).

The mean crystallite sizes are evaluated for monoclinic using the (-111) reflection and found to be in the range of 10.56-28 nm at working pressure range of 1.5 to 2.5 Pa. Crystallite sizes grow from 10.56 to 12 nm as pressure increases from 2.5 to 3.5 Pa. No significant variations are found, as pressure increases from 3.5 Pa to 4.5 Pa. A further increase in pressure from 3.5 Pa to 4.5 Pa leads to increase crystallite size from 28 nm. The applied pressure encourages surface diffusion of the arriving species

which result increase in crystallite quality and crystallite size.

### IV. WETTABILITY

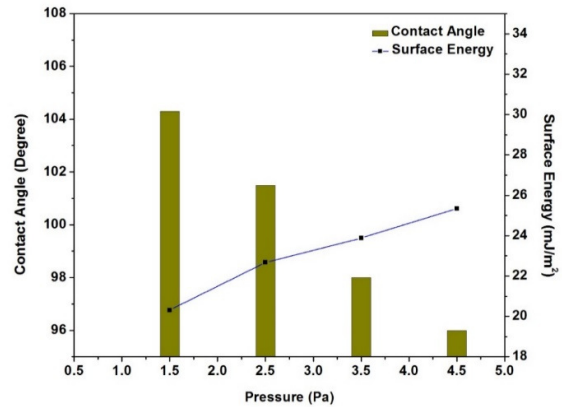


Fig. 2 Variation of surface energy and contact angle values associated with the pressure

As shown in fig.2 the bar graph depicts proportion of the pressure in Pascal, contact angle in degree and the surface energy in (MJ/m<sup>2</sup>). As we can see when pressure increase from 1.5 to 2.5 Pa, there is a small decline in contact angle 104.3° to 101.5° similarly, in pressure 3.5 and 4.5 contact angle is 98.2° and 96° respectively. Hence for all different value of pressure the film shows the contact angle value more than 90° which, shows its property of hydrophobic.

Surface metallurgy science generally contains an instrument known as “contact angle goniometer”. This goniometer is mainly used to measure contact angles, advancing and receding contact angles and also for surface tension. At early stage the goniometer is used manually, which use an eyepiece with a microscope while in modern contact angle goniometer there is a camera and software which is use for capture and analyse the drop shape. It is also better for dynamic and advanced studies.

Furthermore, when the contact angle is 104.3° the surface energy is around 20.3 MJ/m<sup>2</sup> and the peak point of surface energy is 25.3 MJ/m<sup>2</sup> while the contact angle is at its lowest position 96° which we can clearly see in fig.2. No significant

variation is observed while decreasing contact angle. Hence, we concluded that contact angle is inversely proportional to surface energy. Shushant k rawal et.al also established relation between the surface energy and contact angle values which is inversely proportional to each other [12].

## V. CONCLUSIONS

Zirconium oxide films substrate in two substrates namely Glass and Silicon by using radio frequency (RF) magnetron sputtering. From the experimental research it is resulted that the pressure is decline and the contact angle is increase from 90°. The surface energy increase in the range from 20.31 to 25.34 MJ/m<sup>2</sup>. The contact angle shares direct relation with surface roughness and inverse relation with surface energy.

## ACKNOWLEDGMENT

This work has been supported by AICTE grant number 20/AICTE/RIFD/RPS (POLICY-III) 24/2012-13 sanctioned under Research Promotion Scheme (RPS). We are thankful to HOD, mechanical and Prof. Kamlesh Chauhan of CHARUSAT for supporting this research work. We are thankful to Dr. T. K. Chaudhuri, Professor and Head, Dr. K. C. Patel Research and Development Centre (KRADLE) affiliated to Charotar University of Science and Technology (CHARUSAT), India for granting permission to use various equipment's available in their characterization laboratory.

## REFERENCES

- [1] A. A. Voevodin, J. S. Zabinski, and C. Muratore, "Recent Advances in Hard, Tough, and Low Friction Nanocomposite Coatings New Directions in Hard Coatings," vol. 10, no. 6, 2005.
- [2] U. S. Patel, K. H. Patel, K. V Chauhan, and A. Kumar, "Investigation of various properties for zirconium oxide films synthesized by sputtering," vol. 23, pp. 336–343, 2016.
- [3] P. A. K. Elttayef, P. A. K. Abbas, and G. H. Neema, "Nanostructural Properties of Zirconium oxide Thin Films prepared by Magnetron Sputtering," vol. 4, no. 4, pp. 40–43, 2015.
- [4] L. Berkeley, "Plasma and ion sources in large area coating : A review," vol. 200, pp. 1893–1906, 2005.

- [5] P. J. Kelly and R. D. Arnell, "Magnetron sputtering : a review of recent developments and applications," vol. 56, pp. 159–172, 2000.
- [6] F. El Akkad, A. Punnoose, and G. Prabu, "Properties of ITO films prepared by rf magnetron sputtering," vol. 160, pp. 157–158, 2000.
- [7] D. P. Dave, "Characterization of sputtered nano-structured zirconium oxide films," vol. 9, no. 5, pp. 464–469.
- [8] E. Abdullah, A. Idris, and A. Saparon, "PAPR REDUCTION USING SCS-SLM TECHNIQUE IN STFBC MIMO-OFDM," vol. 12, no. 10, pp. 3218–3221, 2017.
- [9] V. V. A. Thampi, A. Bendavid, and B. Subramanian, "Author 's Accepted Manuscript," *Ceram. Int.*, 2016.
- [10] M. R. Chavda, D. P. Dave, K. V. Chauhan, and S. K. Rawal, "Tribological Characterization of TiN Coatings Prepared by Sputtering," *Procedia Technol.*, vol. 23, pp. 36–41, 2016.
- [11] S. K. Rawal, A. Kumar, V. Chawla, R. Jayaganthan, and R. Chandra, "Structural, optical and hydrophobic properties of sputter deposited zirconium oxynitride films," *Mater. Sci. Eng. B*, vol. 172, no. 3, pp. 259–266, 2010.
- [12] W. J. Khudhayer, R. Sharma, and T. Karabacak, "Hydrophobic metallic nanorods with Teflon nanopatches," vol. 275302.
- [13] W. Münz and W. Monz, "Titanium aluminum nitride films : A new alternative to TiN coatings Titanium aluminum nitride films : A new alternative to TiN coatings," vol. 2717, no. 1986, 2014.