

**INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH
TECHNOLOGY****CHARACTERISATION OF MAGNETRON SPUTTERED ZIRCONIUM OXIDE
FILM: A REVIEW****Divyesh Kumar Dave^{*1}, Krutarth Patel², Sujal Patel³, Nikhil Soni⁴, Dharmesh Chauhan⁵**Department of Mechanical Engineering, Sardar Patel College of Engineering (SPEC), Vadatal road,
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ABSTRACT

This review paper describes the recent development & studies in both the fundamental and technological aspects of zirconium oxide. The previous researcher had a commentary on the structural and Mechanical properties of zirconium nitride, zirconium oxide, and zirconium oxy nitride thin films. In their studies they analyze the hardness, roughness, coefficient of friction etc. as per the application. The varied deposition method used are reactive sputtering, physical vapour deposition (PVD), Sol-gel etc. The aim of this paper is to review and to analyze the current knowledge on the unique properties of zirconium oxides like Wettability, Optical and Tribological, properties.

KEYWORDS: Zirconium oxide based coating, Wettability, Tribological properties**I. INTRODUCTION**

Technologically, zirconium oxide is significant material. Some of its characteristics are high melting point (2680° C), excellent thermal stability, large resistance against oxidation, etc. [1,3]. Zirconium oxide found an application in a different field which includes catalysts, optical filter, laser mirrors, broadband interference filters, ionic conductors & many more [1,2,3,4].

Usually, the optimal properties of zirconium oxide are used for better coating. Such gaining properties are low thermal conductivity, high dielectric constant & high refractive index [2]. Additionally, the combine properties with aluminum oxide, ZrO₂ films require the two ingredient materials namely high hardness high electric resistance & chemical attack [5]. Uttkarsh.patelet *al.* [1] had reached to results that when argon partial pressure is increased, thickness of zirconium oxide films decreases from 433 to 385 nm. The ZrO₂ films are hydrophobic for water having maximum contact angle of 101° at 45% argon partial pressure & hydrophilic for aniline having maximum contact angle 52° at 67% argon partial pressure.

These interesting applications have asked different attempt to synthesize amorphous or polycrystalline zirconium oxide films by various techniques such as RF reactive magnetron sputtering, D.C reactive magnetron sputtering, Nitrogen assisted RF reactive sputtering, Sol-gel method, X-ray diffraction (XRD), atomic layer deposition (ALD), chemical vapour deposition (CVD) [1]. However, some procedure has some limitation in real use such as (a) costly equipment & restriction of film area are in chemical vapour deposition & sputtering process. (b) for sol-gel process, there is possibility of change in shape/size & crack deformation [4].

1. Structural/Optical property of zirconium based coating:

In 2013 Y. Adraider *et al.* fabricated zirconium oxide by combined laser/sol-gel method. In sol-gel method the solvent is taken 45g, catalyst 1.9g & distilled water 5.4g. After it is dried by laser treatment [11].

In A. Oritiz *et al.* prepared ZrO₂ films by pyrosol process on silicon substrate. The films were prepared at a temperature ranging from 300-575°C. Finally, they investigated that as substrate temperature increase the thickness decrease [8].

In 2002 Dennis. Hausman *et al.* deposited ZrO₂ films on a silicon substrate by atomic layer deposition (ALD). The thickness of films is 100nm & the wavelength are 1.54nm. The parameter taken during experiment are temperature 50-350°C at 24 torr pressure & 0.02 torr vacuum [9].

In 2016 Pranav Dave *et al.* formed zirconium oxide film by using magnetron sputtering technique. The glass substrate was taken for procedure and the thickness 751 nm. The parameter they analyzed are the target purity 99.99%, pressure 1.0 Pa, temperature 500°C. In their conclusion they reached the increase in roughness (4.1 nm) [10].

Sr. No.	Researcher	Deposition Technique	Parameter study	Structural property
1	K.Koskiet <i>al</i> (1999)	Direct current reactive Magnetron sputtering	Area=182.4 cm ² T=150-200°C P=500-2000 W Voltage-300-350V substrate distance=70 mm Frequency=30KHz	Monoclinic-Tetragonal
2	Prof. Dr. Abdul hussein k eittayeet <i>al.</i> (2015)	RF magnetron sputtering	ZrO ₂ target purity = 99.99% Sputtering team. Evacuated down = 5×10^{-5} Temp = 150°C.	Deposit ZrO ₂ film Nano particles average size in range = 13.17 & 37.29nm

2. Wettability property of zirconium based coating:

In 1997 Monika Agarwal *et al.* studied on self-assembled mono layers (SAMS) deposition technique on ZrO₂ films coating. They take the chamber temperature 100°C, chain length 9 nm, High thermal expand coefficient 10×10^{-6} [7].

In 2002 Dennis Hausmanet *al.* used atomic layer deposition (ALD) method & finalize that the cone angle is approximately 53°, which is operated using Deposition temperature 50-350°C vapour pressure 24 torr, Dynamic vacuum 0.02 torr [9].

In 2005 Yasunoir ohtsuet *al.* formed zirconium oxide films by using RF magnetron sputtering technique. The silicon substrate was taken for procedure. The parameter they analyse are target diameter 100 nm, power 200-500 W, oxygen pressure 0.1~1.0 Pa. Finally, they reached at conclusion that water contact angle is 30° [12].

Sr. No.	Researcher	Deposition Technique	Parameter study	Wettability property
1	Utkarsh.S Patel <i>et al</i> (2016)	RF Magnetron Sputtering	Chamber pr. 4×10^{-4} Substrate distance = 50 nm melting point=2680°C	Contact Angle 101°-45% 52°-67%
2	Pranav.y. daveet <i>al.</i> (2016)	X-ray diffraction (XRD)	Zinc target= 99.99% purity pressure = 1.0 pa RF constant power= 150 w target distance = 50nm Temp =500°C	Water repellent nature about 90°

3	Pengtaogao <i>et al.</i> (2000)	RF magnetron sputtering	Sputtering pressure= 5×10^{-3} mbar substrate target Dist.=60nm O ₂ Concentration rang =1% -30% base pressure = 6×10^{-6} mbar Sputtering power = 1000 w	Max contact angle = 101°
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3. Tribological property of zirconium based coating:

In 1999 K. Koski *et al.* formed ZrO₂ by D.C reactive magnetron sputtering on silicon wafer. The parameter they analyse are temperature 150-200°C, power 500-2000 W, substrate distance 70mm, frequency 30 KHz. It was found that the surface roughness (1.7-47.1) of the thin film was 10 times higher than the hard and fully oxidised thin film [13].

In 2002 S. Venkatraj *et al.* investigated on zirconium oxide on two substrates (Glass and silicon). Whereas the parameter sets like target distance 70 nm at room temperature having pressure 0.8 Pa by using reactive D.C magnetron sputtering as deposition process. They obtained that there is increase in Band gap (4.52 to 4.67 eV) and roughness (0.8 to 4.78) [3].

In 2017 Pranav y Dave *et al.* researched on magnetron sputtering process. They concluded that zirconium oxide roughness is increase on glass (4.11 nm to 16.32 nm) which is operated under temperature 500°C and target distance 50 nm [10].

In 2008 D.H Trinh deposited zirconium oxide thin film on Si (100) by reactive D.C magnetron sputtering. In their experiment they measure the depth of 15-50 nm [5].

Sr. No.	Researcher	Deposition Technique	Parameter study	Tribological property
1	Juyun park <i>et al.</i> (2009)	RF mag. sputtering	pr.=6.07 pa base pr.= 6.67×10^{-8} p=20w Temp=room temp Total time=4 hr Diameter=50 mm	Roughness is const. With O ₂ gas ratio
2	L.Q.zhu <i>et al.</i> (2002)	Nitrogen assisted RF reactive sputtering	Base pr.= 3.6×10^{-4} pa pr.=0.2 pa	Roughness at depo. =1.0 nm at 500°=1.0 nm at 700°=0.8 nm
3	N.L Zhang <i>et al.</i> (2002)	Ultra high vacuum Electron beam Evaporation	T=700°C BS. pr.= 2×10^{-9} m bar Energy=5 kv Area=500×500 nm	Roughness range =0.546 to 0.666 nm
4	Krishna murthy prasada <i>et al.</i> (2011)	Conventional & ultrasonic assisted precipitation	T=400°C to 900°C stirring time=24 hr P=2 kw	Mass fraction for Zr=74.07% for o ₂ =25.93%

5	Uttkarsh s patelet <i>al.</i> (2016)	RF magnetron sputtering	Chamber pr. 4×10^{-4} Substrate distance = 50 nm melting point = 2680°C	Surface roughness = 1.75nm
6	Prof. Dr. Abdul hussein k eittayeet <i>al.</i> (2015)	RF magnetron sputtering	ZrO ₂ target purity = 99.99% Sputtering team. Evacuated down = 5×10^{-5} Temp = 150°C.	Roughness ranged = 0.425nm & 0.512nm

4. Mechanical property of zirconium based coating

In 2016 Uttkarsh Patel *et al.* used RF magnetron sputtering as deposition process on ZrO₂ thin film. They obtained two parameters i.e. melting point 2880°C & ZrO₂ target purity is 99.99% on glass as a substrate. They resulted crystalline size is increased from 19 to 25nm [1].

In 2005 Yasunoirohtsuet *al.* was confirmed coating using RF magnetron sputtering and its thin film oxygen pressure is 0.1 to 1.0 pa. In addition, the ZrO₂ target diameter is 10 nm and the power is 200 to 500 W. They observed the decrease in resistivity of 0.2nm thick [12].

In 2015 Prof. DR Abdul Hussein *et al.* used RF magnetron sputtering process. They deposited thin film as sputtering temperature evacuated down 5×10^{-5} and temperature is 150 °C. They used glass as substrate. The thickness taken is 1.97 to 3.71 nm [24].

In 1997 Monika Agrawal *et al.* studied self assembles monolayers method on ZrO₂ thin film. Temperature is 100°C and chain length is 9nm. They used silicon as substrate. Finally, they conclude that the thickness of coated material is 25 to 30nm and grain size is more than 10nm [7].

In 2002 Dennis m Hausmanet *al.* worked on atomic layer deposition (ALD) technique. The ZrO₂ film deposition temperature is 50°C to 350°C and vapour pressure is 24 torr. Additionally, the dynamic vacuum is 0.02 torr. & the thickness of coating is 100nm [9].

Sr. No.	Researcher	Deposition Technique	Parameter study	Mechanical property
1	K. Koski <i>et al.</i> (1990)	Direct current reactive Magnetron sputtering	Area = 182.4 cm ² T = 150-200°C P = 500-2000 W Voltage = 300-350V distance = 70 mm Frequency = 30KHz	stress = 1000(c)-700(t) Elastic Module 192-228 Nano hardness = 10.2-19.1
2	M A Signore <i>et al.</i> (2009)	RF magnetron sputtering	p = 200w, T = 80°C pr. = 10^{-8} pa square area = 250 × 250 μm ² temp. range = 5° to 100°	Film thickness = 700 mm
3	Juyun park <i>et al.</i> (2009)	RF magnetron Sputtering	pr. = 6.07 pa base pr. = 6.67×10^{-8} p = 20W Temp = room temp Total time = 4 hr Diameter = 50 mm	Thickness = 30/40 mm Grain size ↑ as O ₂ ↑

4	L.Q.zhuet <i>al.</i> (2002)	Nitrogen assisted RF reactive sputtering	Base pr.= 3.6×10^{-4} pa pr.=0.2pa	Thickness at deposition=20.5 nm - 500°=19.7 nm,- 700°=18.4 nm
5	N.L Zhang <i>et al.</i> (2002)	Ultra high vacuum Electron beam Evaporation	T=700°C BS. pr.= 2×10^{-9} m bar Energy=5 kv Area=500×500 nm	Thickness=43-47 nm
6	Takeshi Yao <i>et al</i> (1996)	Aqueous sol.	T=30°C stirring Time=24 hr Rectangular Dim.10×15×1 Nm	Ordinary pr. & temp.
7	D.H..Trinh <i>et al</i> (2008)	Reactive D.C Magnetron sputtering	T~300° Base Pr.= 2.66×10^{-4} pa pr.=0.36 pa Distance from substrate=100 mm p=500 W	Thickness=100 nm Grain size~10 nm

II. CONCLUSION

Zirconium based coatings has a several requirements in various applications such as chemical industries, for casting, laboratories, Nuclear energy etc. because of its high refraction index, good gem material, high temperature, corrosion resistance etc. By keeping film at room temperature, the thickness was in range of 30~40 nm and the roughness was constant [2]. When argon partial pressure is increase thickness of ZrO₂ films decrease (433 nm to 385 nm) the films are hydrophobic for water and hydrophilic for aniline [1]. When annealing temperature increase than 700°C, the films began crystallize [16]. Finally, the Nano hardness and elasticity were below the values of transparent and colorless thin film [13].

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