A Study on Optical Waveguide Application of Lanthanum III Oxide (LA_2O_3) Thin Film Prepared by Sol-gel Method.

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Abstract

Optogeometric properties of La_2O_3 (Lanthanum Oxide) thin films prepared by sol-gel technique have been investigated. Characterization was derived by M-line spectroscopy, X-ray diffractometry and waveguide Raman Spectroscopy. M-line spectroscopy measurements revealed a refractive index of 1.592±0.001 on Pyrex substrate for a wavelength of 543.4nm and thin film thickness of 850nm and 1.589±0.001 on silicon wafer and thickness of single layer is between 40 and 60nm. X-ray diffractrometry has shown that the film has monoclinic structure. Waveguide Raman Spectroscopy has revealed a mixture of La_2O_3 and $La_2O_2CO_3$ (Lanthanum Oxide Carbonate) which are mainly nanocrystalline and polycrystalline respectively. The research has shown that the La_2O_3 thin films produced can be used as a planar optical waveguide. The results obtained are comparable with the works of other scientists using different measurement techniques.

Keywords: Lanthanum Oxide, Nanocrystalline, waveguide, X-ray diffractometry, spectroscopy, optogeometric

Introduction.

Lanthanum III oxide (La_2O_3) is a chemical compound containing the rare earth element Lanthanum and Oxygen. It has the largest band gap (3.4 eV) of the rare earth oxides, it also has the lowest lattice energy with very high dielectric constant (27); characteristics which favours optical waveguide application (Anguscuik *et al.*, 2001).

La_2O_3 has P-type semi-conducting properties because its resistivity decreases with increase in temperature, average room temperature resistivity is 10 Ω-cm (Balsubramanian, 1981). Its phase behaviours are solid liquid and gas, while spectra data are ultraviolet (UV), infrared (IR) and molecular spectroscopy (MS). Lanthanum as an element is a malleable, ductile, soft and silvery white metal belonging to group three on the periodic table; atomic number 57 and atomic mass 138.91, melting and boiling points, of 920°C and 3464°C respectively and specific gravity 6.162 g/cm^3 (Goa *et al.*, 1991, Wang et al, 2000).

At low temperatures, La_2O_3 has an A-M_2O_3 hexagonal crystal structure (Kale et al., 2005). Its crystalline phase can be used for the potential applications of the ceramic–mesoporous structures, as an ingredient for the manufacture of piezoelectric and thermoelectric materials, automobile exhaust-gas converters contain La_2O_3 (Cao et al., 2005). La_2O_3 is also used in X-ray imaging intensifying screens, phophors as well as dielectric and conductive ceramics.

In this work, we have demonstrated the syntheses of hexagonal crystal phase (h-La_2O_3) at room temperature by using Sol-gel method. It is believed that the La^{3+} metal atoms are surrounded by a 7 coordinate group of O^{2-}atoms, while the oxygen ions are in an octahedral shape around the metal atom however, we see that there is one oxygen ion above one of the octahedral faces. In contrast to low temperature case, at high temperatures the La_2O_3 converts to a C-M_2O_3 cubic crystal structure. The La^{3+} ion is surrounded by a 6 coordinate group of O^{2-} ions. However, La_2O_3 in ferroelectric materials content has still needed to be studied due to its structural collapse during formation of the mesoporous phase and its phase transformation from hexagonal crystal phase to cubic crystal phase (Duan *et al.*, 2008; Alexazder *et al.*, 2006; Dercz *et al.,* 2006).

A waveguide is a structure that causes a wave to propagate in a chosen direction with some measure of confinement in the plane transverse to the direction of propagation (Cantel, 2003). A waveguide function as a conduit to transmit electromagnetic energy from the source to the receiver, if possible, with out distortion and attenuation. The attenuation or extinction of the propagating wave along the waveguide can be due to absorption, scattering or both (Bahari *et al,* 2008).

For a thin film coating to function as a waveguide, and not just an antireflection or protection coating, it must have the property that an incident light propagates parallel to the plane of the substrate, (Cole, 1995) figure 1. However, they are broadband anti-reflection coatings (BBAR), beam splitter coatings, dielectric high power laser mirrors, fiber optic coatings, anti-reflection coatings (AR) and reflection coatings fabricated from thin film materials to meet specific needs (Ibanga, *et al,* 2003).

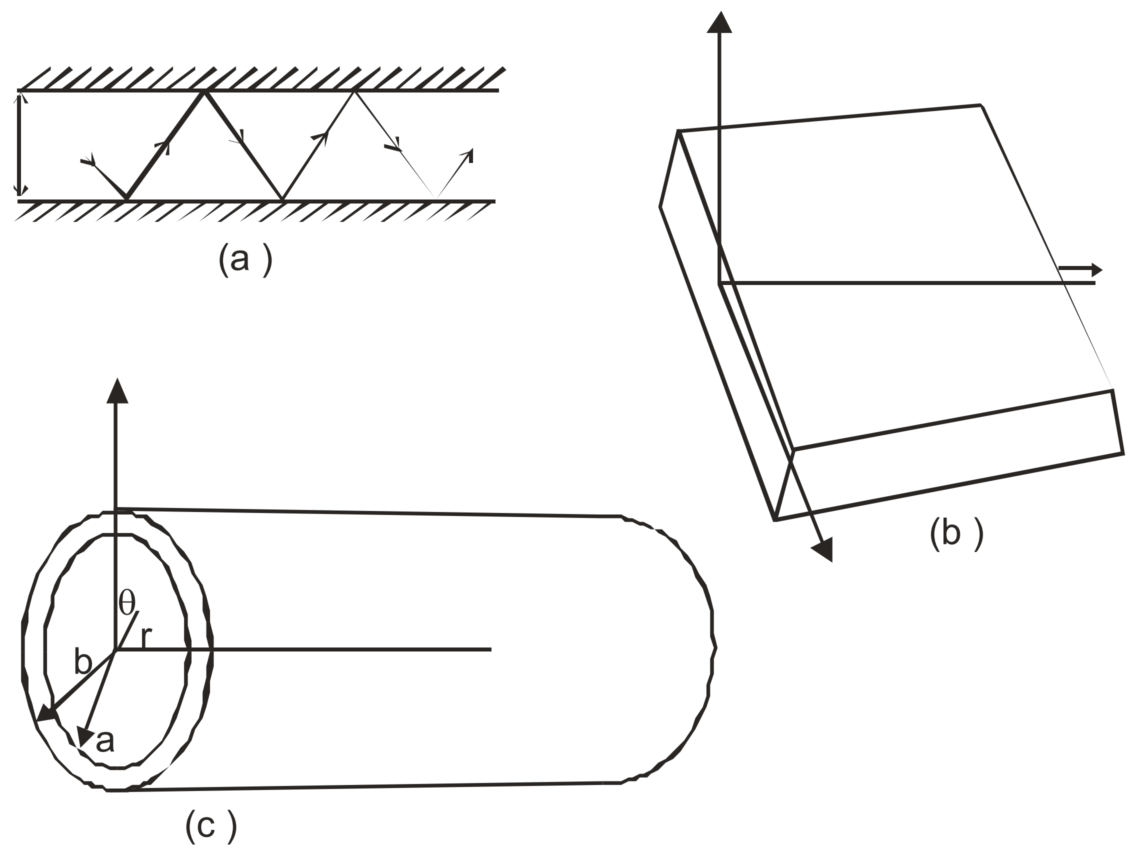

Fig. 1: (a) Guided light in a waveguide, (b) Planar Waveguide, (c) Cylindrical Waveguide.

The attenuation and scattering coefficients of materials adapted for the production of optical waveguides are kept very low and hence materials for these purposes should be non-absorbing, optically homogenous and isotropic. Leakage of transmitted waves through the boundaries of the conduits should be significantly negligible (De Asha *et al.*, 1998). In a waveguide this is achieved by the process of total internal reflection at the boundaries. This is a condition whereby the amplitude of the incident wave becomes equal to the amplitude of the reflected wave. Given two such waves;

$$E_y = A_o \exp(-ik_x) \qquad (1)$$

$$E'_y = B_o \exp(-ik_x) \qquad (2)$$

At total internal reflection, $A_o=B_o$. In an optical thin film waveguide the thin film forms the core. A waveguide is constructed such that the thin film material with index of refraction n_f is sandwiched at the top by air and underneath by a substrate material all satisfying the condition that the refraction index of the film is greater than that of the substrate and that of air or the top covering layer (Duan *et al.*, 2008). This condition is needed for waves to be guided along the waveguide. This follows that wave propagation is sustained along the waveguide when the angle of incidence i_p, on the face of a prism coupled to the waveguide (Niemiem *et al.*, 2001).

In thin films production and applications, determination of optogeometric parameters such as thickness (t) and refractive index (n) of the thin films is very crucial. Refractive index (n) and thickness (t) for isolated films could be measured using very familiar conventional techniques, which become less effective and efficient when the thin film is placed in between other materials either in a mechanical, electronic or optical system.

It was noted that thickness is one of the most important thin film parameters and largely determines the properties of a film (Case 1983). As a corollary, almost all properties of thin films are functions of the film thickness. Several methods of measuring thin film thickness abound. These measurement techniques of thin film parameter may yield different results. Some methods are well adapted for finished films while others are for monitoring the thickness during the process of film fabrication (Hohlbein *et al.* 2004). The monitoring or insitu approach are said to be highly valuable because they allow the fabrication of films of particular thickness.

Refractive index measurements of nitride films like AlGaN compositions of up to 38% obtained by means of reflectance and transmittance spectroscopy have been investigated (Huang *et al.* 2004; Dobrowolski *et al.* 1983). The films used were less than 0.5 μm in thickness, which permitted direct calculation of even the absorption coefficient. Consequent upon thin film formation and growth processes, they are usually not perfectly smooth. Certain areas of the film could be evidently rough in nature, resulting in different thickness at different places. This interesting observation has given rise to the development of models for investigating the effect of surface roughness and thickness variation on the optical spectrum of thin films. In these models their solutions contain an index-thickness product. Thus, the index of refraction (n) and the thin film thickness are intimately related.

For the La_2O_3 thin films, which constitutes the main thrust of this work, their performance as a waveguide when coated on a glass substrate is a function of the refractive index and thickness. Given the relevance of these parameters of a thin film vis-à-vis the wide range of possible applications, La_2O_3 was deposited using the Sol-gel route to produce La_2O_3 thin films that could be used as planar waveguides. In spite of few studies regarding to the sol-gel method, the sol-gel method has some merits, such as the easy control of chemical components, and fabrication of thin film at a low cost to investigate structure and optical properties of La_2O_3 thin films (Ilican *et al.*, 2008). The elaboration conditions are precisely described. Refractive index and thickness of the film will be obtained by M-line Spectroscopy. X-ray Diffractometry and waveguide Raman Spectroscopy shall be used for structural characterization and study of vibration modes.

Experimental Section.
Material Preparation.

The La_2O_3 solution was prepared using lanthanum acetylecetonate $La(acac)_3$ (Aldrich) as the starting material. $La(acac)_3$ was dissolved in absolute ethanol (Prolabo, analytic reagent) and then mixed with 8M hydrochloric acid in an appropriate ratio. The solution was stirred at 60^0C for three (3) hours (Bahari *et al.*, 2011). Then, the solution was filtered using a 0.22 μm filter and kept in a sealed bottle.

La_2O_3 sol-gel thin films were prepared on the silicon wafer using the dip-coating technique. In general, the quality of the sol-gel waveguides depends on many parameters of the film deposition process, such as the viscosity of the solution, the substrate temperature and the humidity of the environment in which the layer is deposited (Bahari, *et al.*, 2005; Cao *et al.*, 2005). Although for many materials, good quality sol-gel films can be prepared without care for the humidity, for La_2O_3, in particular the humidity greatly affects the quality of coated films (Ohring, 2002). For this reason, humidity controllable dip-coating system was used, Figure 2.

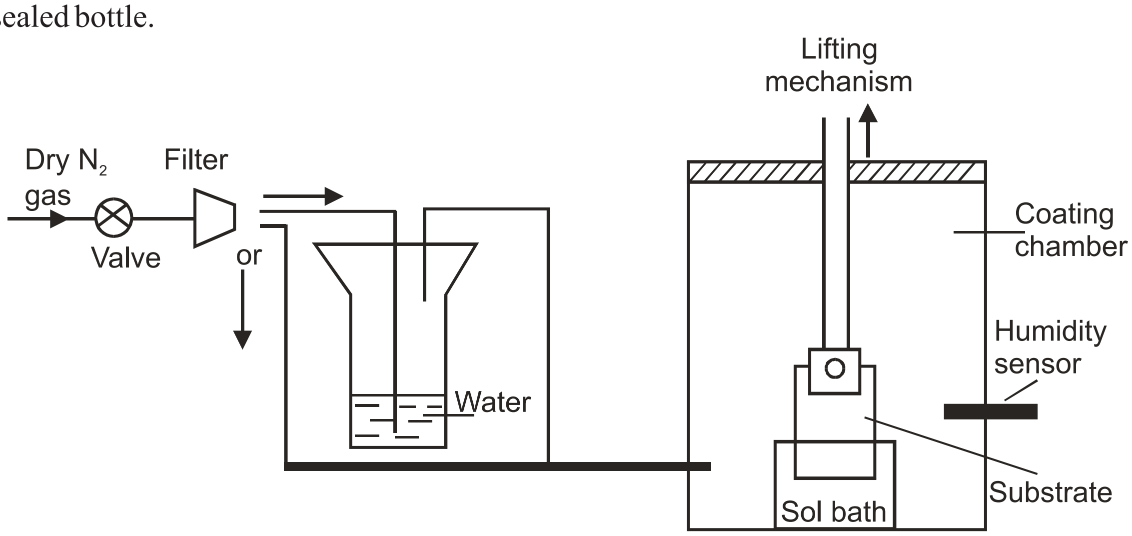

Fig. 2: Schematic diagram of humidity controllable dip-coating system.

As indicated in the diagram, the dry and clean N_2 gas continuously passes through the water and brings the water vapour to the dip-coating chamber or the N_2 gas goes directly to the chamber. The chosen way will depend on the required humidity and the surrounding atmosphere. The relative humidity in the chamber was monitored by a relative humidity sensor and modulated by adjusting a valve to control the flow of the gas. The relative humidity was kept at 15% in order to obtain a high quality transparent La_2O_3 film.

A Pyrex substrate (borosilicate glass; n=1.472 at 632.8nm; softening temperature 650^0C) was used. The carefully cleaned substrates were dip-coated at room temperature in a glove-box, using the filtered solution and a constant withdrawal speed of 80mm min^{-1}. A stack of twenty deposited layers was produced for the waveguide application figure 2. After coating, the sample was dried at 80^0C for 15 min and then heat-treated at 600^0C for 30min between each coating under a pure oxygen flow to promote organic burnout and partial densification of the film (Huang *et al.*, 2004). After heat-treatment, the transparent, crack-free and planar waveguiding layers were stored in a clean dust free bottle to be characterized at room temperature.

Characterization.

The prism-film coupler and m-line spectroscopy.

The structure of the Prism-film coupler as presented schematically in figure. 3 below is ideally suited for the characterization of materials and processes adapted for optical waveguide fabrications.

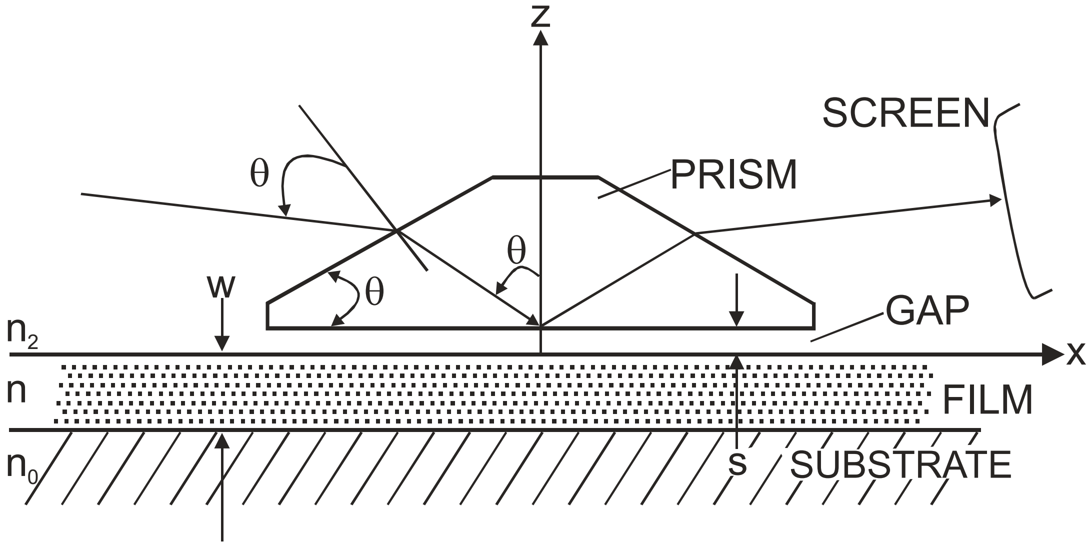

figure 3: Structure of Prison-film coupler for determination of refractive index.

The prism-film or dielectric thin film waveguide coupler consists of a prism placed closely above a dielectric thin film with an air gap left in between them. The film is usually deposited on a substrate of known refractive index. The refractive index (n_f) and thickness (t) of the film is unknown and is often deposited on the substrate by a convenient coating method (chemical vapour deposition, sputtering, dip or spin coating). The waveguide is coupled to the prism by a coupling head or adjustable screw figure 4. A laser beam incident on the face of the prism strikes the base of the prism and experiences a normal total internal reflection of the incident beam at the base of the prism (Monneret et al 2000). The waves in the prism and in the film are coupled through their evanescent fields in the air gap. At certain discrete values of the incident angles, called mode angles, there is optical tunneling of the photons across the air gap into the film which enters into a guided optical propagation mode causing or leading to a sharp drop in the intensity of light reaching the detector. The experimental set-up for the observation of the mode angles is illustrated in figure 5.

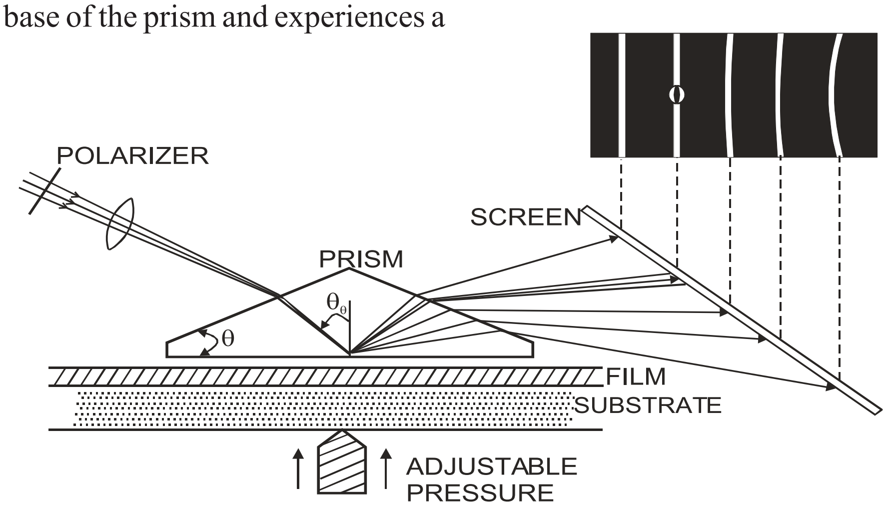

Fig. 4: The Prism-Film Coupler showing the mode spectrum (m-line) due to reflection and refraction from the thin waveguide.

The coupling of a laser beam into the film can be observed in various ways depending on the characteristics of the film, the substrate and also on the type of prism used. Coupling for evaporated and sputtered films with negligible roughness or in homogeneties like ZnS, Al_2O_3 and CO_2 may be measured with the symmetric prism, when deposited on thin substrates that are slightly flexible.

Coupling of the two waves is detected by the appearance of streaks of guided light in the film and by the appearance of bright or dark lines. In cases of the observed modes of the thin film waveguide a very bright spot is observed on the lines. These lines are referred to as m-lines, and the scientific study of the observed lines in relation to the characteristic of the film, is the spectroscopy. In application to biological liquid sensing, Auguscuik, et al investigated multilayer planar waveguide parameters using m-line spectroscopy results. M-line spectroscopy is noted to be a useful method for determining optogeometric properties like refractive index and thickness (Anguscuik *et al.,* 2001). It was noted that the angle of incidence, say i_p on the face of the prism corresponding to a particularly observed m-line which governs the coupling, determines the phase velocity, V_x in the x-direction;

$$v_m = \frac{C}{n_p \sin i_p} \qquad (3)$$

If v_m is the velocity of one of the characteristic modes of propagation in the thin film waveguide, where m is the mode order ($m = 0, 1, 2\ldots$) the m-lines for a particular film are observed on the screen or photo detector when the following conditions given by equation 4, is satisfied; that is

$$v_m = v_x \qquad (4)$$

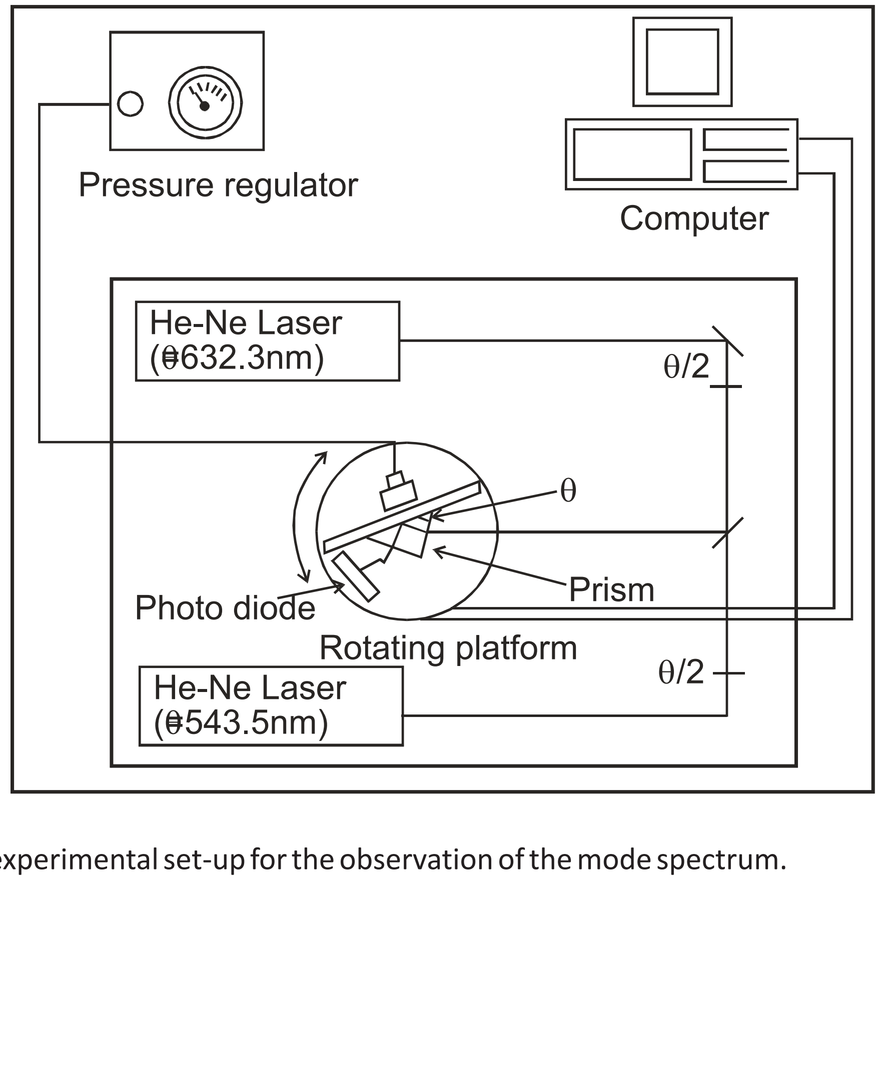

Fig. 5: The experimental set-up for the observation of the mode spectrum.

Hence to form a propagating mode, of any order, the phase velocity of the beam in the x-direction must be equal to the phase velocity of one of the propagating modes in the guide. They equally highlighted the fact that, by determining these synchronous angles of strongest coupling, the characteristic propagation constant of a given film relative to the propagation constant k in free space can be found experimentally and theoretically.

$$k = \frac{\omega}{c} \qquad (5)$$

Where ω is the angular frequency and c is the velocity of light. As soon as a minimum of two modes are found, film thickness and refractive index can be calculated. In a rough approximation, the angular location of the first observed mode, determines film index while the angular difference between the modes give the thickness. The experimental approach is fully automated and requires an average of twenty seconds. The number of modes supported by a film of given refractive index increase with film thickness. For most films and substrate combinations, a thickness of about 1000-2000Å is required to support the very first mode while films in the one-micron range can support as may as four or five modes. The results obtained for thickness and refractive index will be more accurate if the film of the waveguide can support at least two different modes of the same polarization (Urlacher, *et al.,* 1996).

M-line spectroscopy and evaluation of refractive index and thickness.

Optical measurements were conducted at a wavelength of 543.4nm (He-Ne laser). The refractive index and thickness of the La_2O_3 were determined by m-lines obtained from the spectroscopic analysis.

X-ray Difractometry:

X-ray diffractometry measurements were performed using a SIEMENS D500 X-ray diffractometer with a wavelength of 1.5406Å from the Cu Kα line. X-ray diffraction was used to follow the structural evolution of the film at the annealing temperature. A special sample of La_2O_3 was dip coated on a pyrex substrate. It has twenty layers. After coating of each layer the sample was dried at 80^0C for 15min and then annealed at 400^0C for 15min. The samples with different annealing temperatures T were analyzed by X-ray diffractometer at room temperature.

Waveguide Raman Spectroscopy:

The laser propagation length when using the twenty coated sample was around 2cm. This propagation was enough to allow Waveguide Raman Spectroscopy (WRS) measurement. Room temperature WRS experiment was used to ascertain the structural and nanostructural attributes of the sol-Gel La_2O_3 films (Cao *et al.,* 2005; Hohlbein *et al.*, 2004).

Experimental set-up for Waveguide Raman Spectroscopy is shown in figure 6. Briefly the Krypton laser beam (power beam: 650mW, $\lambda = 647.1nm$) was focused near the edge of a prism coupler (LaSF 35) used to launch the light into the film. The scattered light was analyzed with a Jobin-Yvon U1000 double-monochromator followed by an RCA photomultiplier tube. With this geometry, scattered radiation was collected normal to the propagation direction of the beam within the planar waveguide and allowed to obtain high signal-to-noise Raman spectral to be obtained without any Raman contribution from the substrate. The signal was processed by an Ortec Photon counting system and recorded under computer control.

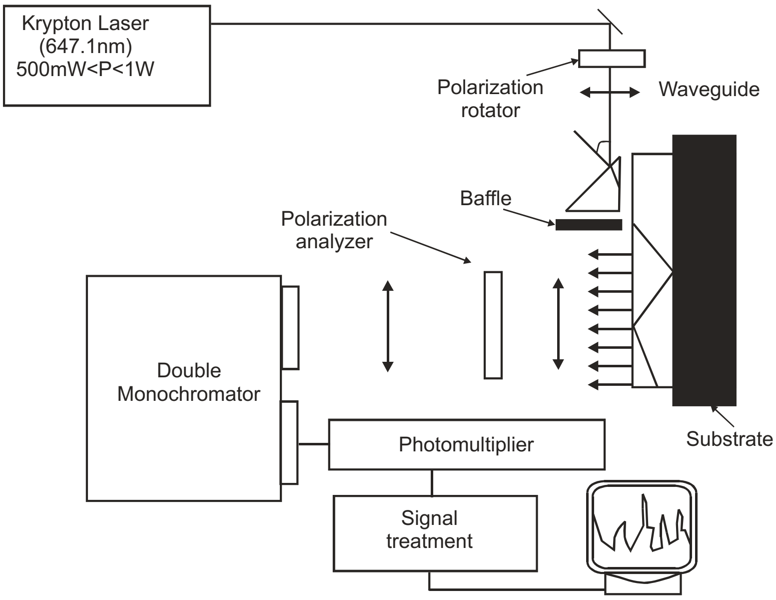

Fig. 6: Schematic representation of the experimental set-up for Waveguide Raman Spectroscopy (WRS).

Results and Discussion.

The La_2O_3 films obtained were homogeneous, transparent and crack free. Since planar optical waveguiding involves light propagation within a thin layer of a transparent material, it can only occur when the layer has a higher refractive index than the adjacent layers and also has sufficient thickness to support a guided mode. A multicoated film should be used for m-line spectroscopic measurement to provide multimode waveguide. It was found that the La_2O_3 films with twenty layers can support one mode of each polarization (1 transverse electric mode: TE_0 and one transverse magnetic mode: TM_0). In m-line spectroscopy measurements, more accurate results can be obtained if the films support at least 2 TE and 2 TM modes. In order to elaborate a 2TE – 2TM waveguide, it is necessary to deposit around 40 stacked layers on the substrate.

The mode-coupled conditions are used to determine the optical properties of La_2O_3 films (Ibanga *et al.,* 2003). Assuming a homogeneous layer with stepwise refractive index and constant thickness, refractive index of the twenty coated layers of La_2O_3 waveguide were obtained using m-line spectroscopy and spectroscopic ellipsometry methods as presented in table 1 at a wavelength of 543.4 nm.

Table 5.2: Results for the zero order mode for TE and TM.

Table 5.2a: *Results for the zero order mode using m-line spectroscopy.*

Graphical Result	Numerical Results	Experimental Results
n 1.590 ± 0.020	n_f =1.592 ± 0.001	n_f =1.594
t = 860.00 ± 0.03 nm	t = 850.18 ± 0.002 nm	t = 852

Table 5.2b: *Results for the zero order modes using spectroscopic ellipsometry.*

Graphical Results	Numerical Results	Experimental Results
n_f = 1.588±0.020	n_f = 1.589±0.001	n_f =1.590 ± 0.013
t = 859.00±0.03nm	t = 852.180± 0.002 nm	t = 855.16 ± 0.03

These results compares favourably with those obtain elsewhere using graphical and numerical methods as presented in the table.

The X-ray diffraction pattern for the thin films shown in figure 7 indicates sharp and narrow diffraction peaks with preferential growth along 130, 101 planes followed by 011, 200, 020 and 060 planes which show that La_2O_3 was obtained. It was observed that the films also contain lanthanum oxide carbonate ($La_2O_2CO_3$) phase with a monoclinic structure (Boldish *et al.,* 1979, Forastiere et al, 2003) (JCPDS-48-1113). The X-ray diffraction pattern for the La_2O_3 thin films also indicates that the formation of lanthanum oxide carbonate has been attributed to the inclusion of large carbon residue in the films. In thermo analytical literature, $La_2O_2CO_3$ phase is reported to be stable during dynamic heating under vacuum conditions from ≈300 to ≈500^{0}C, above which its decomposition to oxide begins (Urlacher *et al.,* 1997; Urlacher and Mugnier, 1996).

In the processing of the La_2O_3 thin films, annealing was performed at 600^{o}C which is above the stability temperature for $La_2O_3CO_3$. Therefore, it is believed that the films were most likely a mixture of La_2O_3 and $La_2O_3CO_3$ as confirmed by WRS (fig. 8). A study of the stability of lanthanum oxide thin films elsewhere also arrived at similar conclusions (Kale *et al.,* 2005; Bahari *et al.,* 2011). Obviously, this carbonate phase explained, on the one hand, the relatively short propagation of the laser beam in wave guiding configuration and the high refractive index of the layer on the other hand.

The Waveguide Raman spectroscopy (WRS) result of La_2O_3 waveguide heat-treated at 600^{o}C is shown in Figure 8. WRS is a non-destructive method which provides a measure of the average parameters of the analyzed waveguide.

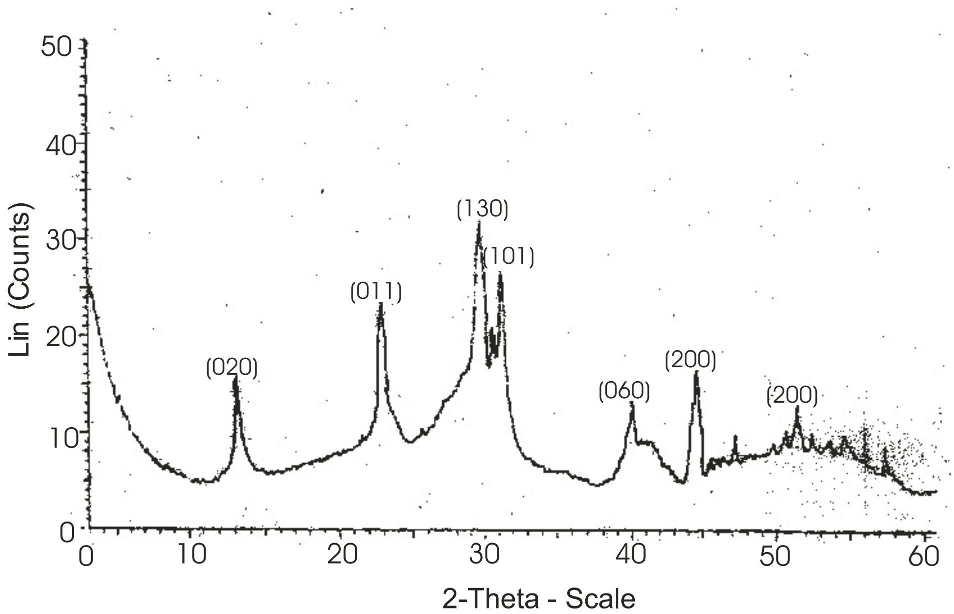

Fig. 7: X-ray diffraction spectra of La_2O_3 sol-gel thin films.

The Raman bands are located at 106, 190, 246 and 410cm^{-1}, respectively, and these peaks are due to polycrystalline La_2O_3 according to literature (Ibanga et al, 2003). This result confirms that the prominent phase La_2O_3 is consistent with the XRD patterns. The Raman frequency at 235cm^{-1}, which is not assigned to the La_2O_3 Raman scattering, proves that another phase exits.

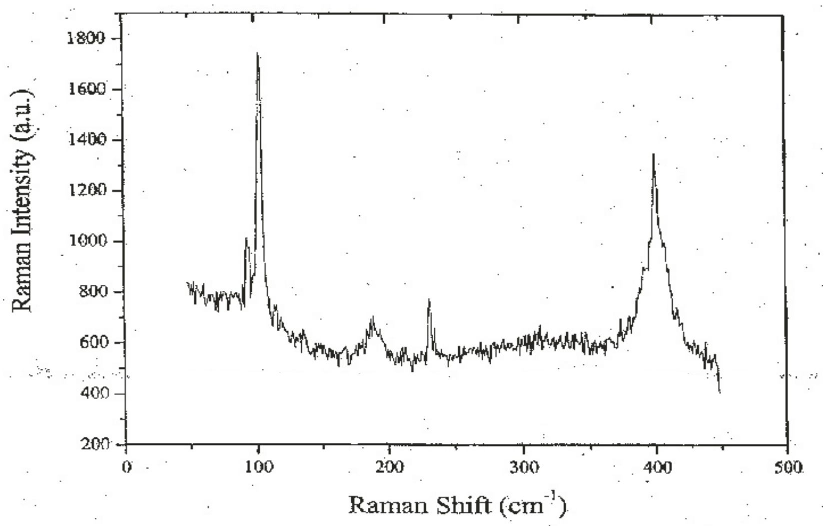

Fig. 8: Waveguide Raman spectroscopy analysis of a La_2O_3 sol-gel film excited with a 647.1nm krypton beam at room temperature.

Conclusion.

Sol-gel route has been discovered a technique that has never been used or is scarcely used for thin film deposition of La_2O_3. It is deliberately employed in this work to prepare La_2O_3 thin films exhibiting waveguiding properties suitable for optical and electrical applications.

In this study, waveguiding La_2O_3 thin films have been obtained by the Sol-gel process. These waveguides are characterized using m-line spectroscopy, spectroscopic ellipsometry, XRD, and WRS. M-line spectroscopic measurements revealed that the refractive index on Pyrex substrate is 1.592±0.001 for 543.4nm wave length and thin film thickness of 850nm and 1.589±0.001 on silicon wafer and thickness of single layer is between 40 and 60nm. Similar results were obtained for spectroscopic ellipsometry measurements.

The XRD measurement revealed that monoclinic La_2O_3 was obtained with carbon impurity in the form of Lanthanum oxide carbonate. Waveguide Raman spectroscopy experiment conducted on waveguiding samples, confirmed that, the nanocrystalline phase in the wave guide is mainly La_2O_3. It is established for the first time, that nanocrystalline Sol-Gel prepared La_2O_3 thin films could be used as planar optical waveguide.

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