A Review on the effects of PVD RF sputtering parameters on rare earth oxide thin films and their applications

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Abstract: This article provides a review on effect of processing parameters on the quality and performance of thin films of rare earth materials deposited through PVD RF sputtering technique. The applications of these thin films include optical, electronics, medical films, defence and renewable energy technologies. A systematic review of the literature revealed that the sputtering parameters such as plasma power and substrate temperature have the most impact on the grain size of the deposited thin films. Larger grain size was observed to be at higher sputtering power and higher substrate temperatures, while lower grain size was obtained when the sputtering power is decreased. The optical property, in particular the band energy gap (Eg) was found to improve with the film thickness. Thin films produced at higher doping concentration and subsequently annealed at higher temperature was found to increase the crystallinity of thin films.

Keywords: Physical vapour deposition (PVD), RF sputtering, Rare earth materials.

1. Introduction
In recent year, rare earth oxide (REO) materials have been emerges as one of the demanding materials worldwide for various applications because of their unique scintillation, phosphorescent, magnetic and catalytic properties. As per report almost 79% of rare earth in worldwide is produced by China. As recent increases demands of rare earth oxide over worldwide, China becomes the largest supplier of these materials. These oxide elements are a group of seventeen chemical elements of lanthanides in periodic table from atomic number 57 to 71. Their atomic masses subsequently vary from 138.9055 AMU for La to 174.967 AMU for Lu. They have been established as unique materials because of their electronic structure of the materials, where their generalized electronic structure may be represented as (Xe) 4fⁿ 6s², where n varies from 1 to 14. Their unique optical and electronic properties are dictated by their valence electrons in the partially filled 4f orbitals. These include applications in optical, electronics, medical and renewable energy devices because of their excellent magnetic, scintillating, phosphorescent, and catalytic properties [1,2,3].

The applications of these rare earth oxides include computed topography (CT) scanner x-ray imagining system [4], ultrahigh-resolution imaging and light emitting diodes(LEDs) [5,6,7], TFT(thin-film transistor
devices [8], high-efficiency silicon solar cell [9], Si-based optoelectronics devices [10], spintronics fields [high-speed magnetic filters, sensors, quantum transistors] [11], light emitting diode (LEDs) [12], active devices like non-volatile memory, pyro-electric detectors and microwave devices [13], ramp junctions & field effect devices [14], oxygen sensing instruments [15], dynamic random access memories (DRAMs), spintronics and humidity sensors [16,17], metal-oxide-semiconductor (CMOS) devices [18], switching mechanism for logic devices and memories [19] and high-efficiency silicon solar cells applications [20,21]. Table 1 shows the applications of some rare earth materials.

Table.1 Application based on research area [1]

| Research Area                  | Applications                                                                 |
|--------------------------------|-------------------------------------------------------------------------------|
| Opto-electronics and electronics| Television screen, LEDs (light emitting diode), CFLs (compact fluorescent lamps), baggage scanners, Lasers, optical glass, fiber optics, masers, radar detection devices. |
| Manufacturing                  | Metal alloys, High strength magnet, stress gauges, polishing powder            |
| Medical and Bio-medical         | X-ray machines, nuclear medicine imaging, X-ray tubes, dental laser, cancer treatment applications, genetic screening tests, |
| Industrial                     | Ceramic pigments, colorants in glassware, chemical oxidizing agent, polishing powders, |
| Renewable energy technologies   | Wind turbines, batteries, catalysts and electric cars                         |

In this report, review was done on effects of process parameters and applications of rare earth oxide thin film deposition by Physical vapour deposition (PVD) rf sputtering. PVD rf sputtering is an effective method for depositing high quality thin films of oxides, carbides and nitrides of metals and semiconductors. The process is capable of obtaining high quality thin films with high deposition rates, good adhesion, uniformity in surface morphology, homogeneity, ease of operation and economical for depositing even large areas at room temperature without the need for melting as in thermal spraying. The main disadvantages that accompany this process are target material poisoning and possibility of arcing from the plasma during operation [22,23,24].
A typical flow chart of the deposition process has been explained in Figure 1. Here initial setup such as target material setting and substrate cleaning is conducted, then the chamber is evacuated and argon gas (at a predetermined flow rate) is introduced into the chamber. The plasma is initiated inside the chamber by striking with the power source. As a result energetic ions (argon ions) are formed in the plasma that bombards the target materials (cathode of discharge) atoms. As a result of the plasma bombardment, molecules of the target material are ejected/vaporized from its surface in a steady stream that is deposited on the substrate (anode).

![Figure 1. Flowchart of PVD rf sputtering working principal](image)

Other than the PVD RF sputtering method there are other methods used for deposition namely pulsed electron beam, pulsed laser deposition (PLD)[25,26], spray pyrolysis[27], sol gel[28,29], DC sputtering[30], and metal organic vapour phase epitaxial[31]. Morea et al.[25] and Prakash et al.[26] grown rare earth materials by PLD method and thin films was good quality but some irregularities were observed when deposited in large area surface. Similarly Anand et al.[27] have grown using spray pyrolysis and observed that grain size of thin films produced was not interconnected to each other. Zhuang et al.[28] and Zargouni et al[29] grown rare earth by sol gel method and noticed that surface roughness of the thin films was in critical stage than required criteria. Thus compared to the other processes this is capable of growing smooth, uniform surfaces with excellent adhesive properties than the other methods [4-21]. This technique involves less time for fabrication and capable for mass production for large area depositions whenever required. This is advantageous since in electronic and opto-electronics industry high quality thin films are needed with smooth and good surface finish.

There are many review articles on deposition of rare earth materials deposited through PVD rf sputtering method, i.e., Goh et al. [32] reviewed rare earth Samarium oxide (Sm$_2$O$_3$), Alam et al.[33] reviewed rare earth materials doped with ferrite garnet-type thin films, Gagnier et al. [34] reviewed rare earth neodymium and samarium for permanent magnet and superconducting materials. Winick et al. [35] reviewed rare earth erbium and neodymium by rf sputtering method. However they have not correlated their processing parameters to their quality and performance.

RF sputtering deposition process is mainly affected by its parameters such as plasma power, substrate temperature, deposition time, substrate orientation, argon pressure, substrate to target materials distance, substrate rotation, target tilt etc [5]. These parameters can affects coated parts individually or by coupling others parameters also. However doping of materials on base materials and flow of gas has also affected thin surface morphology such as optical and structural properties. This paper therefore correlated through the review of published articles the effects of processing parameters on the quality and performance of thin films of rare earth materials deposited through PVD rf sputtering technique.
2. Review and Discussion

2.1 Sputtering Power

RF sputtering power has vital role in achieving smooth, adhesive, uniform thin films. The deposition rate of the coating largely depends on its sputtering power. Topping et al [4] have grown lutetium oxide on graphite substrate and shown that the maximum working sputtering power for deposition of lutetium oxide could be 100 W. At powers higher than 100 W the oxide target material was shown to get irreparably damaged. With copper backing and water cooled oxide target materials can resist sputtering power up to 100 W. The sputtering power significantly affects the surface morphology of thin films. Thin films deposited at 50 W plasma intensity provided low deposition rate, while at 75W produced balanced deposition rates, but that at 100 W produced high deposition rate. The higher deposition rates produced poor crystallinity due to insufficient time for atomic ordering. Thin films with poor or partial crystallinity, however, improved its crystallinity and its performance after sufficient heat treatment. Application of these thin films include for x-ray imaging system of computed topography (CT) scanner [4].

Nagarkar et al. [5] deposited europium oxide doped lutetium oxide on transparent optical ceramic and revealed that morphology of thin films largely dependent on input sputtering plasma power. High x-ray absorption efficiency of the thin films was capable to provide full absorption of the incident irradiation with minimal thickness that enables its use in ultra high-resolution imaging application [5]. Kaya et al. [18] have similarly grown samarium oxide thick films on silicon substrate and shown that the crystallization of the films improved as the sputtering power increased and moreover, the grain size also increases with the increase in plasma power. The samarium oxide thick films were monoclinic B-type structure and with increased crystallinity their performance in complementary metal-oxide-semiconductor (CMOS) devices improved. Application includes for metal-oxide-semiconductor (CMOS) devices [18].

2.2 Deposition time

The deposition time, like the sputtering power also affects the thin films thickness of films are largely depends on deposition time. However, the deposition time is usually counted after the ignited plasma is stabilised so a buffer time, depending on the instrument and its condition is kept that is not included in the deposition time. Chen et al.[15] have grown cerium oxide thin films on both glass and silicon substrate and suggested that the deposition time has influenced the films smoothness as well its thickness. The lower the deposition time the more the average roughness of the thin films. While at higher deposition time the surface shows a smoother surface and increased adhesion to the substrate. With decreased deposition time and oxygen flow rate the crystal (grain) size decreased which improved its performance in oxygen sensing instruments. Application of these thin films is for oxygen sensing instruments.

2.3 Substrate orientation

The substrate orientation, in case of the single crystal substrate, is very important in deciding the properties of the thin film. The crystallinity and residual stress due to lattice mismatch largely depends on substrate orientation. The substrate orientation should be chosen such that the lattice mismatch between the coating and substrate is minimised. Roy et al.[7] have shown that for europium doped lutetium oxide thin films when grown on different orientations of (100), (110) and (111) single crystal yttria-stabilized zirconia (YSZ) substrates, the surface morphology, roughness and crystallinity changed enormously for the same experimental conditions. Their radioluminescence (scintillation in incident x-rays) also changed greatly with the orientation which make them suitable for application in high resolution x-ray detectors used in computed topography (CT) scanners [7].

2.4 Substrate to Target material Distance

The distance of the substrate to target materials, also referred to as standoff distance, also affects
the deposition rate as well as the grain size distribution. Mandal et al. [6] have shown that the microstructure of europium doped lutetium-gallium oxide could be varied greatly by changing the standoff distance from 8 cm to 20 cm. By increasing the standoff distance by more than twice, the thickness, roughness and adhesiveness increased structural properties drastically. This also improved its optical properties for use in wide band gap semiconductors. The standoff distance is related directly to the ions evaporated by the plasma and hence affects its deposition rate. Application of these films includes for opto-electronics devices such as LEDs.

2.5 Substrate temperature

The substrate temperature plays a vital role in achieving better crystallinity in the thin films. It is well known that the substrate temperature improves the nucleation and growth kinetics of the thin film on the substrate which affects its grain size. With increasing substrate temperature the grain size increases as the crystallinity of the grains increase from amorphous to columnar to equiaxed [36]. Sibaja et al. [13] have shown that for lanthanum oxide deposited on silicon substrates the crystal structure changes from hexagonal at 80°C to FCC above 200°C. Moreover, with change in crystal structure the optical properties also improved for its application in sensory devices like non-volatile memory, pyro-electric detectors and microwave devices. Tsai et al. [16] have shown that cerium oxide deposited on silicon substrates the substrate temperature significantly affected its structural properties. Here the orientation of the thin films changed as the substrate temperature was increased which improved its optical performance in dynamic random access memories (DRAMs). Application includes for DRAMs devices.

2.6 Doping of materials

The purity and hence concentration of dopant, affects its crystallinity and grain size. Ren et al. [8] deposited erbium doped tin oxide on quartz substrates and revealed that as the erbium concentration increases from 0 to 4.6%, its electrical performance improves drastically which improved its potential applications in thin-film transistor (TFT) devices. Miura et al. [9] on the other hand have also doped erbium in tantalum oxide and suggested that addition of erbium although improves the uniformity and adhesiveness of the coating, however, significantly affects its photoluminescence (PL) properties. Applications includes for high-refractive-index and light-emitting material of a multilayered photonic crystal and high-efficiency silicon solar cell.

Cerqueira et al. [10] grown erbium doped nano crystal silicon thin films and shown that erbium concentration of 0.03 % gives the characteristic of Er³⁺ emission, while 0.55 % do not give the characteristics emission. This indicated that with increase in the erbium concentration the metallic erbium clusters in the films do not get activated. Hence this affects its performance for Si-based optoelectronics devices. Fang et al. [20] have grown terbium doped zinc oxide on glass substrate and suggested terbium acts as good dopant for formation of transparent and conductive oxide (TCO) ZnO films. The crystallinity of the thin films were found to improve as the dopant concentration was increased up to 4.1% which improved its performance in thin film solar cells.

2.7 Flow of gases (Argon / Oxygen/Nitrogen gas)

The gas flow during the sputtering process also plays a vital role in the deposition process. The gas mixture can vary both the ratio of the oxygen to argon or nitrogen and also the flow rate of the gases for achieving the desired crystallinity of the thin films. Shalaan et al. [11] have grown gadolinium nitride films on glass substrate by keeping Ar/N₂ mixed ratio (5/1) and it was observed that the guttering during pre-sputtering reduces the oxygen ratio in the chamber resulting in formation of amorphous thin films with high band gap energy which makes it suitable for applications in spintronics such as high-speed magnetic filters, sensors and quantum transistors[11]. Deng et al. [12] have shown that when gadolinium-doped yttrium aluminum garnet deposited on boron doped silicon substrate their cathodoluminiscence (CL) properties were substantially improved when the oxygen flow rate was low thus improving its application.
in light emitting diode (LEDs)[12].

Owens et al. [14] on the other hand have shown that the structure and surface finish of the cerium oxide thin films deposited on YSZ substrate improved with decreasing Ar/O\textsubscript{2} ratio. Moreover the resistivity of the thin films results decreased substantially improving its superconductivity, which can be applied for ramp junctions & field effect devices[14]. Atta et al. [19] have also deposited samarium oxide thin films on quartz substrate observed that the band energy gap of the thin films were found to improve by increasing the Ar/O\textsubscript{2} ratio which improves its optical properties for potential use in application for switching mechanism of logic devices and memories.

3. Discussion

PVD rf sputtering deposition method has produced rare earth oxide thin films with smooth, uniform, good adhesive and defects free surface in short time of periods. Processing parameters of rf sputtering has significantly influences the thin films of rare earth materials deposited. Based on processing parameters such as plasma power, substrate temperature, deposition time, substrate orientation, flow of gases, substrate to target materials distance, and doping of materials the review is carried out. The effects of processing parameters and application of deposited thin films have been discussed here briefly. Throughout it was observed that each parameter has its own effectiveness over surface morphological behaviour of rare earth oxide thin films. Optimal choice of parameters had showed better surface morphological behaviour, better phase, electrical and optical properties. Rare earth materials has capability for wide application for various field such as opto-electronics, electronics device, medical films, industrial field and renewable energy technologies because of their excellent magnetic, scintillating, phosphorescent, and catalytic properties.

4. Conclusion

This report reviews the effects of rf sputtering parameters on surface morphology, structural and optical properties of rare earth oxide materials thin films and their applications. Effects of several parameters such as plasma power, deposition time, substrate orientation, substrate to target material distance, substrate temperature, doping concentration of materials, Argon/Oxygen/Nitrogen gas mixing ratio on rare earth thin films was studied. Through observation it was concluded that, Sputtering power has more influenced surface characteristics of thin films, as sputtering power was increased. Grain size as well as crystallinity of thin films surface showed excellent improvements upon sputtering power increased. 75W sputtering power showed a balanced and controllable than other sputtering power. Deposition time play an vital role for obtaining smooth and defects free surface finish, through study observation was concluded that higher deposition time has showed smooth, adhesive as well as well good surface roughness than lower deposition time. Substrate orientation affects the crystallinity of surface structure hence preferred orientation of substrate must be used during deposition process. Substrate to target materials has also shown impacts on surface morphology, lower distance showed thick and more surface roughness. Substrate temperature plays an important role for obtaining good optical properties. Doping concentration affects the surface morphology of thin films as the doping concentration was increases optical as well as electrical properties showed excellent improvements. Excellent photoluminescence properties of thin films showed excellent in low argon/oxygen mixing ratio.

The applications of rare earth oxide thin films are available for optical, electronics, medical films, defence and renewable energy technologies.

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