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Effect of substrate temperature on microstructure and optical properties of nanocrystalline alumina thin films

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Abstract

Aluminum oxide (Al_2O_3) thin films were deposited on silicon (100) and quartz substrates by pulsed laser deposition (PLD) at an optimized oxygen partial pressure of 3.0×10^{-3} mbar in the substrate temperatures range 300–973 K. The films were characterized by X-ray diffraction, transmission electron microscopy, atomic force microscopy, spectroscopic ellipsometry, UV–visible spectroscopy and nanoindentation. The X-ray diffraction studies showed that the films deposited at low substrate temperatures (300–673 K) were amorphous Al_2O_3, whereas those deposited at higher temperatures (≥773 K) were polycrystalline cubic γ-Al_2O_3. The transmission electron microscopy studies of the film prepared at 673 K, showed diffuse ring pattern indicating the amorphous nature of Al_2O_3. The surface morphology of the films was examined by atomic force microscopy showing dense and uniform nanostructures with increased surface roughness from 0.3 to 2.3 nm with increasing substrate temperature. The optical studies were carried out by ellipsometry in the energy range 1.5–5.5 eV and revealed that the refractive index increased from 1.69 to 1.75 (λ=632.8 nm) with increasing substrate temperature. The UV–visible spectroscopy analysis indicated higher transmittance (> 80%) for all the films. Nanoindentation studies revealed the hardness values of 20.8 and 24.7 GPa for the films prepared at 300 K and 973 K respectively.

Keywords: C. Optical properties; Thin films; Alumina; Pulsed laser deposition; X-ray diffraction

1. Introduction

Aluminum oxide (Al_2O_3) thin films possess the excellent properties such as high melting point, high hardness, medium refractive index, high transparency, low absorption, wide bandgap, high thermal conductivity, low electrical conductivity, high radiation resistance, high corrosion resistance with good chemical and thermal stability [1–3]. Hence, the Al_2O_3 thin films are used as buffer layer for silicon-on insulator devices, gate insulator for metal-oxide-semiconductor devices, metal-nitride-oxide-semiconductor and complementary metal-oxide-semiconductor devices [4–8]. Al_2O_3 thin films are used in anti-reflection coatings, water repellent coatings, organic light emitting devices and improving the adhesion of bioactive glasses and hydroxyapatite coatings for medical implants [9–14]. They also find many important applications in opto-electronics, wear and corrosion resistant coatings for cutting tools, oxygen permeation barrier coatings for turbine blades, hydrogen permeation barrier coatings for nuclear fusion reactors, bioceramic, catalysis, and sensors [15–18]. The surface properties of various materials such as glasses, metals, bioceramics and polymers can be modified using Al_2O_3

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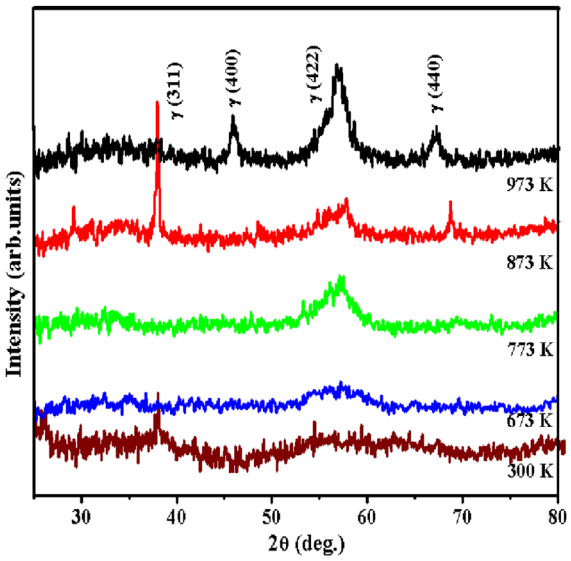

Fig. 1. XRD pattern of the Al_2O_3 films prepared at various substrate temperatures at an oxygen partial pressure of 3×10^{-3} mbar.

coatings [19]. The anti-reflection coatings are commonly used in instrument panels, displays, camera lenses, binoculars and telescopes to help produce clear and sharper images and all these applications require smooth films with stoichiometry and low defects density. It is well known that the properties are dependent on the preparation method and processing parameters. Hence it is important to investigate the correlation among the process parameters, microstructure and properties for several technological applications.

Al_2O_3 exhibits different crystallographic polymorphs such as γ, η, θ, δ and α phases with respect to temperature. Among the different phases of Al_2O_3, γ phase occurs in low temperature and find several technological applications [20]. Al_2O_3 thin films are deposited by different methods [21–25]. Among these methods, the pulsed laser deposition (PLD) is very flexible, simple, free from contamination, fast and controllable method for making high quality thin films of metals, semiconductors, insulators, polymers and biological materials. There are several reports on the preparation of Al_2O_3 thin films by PLD. However, there is no systematic study on microstructural, optical and mechanical properties of γ-Al_2O_3 films prepared as a function of substrate temperature. Hence, the present investigation deals with the preparation of Al_2O_3 thin films on Si (100) and quartz substrates as a function of substrate temperature (300–973 K) in order to understand the influence of substrate temperature on the microstructure, optical and mechanical properties of the films.

2. Experimental details

Al_2O_3 (99.99% purity) powder was compacted into a pellet of 25 mm diameter and 3 mm thickness at a pressure of 10 MPa using a uni-axial press. The pellet was sintered at 1473 K for 6 h. PLD experiments were performed using a KrF excimer laser (λ=248 nm) and the other deposition parameters are given elsewhere [23]. The thickness of the films was measured by Dektak profilometer (DEKTAK 6M-stylus profiler). The crystallinity of films were studied in an INEL XRG-3000 X-ray diffractometer (GIXRD) using CuKα_1 radiation. HRTEM investigations were carried out on a JEOL 2000 EX II (T) operated at 200 kV. The surface morphology and root mean square (RMS) surface roughness of the films were examined by Nanoscope E (Digital instruments Inc., Model: NSE, USA) atomic force microscope (AFM) in contact mode. The optical properties were measured by a SOPRA ESVG model rotating polarizer ellipsometer in the energy range 1.5–5.5 eV for three different angles of incidence (65°, 70° and 75°). The optical properties were also measured by a UV–vis–NIR (model no:3101/PC, Shimadzu) spectrophotometer in the wavelength range 190–800 nm. The mechanical measurements were carried out by nanoindenter (CSM, Switzerland) equipped with a Berkovich diamond indenter tip.

3. Results and discussion

3.1. Microstructural characterization

3.1.1. XRD, TEM and AFM analysis

The sintered Al_2O_3 pellet (α-Al_2O_3 of hexagonal structure, a=4.75 Å, and c=12.99 Å) was used as a target for PLD [23]. Al_2O_3 thin films were deposited on Si (100) and quartz substrates by PLD in the substrate temperatures range 300–973 K. Fig. 1 shows the XRD pattern of the Al_2O_3 films and revealed that the films were amorphous at low substrate temperatures (300–673 K), and polycrystalline Al_2O_3 in the temperatures ≥773 K. The films deposited in the temperatures ≥773 K indicated (311), (400), (422) and (440) reflections, correspond to γ-Al_2O_3 of face centered cubic structure [26,27]. The peak intensities of the films increased with increasing substrate temperatures. The mean crystallite size was determined from the Scherrer formula after subtracting the instrumental broadening. The crystallite size was found to increase from 5 to 10 nm as the temperature increased from 300 K to 973 K. The phase formation depends on the preparation method and process parameters. In general, phase formation of Al_2O_3 depends strongly on the substrate temperature [28]. It was reported by Cibert et al. [24] that the films prepared by plasma enhanced chemical vapor deposition (PECVD) at room temperature were amorphous, whereas the films showed cubic γ-Al_2O_3 structure at 1073 K. Also, the γ-Al_2O_3 films prepared by the PLD technique were reported to be formed at a substrate temperature of 1063 K only [29]. Pradhan et al. [30] have deposited Al_2O_3 thin films by metal organic chemical vapor deposition as a function of temperature. The films were amorphous at the low temperature range 623–823 K and crystalline Al_2O_3 phase in the temperature range 823–1023 K. Zywitzki et al. [22] have reported the nanocrystalline γ-Al_2O_3 layers with a grain size of 12–15 nm at a substrate temperature of 973 K. Anders et al. [31] reported the α-phase Al_2O_3 by post-annealing at 1273 K for 16 h. In the present work, γ-Al_2O_3 films were obtained at a low substrate temperature of 773 K due to the optimized oxygen partial pressure

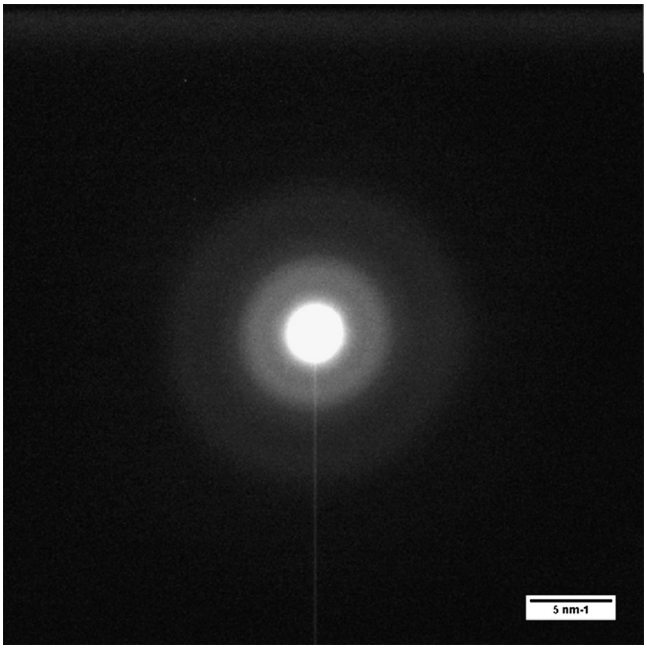

Fig. 2. SAED pattern of the Al_2O_3 film. The diffuse ring pattern indicates the amorphous nature of alumina.

(3.0×10^{-3} mbar) and higher energy (~30–100 eV) possesed by the ablated species produced from the target.

The Al_2O_3 film was also deposited on NaCl single crystal at 673 K in an optimized oxygen partial pressure of 3×10^{-3} mbar. The as-deposited film on NaCl single crystal was immersed in distilled water and the film floated on the surface was transferred to 3 mm Cu grid. The structure of the Al_2O_3 film was investigated by TEM. Fig. 2 shows the selected area of electron diffraction (SAED) pattern of the film. The diffraction pattern showed a diffuse ring pattern indicating the amorphous nature of the film. Hence, the TEM analysis confirmed the formation of amorphous Al_2O_3 film at a substrate temperature of 673 K and the result is in agreement with XRD results.

The surface morphology of the films was analyzed by AFM. Fig. 3(a–d) shows the surface morphology of the films deposited at (a) 300 K, (b) 773 K, (c) 873 K and (d) 973 K. The film indicated a few crystallites at room temperature due to very low nucleation rate and the crystallites were more at 973 K. As a consequence the RMS roughness was found to be 0.3 nm at 300 K, 0.9 nm at 773 K and 2.3 nm at 973 K. Fig. 4 shows that the surface roughness increased with increasing substrate temperature. The increase in atomic mobility with increasing substrate temperature, enables thermodynamically favored grains to grow, resulting in increase of surface roughness [32,33].

3.2. Optical characterization

3.2.1. Spectroscopic ellipsometer

The changes in amplitude ($\tan \psi$) and phase difference ($\cos \Delta$) between the parallel (r_p) and perpendicular (r_s) components of the reflected light polarized with respect to the plane of incidence are measured from the ellipsometry. The optical pseudo-dielectric function, $\varepsilon(E)$, was deduced from the ellipsometric parameters ($\tan \psi$ and $\cos \Delta$) [23,34]. The real and imaginary parts of the pseudo -dielectric functions ε_1 and ε_2 were computed from the experimentally measured ellipsometric parameters for the films prepared at different substrate temperatures. A three phase model (ambient/Al_2O_3/c-Si) was assumed to evaluate the thickness and refractive indices of the films. A linear regression analysis was carried out, until the mean square deviation (χ^2) between computed and experimental values was less than $\sim 10^{-3}$. To improve the quality of fit, roughness was taken into consideration. Hence, the four phase model (substrate/Al_2O_3/roughness/ambient) [35] was used to estimate the surface roughness, which was assumed to be a mixture of Al_2O_3 and voids on the basis of Bruggemen effective medium approximation (BEMA) [36]. The experimental and calculated ellipsometric parameters ($\tan \psi$ and $\cos \Delta$) of a representative film prepared at 773 K are shown in Fig. 5(a, b). The thickness and refractive indices of films were extracted from BEMA model and given in the present work. The complex refractive index, N ($N=n+ik$) of the films was extracted using a Cauchy dispersion model given by

$$N(\lambda) = A + (B/\lambda^2) + (C/\lambda^4)$$

where n is the refractive index; k is the absorption; A, B and C are the Cauchy parameters; and λ is the incident wavelength. The A parameter is very dominant compared to B and C parameters. That is why, only A parameter is reported in Table 1. The typical values of B and C in our case are $\sim 1.9 \times 10^{-3}$ and 2.8×10^{-4} respectively. Fig. 6 shows the variation of refractive index as a function of photon energy. The refractive indices were found to increase from 1.698 to 1.749 (at 632.8 nm) as the temperature increased from 300 K to 973 K. For all the films, the refractive indices increased with increasing photon energy. The refractive indices of the films increased with increasing substrate temperature and in accordance with the trends reported in the literature [37,38]. At higher substrate temperatures, the adatom mobility is higher compared to those at low substrate temperatures. The higher mobility causes formation of films with increased crystallite size, reduced porosity and defects. These beneficial factors could produce films of higher packing density and hence its refractive index. No absorption (k=0) was observed in all the films deposited at different substrate temperatures.

3.2.2. UV–visible spectrophotometer

The transmittance of Al_2O_3 films was measured over the range 190–800 nm wavelength using UV–visible spectrophotometer. The transmittance spectra of Al_2O_3 films deposited at different temperatures are shown in Fig.7. The oscillations in the spectra indicating the interference of light between the film and substrate. The UV–visible spectroscopy studies showed that all the films had transmittance $> 80\%$. The refractive indices were calculated using the

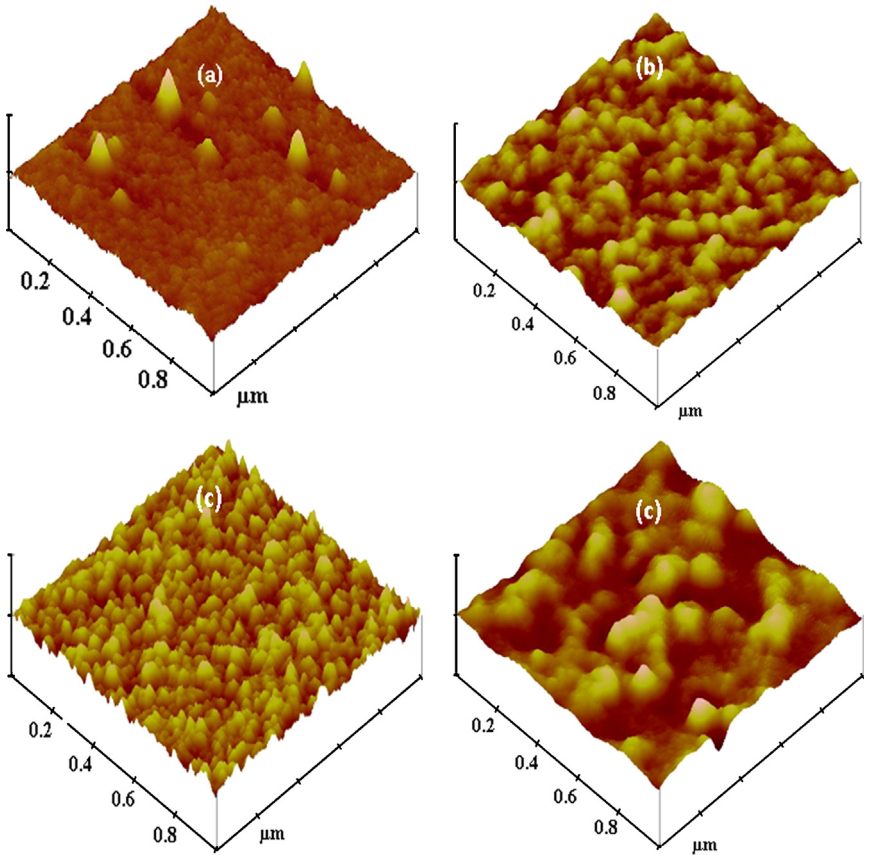

Fig. 3. AFM images ($1 \times 1\ \mu m^2$) of the Al_2O_3 thin films deposited at (a) 300 K, (b) 773 K, (c) 873 K and (d) 973 K.

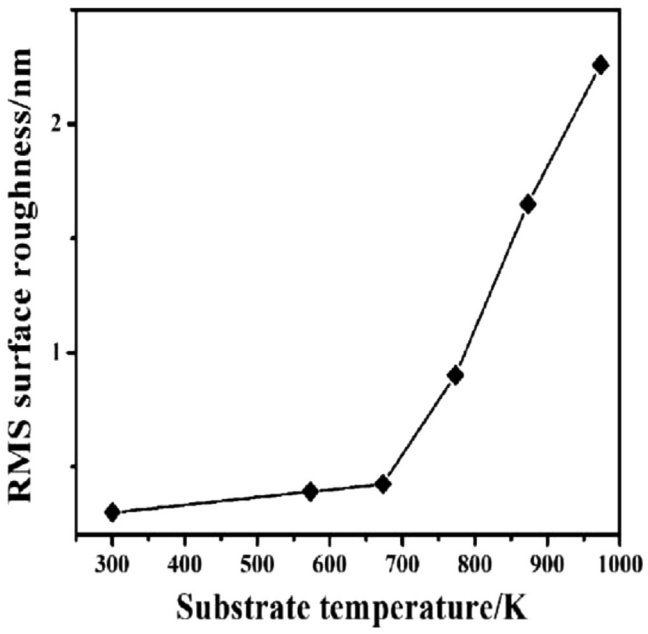

Fig. 4. The plot of RMS roughness of the Al_2O_3 films versus substrate temperature.

transmittance spectra [33,39]. The study showed that the refractive indices increased from 1.65 to 1.80 (at 632.8 nm) as the temperature increased from 300 K to 973 K [24]. The refractive indices of the films increased with increasing substrate temperature (Fig. 7 inset) [32,33]. The increase in refractive indices could be due to an increase of films density. The extinction coefficients (k) for the films were calculated and found to be 0.0165 and 0.00484 for the films deposited at 300 K and 773 K, respectively and then the k value decreased to 0.0044 at 973 K. Thus, it was found that the extinction coefficient decreased with increasing substrate temperature. There are several reports on the influence of deposition temperature on the refractive índices of the films. Lin et al. [17] deposited Al_2O_3 thin films on glass and silicon substrates by RF plasma enhanced chemical vapor deposition and refractive index of 1.61–1.73 was obtained. Koski et al. [40] measured the refractive indices and found to be in the range 1.52–1.83 with a hardness of 7–12 GPa [41,42]. Amorphous Al_2O_3 films were prepared on glass and silicon substrates by low-pressure metal-organic chemical vapor deposition and showed refractive index values in the range 1.60–1.74 [43]. Al_2O_3 thin films were deposited on Si (100) by spray pyrolysis in the substrate temperature range 723–923 K and found the refractive index of 1.66 (at 630 nm) from the ellipsometric measurements. Al_2O_3 films were produced by oxygen-ion assisted deposition as a function of substrate temperature (300–573 K). The as-deposited films were amorphous and the maximum refractive index of 1.73 was obtained for the film deposited at 573 K. The films deposited at elevated substrate temperatures were more

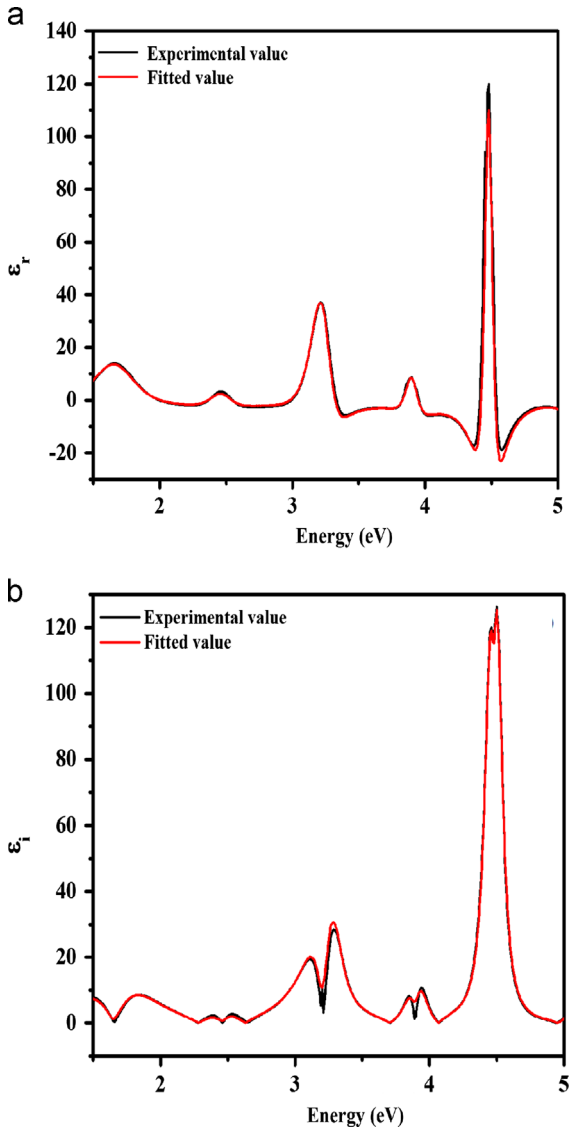

Fig. 5. Experimental and fitted ellipsometric parameters for a representative Al_2O_3 film deposited at 773 K, (a) real part of dielectric function versus photon energy and (b) imaginary part of the dielectric function versus photon energy.

Table 1
Ellipsometry measurements of thickness and refractive index of the Al_2O_3 films.

Temperature (K)	A parameter	Thickness (nm)	Refractive index @ 632.8 nm Ellipsometer
300	1.66	270	1.698
673	1.67	269	1.718
773	1.72	258	1.735
873	1.73	279	1.749
973	1.74	260	1.749

stable with high refractive index and low extinction coefficient [44]. In the present work, the refractive indices and extinction coefficients values obtained from both ellipsometry and UV–visible studies are nearly same. Therefore, it is clear that the variation in the refractive indices quoted in the literature and in the present work is due to the differences in the microstructure of the films prepared by the different deposition methods and process parameters [3,6,9,11].

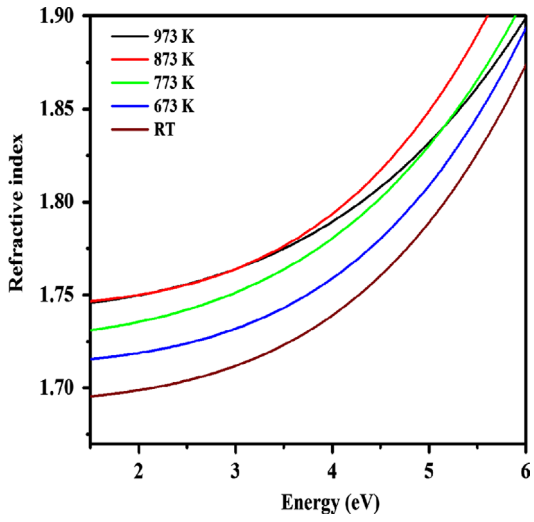

Fig. 6. Refractive index versus photon energy (eV) of the Al_2O_3 films deposited at different substrate temperatures.

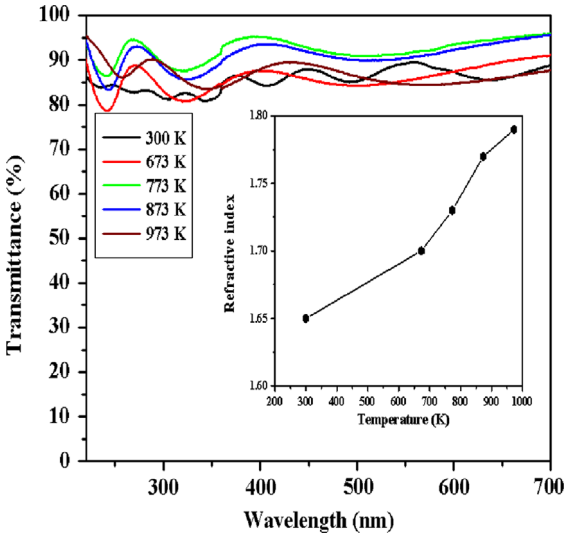

Fig. 7. Transmittance versus wavelength of the deposited films on quartz substrate at various temperatures. Inset shows the refractive index (at 632.8 nm) versus substrate temperature.

3.3. Nanoindentation studies

Nanoindentation measurements were carried out to find the hardness and elastic modulus of Al_2O_3 films (~1 μm thickness) deposited at 300 K and 973 K temperatures. Fig. 8 shows the typical load-depth curve for finding the hardness and Young's modulus of the film prepared at 973 K. The hardness of the γ-Al_2O_3 films was found to be 20.8 GPa and 24.7 GPa for the films deposited at 300 and 973 K respectively. The elastic modulus of the films were found to be 320 and 360 GPa for the above temperatures [40–42]. The increased hardness of the films could be correlated with higher deposition temperature (higher

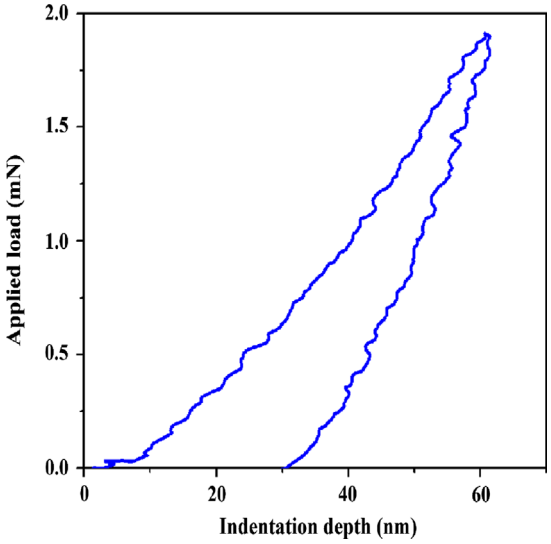

Fig. 8. Load versus indentation depth curve of γ-Al_2O_3 film prepared at 973 K.

mobility of ad-atoms) causes higher packing density of the films. Schutze et al. [45] deposited 2 μm γ-Al_2O_3 film by reactive sputtering and nanoindentation studies showed the hardness and elastic modulus of ~25 GPa and 350 GPa respectively. Zhao et al. [46] deposited the Al_2O_3 films on Si (100) and quartz substrates at 673 K by filtered cathodic vacuum arc system and found the hardness of 7.6 GPa. The refractive index and hardness of the films increased with substrate temperature at optimized conditions, which makes films for potential applications in optical, hard and wear-resistant coatings [8,11]. Therefore, the microstructural factors play a decisive role in influencing the optical and mechanical properties of the films [3,6,9,11].

4. Conclusions

The Al_2O_3 films were deposited on Si (100) and quartz substrates in the substrate temperature range 300–973 K by PLD. The XRD studies indicated the amorphous nature of the films at low substrate temperatures (300–673 K) and cubic γ-Al_2O_3 phase at higher substrate temperatures (≥773 K). The TEM investigation confirmed the amorphous nature of the Al_2O_3 film prepared at a substrate temperature of 673 K. The AFM investigation showed the smooth morphology of the films and RMS surface roughness increased from 0.3 to 2.3 nm with increasing substrate temperature. The ellipsometry and UV–visible spectroscopy analysis revealed that the refractive index values increased from 1.698 to 1.749 and 1.65 to 1.80 (at 632.8 nm), respectively as the temperature increased from 300 to 973 K. The hardness values of 20.8 GPa and 24.7 GPa were obtained for the amorphous (300 K) and γ-Al_2O_3 films (973 K), respectively. The variation in the optical and mechanical properties is correlated with the changes in the microstructural features of the films prepared as a function of substrate temperature.

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