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Evolution of optical properties with deposition time of silicon nitride and diamond-like carbon films deposited by radio-frequency plasma-enhanced chemical vapor deposition method

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The paper presents investigations of the optical properties of thin high-refractive-index silicon nitride (SiN_x) and diamond-like carbon (DLC) films deposited by the radio-frequency plasma-enhanced chemical vapor deposition method for applications in tuning the functional properties of optical devices working in the infrared spectral range, e.g., optical sensors, filters or resonators. The deposition technique offers the ability to control the film's optical properties and thickness on the nanometer scale. We obtained thin, high-refractive-index films of both types at deposition temperatures below 350 °C, which is acceptable under the thermal budget of most optical devices. In the case of SiN_x films, it was found that for short deposition processes (up to 5 min long) the refractive index of the film increases in parallel with its thickness (up to 50 nm), while for longer processes the refractive index becomes almost constant. For DLC films, the effect of refractive index increase was observed up to 220 nm in film thickness.

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1. Introduction

Plasma-deposited silicon nitride (SiN_x) and diamond-like carbon (DLC) thin films already enjoy wide application in both electronic and optical systems [1-4]. These amorphous films exhibit good (DLC) or excellent (SiN_x) adhesion to Si and SiO_2, which makes them suitable materials for silicon-based devices. In complementary metal-oxide semiconductor technology, the films typically play the role of a thin gate dielectric (usually less than 100 nm in thickness) for field-effect transistors including ion-sensitive field-effect transistors, or in their thicker form (typically 750 nm) are often employed as a passivation layer for a variety of integrated devices [2,5,6]. For both passivation and gate dielectric purposes, the films provide a good (SiN_x) or excellent (DLC) diffusion barrier against water molecules and sodium ions, two major sources of corrosion and instability in microelectronics [7,8]. Moreover, both materials exhibit very good chemical stability and inertness, qualities which are important in the design of reliable biochemical and biomedical devices. Together with high chemical stability, silicon nitride films show high values for hardness (~19 GPa) and for Young's modulus (~150 GPa), values that are respectively 2–5 and 3 times higher than those of SiO_2[9]. For their part, DLC films are considered one of the best wear-resistant materials, exhibiting hardness of over 20 GPa [10]. Due to good mechanical properties, both films are often used as a masking layer during the fabrication of integrated devices [11,12]. The combination of high hardness and high refractive index allows for application of these thin films (ranging in thickness from 50 to 200 nm, depending on their refractive index [13]) as an optimum, single-layer antireflecting and protective coating for silicon solar cells [5,14,15].

In addition to their use in microelectronics, silicon nitride and diamond-like carbon films have been applied in the fabrication of various types of optical waveguides and planar optical systems [10,16-18]. For all of the optical applications, the thickness and optical properties of the films play a critical role in ensuring the proper functioning of the device. The SiN_x films have a high refractive index in the infrared spectral range, which can be adjusted from that of Si_3N_4 ($n = 2$) to that of amorphous silicon ($n = 3.5$). In the case of DLC films, the refractive index varies with the hydrogen content and the sp^2/sp^3 ratio, exhibiting values from 1.8 to 2.2 in the infrared spectral range. Moreover, both films show very low optical absorption in the infrared and in the case of SiN_x in the visible spectral range as well. Due to these excellent optical properties, waveguides based on the films have transmission losses as low as 0.1 and 0.3 dB/cm for SiN_x and DLC films respectively [16,17]. The thickness for this type of application may be as much as 200 nm. A free-standing silicon nitride

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membrane of the same thickness has also been considered as a substrate in the design of photonic devices [7,19].

Recently some modern applications have been presented involving thin SiN_x and DLC films. The nanocoating significantly modifies the conditions of propagation for guided modes. Thus Park et al. [11] have shown that thin silicon nitride film measuring in the tens of nanometers can adjust the resonance wavelength of ring resonators. Furthermore, the sensitivity of optical fiber sensors within a specified range of the external refractive index can be precisely adjusted by appropriate choice of the thickness and optical properties of the coatings [20-22]. In previous articles, we have reported sensitivity tuning of optical fiber sensors using thin DLC films [23,24]. It can be concluded that for these applications in an ideal case, the coating should maintain its optical properties regardless of its thickness.

Deposition of SiN_x and DLC films can be achieved using one of the well established for standard silicon-integrated-circuit plasma-enhanced chemical vapor deposition (PECVD) methods. These methods offer the possibility of low-cost fabrication and high efficiency. The growth of the film is due to activation of the gas-phase precursors in a glow-discharge (plasma) environment. The chemical reactions activated by the plasma take place over the substrate as well as at the substrate. Using the PECVD system, one can deposit uniform films even on three-dimensional objects [24,25]. The materials can also be deposited as graded-refractive-index films or as a stack of nanofilms each with different properties. PECVD methods currently used include Low Pressure (LP), Radio Frequency (RF, f = 13.56 MHz) and Low Frequency (LF, f = 380 kHz) PECVD. The RF and LF PECVD methods are more attractive as processes for fabricating optical devices because they operate at lower temperatures (200 °C to 400 °C) than LP PECVD which requires temperatures in the 700° to 800 °C range [26].

In this work, we focused on investigations of SiN_x and DLC thin films deposited by RF PECVD for application in tuning the functional properties of optical devices such as optical sensors, filters and resonators. Both films showed very attractive properties from the point of view of optical applications. Our aim was to achieve thin (<100 nm), high-refractive-index SiN_x films at a substrate temperature below 350 °C, which is acceptable for the thermal budget of most of optical devices. Moreover we deposited a series of slightly thicker (up to 350 nm), high-refractive-index DLC films, where substrate temperature was significantly lower (20 °C).

2. Experimental details

The films were prepared using different plasma setups working at f = 13.56 MHz. Since for most of the applications of silicon nitride films SiO_2 or oxidized Si substrates are used, the films were deposited on oxidized p-type <111> silicon wafers (ρ = 0.005–0.02 Ω cm) using Plasmalab 80+ (Oxford Plasma Technology) [5]. We employed a high $[SiH_4:N_2]/[NH_3]$ flow ratio (2% SiH_4 diluted in N_2) in order to obtain high-refractive-index SiN_x films. DLC films, on the other hand, were deposited on oxidized p-type <100> silicon wafers (ρ = 6–8 Ω cm) using a home-made RF plasma system [27] equipped with a water-cooled electrode. In the setup, instead of setting RF power, we monitored negative self-bias voltage, which is a characteristic parameter of RF PECVD. We used methane (CH_4) as a carbon film gas precursor. The deposition parameters of the DLC films were optimized in terms of adhesion of the films to oxidized-silicon substrate. The deposition parameters for both types of films are given in Table 1.

Before plasma deposition and oxidation, all samples were cleaned according to the common practice in microelectronics RCA procedures. The wet oxidation process was performed in a furnace heated to 1000 °C. The oxidized wafers were then cooled down and used directly for deposition of both types of films.

Film parameters such as the index of refraction (n), the extinction coefficient (k) and the thickness were determined by a Horiba Jobin-Yvon UVISEL spectroscopic ellipsometer operating in the wavelength range from 250 nm to 2050 nm with the increment of 20 nm. The equipment was calibrated before each measurement. To fit the measurement data to a physical model, a structure containing a first SiO_2 layer covering the silicon substrate and a second SiN_x or DLC layer was applied and fitted with $\chi^2 < 1$. The SiN_x and DLC films were modeled using the single-layer Tauc–Lorentz dispersion formula [13]. In order to minimize error coming from precision of angle of incidence, which was initially set to 70°, the angle of incidence in addition to the other parameters was determined using the fitting procedure. Thickness of the SiO_2 film after the oxidation process was determined to be in range of 385 nm to 400 nm, and confirmed to be in the same range after the deposition.

Table 1
Parameters of SiN_x and DLC films deposition by the RF PECVD method.

	SiN_x	DLC
RF Power [W]	15	
Negative self-bias voltage [V]		500
Gas precursors	$(SiH_4:N_2)/(NH_3)$	CH_4
Gas flow [ml/min]	285/15	50
Pressure [Pa]	53	70
Deposition time [min]	1 to 10	1 to 7
Substrate temperature [°C]	300 to 340	20

3. Results and discussion

3.1. Silicon nitride films

As can be seen from Fig. 1, the optical properties of SiN_x films depend on both temperature and deposition time. The influence of substrate temperature on the refractive index of the films is greater when films are deposited in processes of shorter duration (Fig. 1a). The obtained films show significant absorption in the UV spectral range, which increases with deposition time (Fig. 1b). For films with shorter deposition times, the extinction coefficient has higher values in the visible spectral range. However, in the infrared range the extinction coefficient is negligible for all the measured films.

The dependence between the refractive index (measured at λ = 1560 nm) and the substrate temperature for different deposition times is shown in Fig. 2. Increases in temperature clearly correspond with increases in the refractive index. However, even when the substrate temperature is at the higher end of the investigated temperature range, e.g., 335 °C for the 1 min-long process, the refractive index is still highly dependent on the duration of the deposition procedure. The temperature increase cannot compensate for that effect. It can be clearly seen in Fig. 3, that the refractive index increase with film thickness up to 50 nm is approximately $5 \cdot 10^{-3}$ refractive index unit per nm (RIU/nm). For greater film thickness, the refractive index can be assumed to be deposition-time-independent and reaches 2.4 (at λ = 1560 nm). Fig. 3 also shows that the SiN_x films were deposited at a rate of 9.25 nm/min, which is low enough for good control of thickness of the films for optical applications. In contrast to the influence on optical properties, it can be seen that substrate temperature in the investigated range has very little effect on the thickness of the films.

It was shown by number of authors that the optical properties of plasma-deposited SiN_x films can be adjusted over a wide range by varying the process parameters. Typically, an increase in the refractive index can be achieved by increasing the SiH_4/NH_3 flow ratio [13,28]. Increasing the flow ratio will also reduce film roughness [28], which is beneficial from the perspective of optical applications. Moreover, it is possible to increase the pressure in the plasma chamber (up to 100 Pa), resulting in an increase of both the refractive index and the deposition rate [28].

In our experiment, we altered just two factors, substrate temperature and deposition time. The influence of substrate temperature on

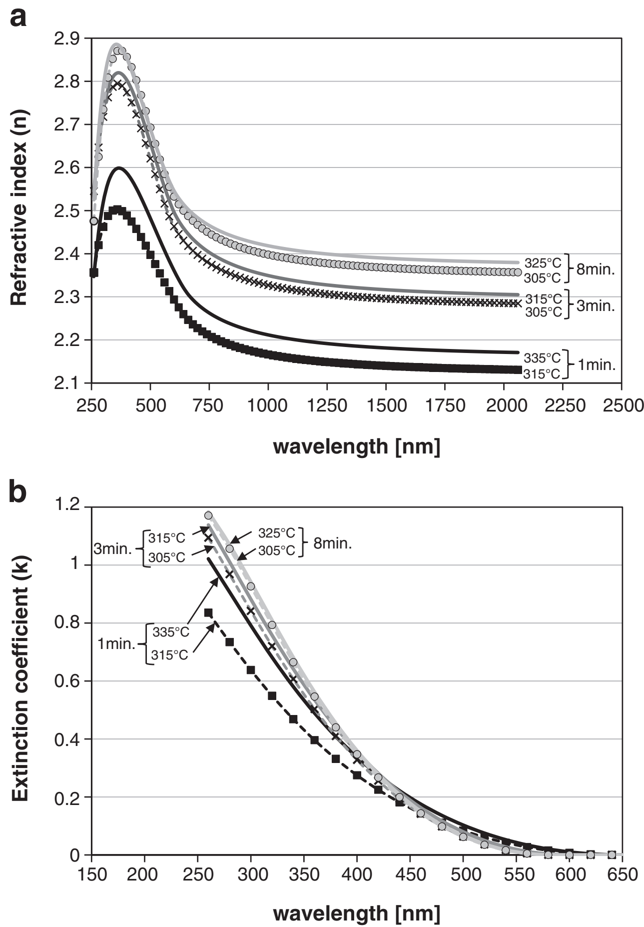

Fig. 1. Dispersion of (a) refractive index and (b) extinction coefficient of SiN_x films deposited in processes lasting 1, 3 and 8 min. Temperature of the substrate during the process is given next to each line.

the properties of SiN_x films has been already investigated by a number of authors [e.g., [5,29,30]]. Suchaneck et al. [31] showed that at temperatures below 150 °C, a polymer-like film, rich in hydrogen, could be grown. The growth of good quality SiN_x films by the RF PECVD method takes place at around 350 °C [1]. According to our experimental results, an increase in the substrate temperature in the

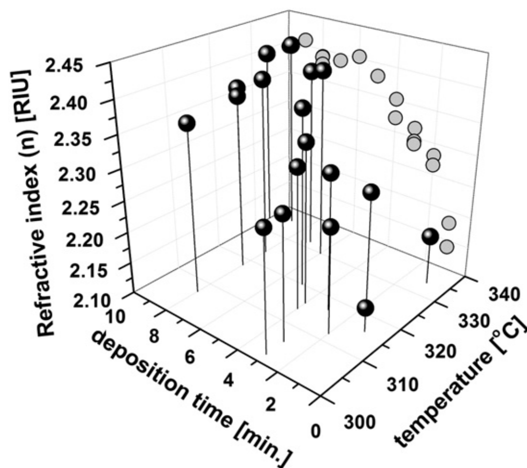

Fig. 2. Refractive index (at λ = 1560 nm) of SiN_x films deposited with various process durations and substrate temperatures. The gray points represent the variation of the refractive index as a function of deposition time for all tested temperatures.

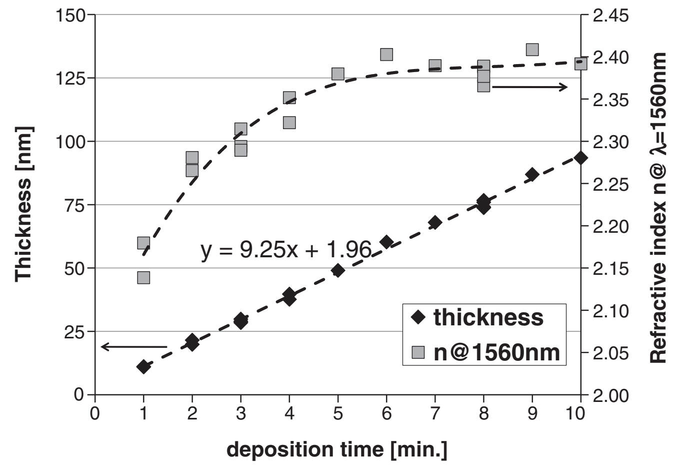

Fig. 3. Thickness and refractive index (at λ = 1560 nm) of SiN_x films deposited with various process durations. The data presented for the same deposition time correspond to a different substrate temperature given in Fig. 2.

investigated range (300 °C to 340 °C) causes an increase in both the refractive index and the extinction coefficient in the whole investigated spectral range. Two phenomena can be responsible for this behavior: changes in the films' hydrogen content and the formation of silicon nanocrystals.

Yen et al. [32] noticed that plasma-induced decomposition of SiH_4 and NH_3 at lower temperatures (300 °C to 450 °C) results in nitride films that contain a significant amount of hydrogen. Studies on SiN_x:H have shown that the properties of such films critically depend on the H content, which in turn is influenced by the deposition temperature. The reduction of hydrogen content can be seen as an increase in the refractive index [32]. This phenomenon was observed in our experiment. Moreover, the content of covalently bonded hydrogen has an impact on the optical absorption of SiN_x films. For silicon-rich nitrides, higher temperature promotes replacement of Si–H bonding with Si–Si bonding and increase of absorption [10]. As shown in our experiment, the increase of the extinction coefficient with temperature can be then attributed to the release of hydrogen.

The formation of nanocrystals in the film also influences the relation between optical properties and deposition temperature. Low deposition-temperature and low energy conditions promote a columnar structure of the film, with the result that more energy must be brought to the surface in order to achieve higher film density [10]. In the case of silicon-rich films, the increase in density can promote the formation of silicon nanocrystals [26]. The density stays in correlation with the refractive index. The effect of increase in the refractive index with increasing temperature is clearly visible in our experimental results. According to Gorin et al. [26], the silicon nitride films deposited by RF PECVD increase both their optical absorption and their refractive index due to an increase in the number and size of silicon nanocrystals in the silicon nitride matrix.

The dependence between the optical properties of SiN_x films and their thickness correlated with deposition time must be also discussed. An increase in refractive index with the thickness of plasma-deposited carbon-doped SiO_2 films was previously observed [33]. For these films, the increase takes place up to 400 nm with a ratio below $0.2 \cdot 10^{-3}$ RIU/nm, where the refractive index starts to be constant with the thickness. The tendency seems to be independent of the deposition method, since it has been also observed for spin-coated and sintered polytetrafluoroethylene (PTFE) films [34]. In that case, an increase in thickness was followed by an increase in the refractive index, up to a thickness of 1100 nm at a rate of $0.03 \cdot 10^{-3}$ RIU/nm. It should be emphasized that the constant value of the refractive index for carbon-doped SiO_2 and PTFE was relatively low, reaching only 1.47 and 1.37, respectively.

Like the effect obtained when the temperature was increased, the increase in deposition time can induce an increase in film density, i.e., by the formation of silicon nanocrystals, and a decrease of hydrogen content. According to Kessels et al. [35], the growth mechanism of SiN_x films is based on SiH_3 radicals forming an Si:H-like surface layer that is simultaneously nitridated by the N replacing H radicals and converting the surface layer into SiN_x:H. For short processes that result in films with a lower refractive index, the hydrogen content is higher than in the case of films deposited in longer processes. A reduction of the extinction coefficient of the films in the visible spectral range is evident with deposition time, which can be caused by reduction of hydrogen content and replacement of Si–H bonds with Si–N ones. On the other hand, the film densifies with deposition time and the densification process favors the formation of Si nanocrystals. Both effects, the release of hydrogen and film densification, result in an increase in the refractive index and a decrease in the extinction coefficient in the visible spectral range.

3.2. Diamond-like carbon films

Fig. 4 shows the dispersion of the optical properties of DLC films deposited in processes of various durations. Compared to the SiN_x films discussed above, DLC exhibits lower absorption in the UV range, and higher absorption in the near infrared spectral range. As with the SiN_x films, the dependence between optical properties and the deposition process duration is evident in the case of carbon films (Fig. 5). The refractive index increases with thickness up to a thickness of 220 nm with a ratio of approximately $0.3 \cdot 10^{-3}$ RIU/nm. Over that thickness, the refractive index at λ = 1560 nm can be assumed to be deposition-time-independent and reaches 2.07. When the thickness exceeds 300 nm, a rapid decrease is evident in the refractive index of the films. It is also shown (Fig. 5) that the DLC films were deposited with deposition ratio of 40.7 nm/min, which is low enough for good control of thickness of the films for optical applications. Moreover, the evolution of optical properties with deposition time does not affect the deposition rate in the investigated range of thickness.

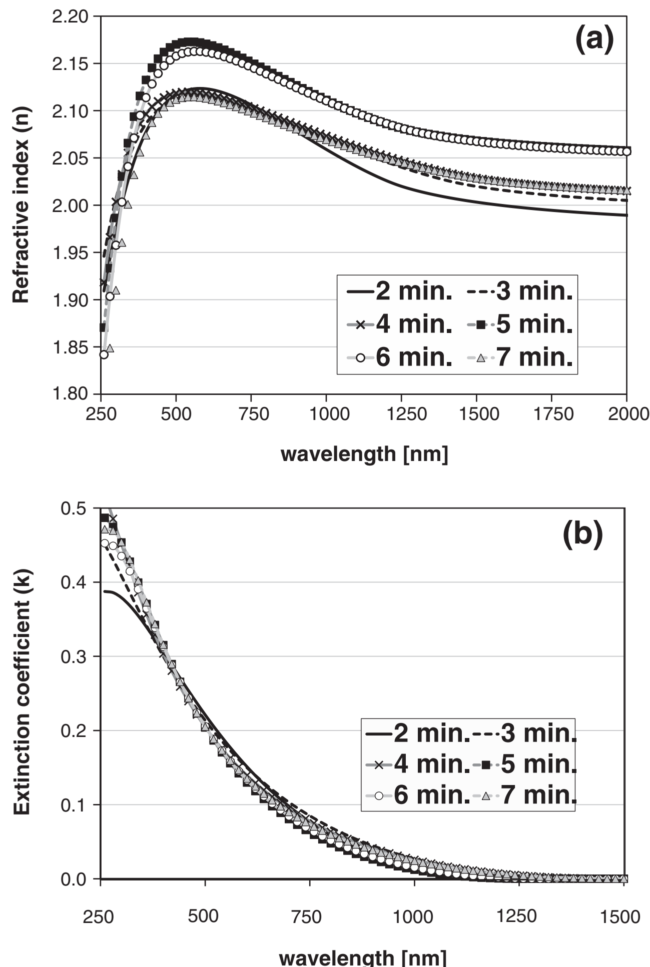

Fig. 4. Dispersion of (a) refractive index and (b) extinction coefficient of DLC films deposited in processes of various durations.

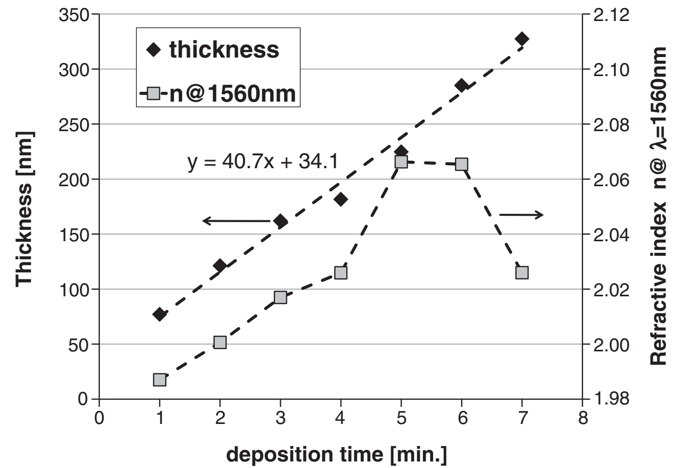

Fig. 5. Thickness and refractive index (at λ = 1560 nm) of DLC films deposited with various process durations.

According to Robertson [3], DLC is a metastable form of amorphous carbon (a-C) or hydrogenated amorphous carbon (a-C:H) that has a significant content of sp^3 bonds typical of diamonds. The influence of various parameters of the deposition process on the properties of DLC films is widely reported in the literature of plasma CVD methods [3,4]. Typically, authors investigate the influence of deposition parameters on the thickness and mechanical properties of the films [11,36,37]. The relation between self-bias voltage (V_b) and both the thickness and the optical properties of DLC films measured in the visible spectral range (λ = 600 nm) has been investigated and discussed in our previous work [38]. According to the subplantation DLC growth model [3], when the ions are in a higher energy state (typically $V_b = -300$ to -500 V), they not only stick to the film surface, but can penetrate it and enter the subsurface interstitial state, increasing local density and converting to sp^3 bonding. At that voltage, the films exhibit a typical diamond-like character.

The optical properties of DLC film strictly depend on the phase composition of the film, i.e., the sp^2/sp^3 ratio and the hydrogen content. The increase in sp^3 content correlates well with an increase in the refractive index and the hardness of the DLC film [37,39]. We believe that the densification is the main phenomenon responsible for the increase in refractive index with increasing deposition time. Lemoine et al. [36] found that the hardness of DLC films increases with their thickness, which correlates well with our finding. However the densification phenomenon of DLC is limited by film thickness — in our case up to 220 nm, after which point the refractive index starts to decrease as the film thickness continues to increase. This effect may be caused by an increase in substrate temperature. Despite the precaution of cooling the sample, we believe that there must be a significant increase of temperature at the surface of the film induced by ions constantly hitting the surface. Once a certain length of time has passed (in our case equal to 5 min), the sample cooling is not effective enough. At higher temperatures, hydrogen is released more intensively from the film structure. The hydrogen release promotes conversion of sp^3 bonding to sp^2 one and further formation of sp^2 clusters [3]. In terms of optical properties, this phenomenon can be

seen as a decrease in the refractive index and an increase in the extinction coefficient.

4. Conclusions

In this paper we discuss the influence of deposition time on the thickness and the optical properties of SiN_x and DLC thin films which are already widely used in optical applications. Both types of film exhibit very attractive properties including low absorption, high refractive index and high hardness. Moreover, they can both be deposited in relatively low-temperature processes, which makes them suitable for a number of substrate materials. In our study, we show that the RF PECVD deposition technique has good ability to control film thickness on the nanometer scale. Our results suggest that in the initial stage of growth of both films, significant densification of the structure and variation of hydrogen content takes place, resulting in an increase of their refractive index with deposition time. The refractive index/thickness ratio at this stage increases with the refractive index for both of the investigated films, reaching as high as $5 \cdot 10^{-3}$ RIU/nm in the case of SiN_x films. A refractive index that remained constant with thickness was achieved for films thicker than 50 nm for SiN_x and 220 nm for DLC films. It is important to realize that by varying the length of the thin-film deposition process, not only do we determine the thickness of the films but we also change their optical properties by modifying their structure. This phenomenon must be taken into account in the process of designing optical applications employing these nanofilms. Nevertheless, both films seem to be suitable as nanolayers tuning the functional properties of optical devices.

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