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EFFECT OF THERMAL AND PULSE LASER ANNEALING ON PHOTOLUMINESCENCE OF CVD SILICON NITRIDE FILMS

Abstract. The light-emitting properties of Si-rich silicon nitride films deposited on the Si (100) substrate by plasma-enhanced (PECVD) and low-pressure chemical vapor deposition (LPCVD) have been investigated. In spite of the similar stoichiometry ($SiN_{1.1}$), nitride films fabricated by different techniques emit in different spectral ranges. Photoluminescence (PL) maxima lay in red (640 nm) and blue (470 nm) spectral range for the PECVD and LPCVD $SiN_{1.1}$ films, respectively. It has been shown that equilibrium furnace annealing and laser annealing by ruby laser (694 nm, 70 ns) affect PL spectra of PECVD and LPCVD $SiN_{1.1}$ in a different way. Furnace annealing at 600 °C results in a significant increase of the PL intensity of the PECVD film, while annealing of LPCVD films result only in PL quenching. It has been concluded that laser annealing is not appropriate for the PECVD film. The dominated red band in the PL spectrum of the PECVD film monotonically decreases with increasing an energy density of laser pulses from 0.45 to 1.4 J/cm^2. Besides, the ablation of PECVD nitride films is observed after irradiation by laser pulses with an energy density of > 1 J/cm^2. This effect is accompanied by an increase in blue emission attributed to the formation of a polysilicon layer under the nitride film. In contrast, the LPCVD film demonstrates the high stability to pulsed laser exposure. Besides, an increase in the PL intensity for LPCVD films is observed after irradiation by a double laser pulse (1.4 + 2 J/cm^2) which has not been achieved by furnace annealing.

Keywords: Si-rich silicon nitride, photoluminescence, furnace annealing, laser annealing, polysilicon

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ВЛИЯНИЕ ТЕРМИЧЕСКОГО И ИМПУЛЬСНОГО ЛАЗЕРНОГО ОТЖИГА НА ФОТОЛЮМИНЕСЦЕНЦИЮ CVD-ПЛЕНОК НИТРИДА КРЕМНИЯ

Аннотация. Изучены светоизлучающие свойства обогащенных кремнием пленок нитрида кремния, осажденных на кремниевые подложки Si(100) методами плазмохимического осаждения (PECVD) и газофазного химического осаждения при низком давлении (LPCVD). Несмотря на сходный стехиометрический состав (отношение Si/N), пленки нитрида кремния $SiN_{1,1}$, полученные различными способами, излучают в разных спектральных областях. Максимумы фотолюминесценции (ФЛ) лежат в красной (640 нм) и синей (470 нм) областях спектра для пленок, полученных методами PECVD и LPCVD соответственно. Печной и лазерный отжиг рубиновым лазером (694 нм, 70 нс) по-разному влияет на светоизлучающие свойства PECVD- и LPCVD-пленок нитрида кремния. Так, печной отжиг при температуре 600 °С приводит к резкому возрастанию интенсивности ФЛ для пленки, полученной методом

PECVD, тогда как печной отжиг пленки, сформированной методом LPCVD, приводит только к тушению исходного сигнала ФЛ. Напротив, лазерный отжиг не подходит для пленки, полученной плазмохимическим методом. Для данной пленки наблюдается уменьшение интенсивности доминирующей полосы в красной области с увеличением плотности энергии в лазерном импульсе от 0,45 до 1,4 Дж/см^2. Кроме того, после облучения импульсами с энергией больше 1 Дж/см^2 наблюдается абляция нитридной пленки. При этом увеличивается интенсивность свечения в синей области, природу которого мы связываем с формированием поликремния под нитридным слоем. С другой стороны, пленка, полученная методом LPCVD, демонстрирует высокую стойкость к лазерному воздействию. При этом облучение LPCVD-пленки двойным импульсом (1,4 + 2 Дж/см^2) приводит к усилению сигнала люминесценции, чего не удавалось достичь с помощью печного отжига.

Ключевые слова: обогащенный кремнием нитрид кремния, фотолюминесценция, печной отжиг, лазерный отжиг, поликремний

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Introduction. Amorphous silicon nitride films is one of the most used dielectric in current Si technology. At present, a lot of efforts is paid to expand application of this material in optoelectronics, namely, for development of Si-based light-emitting diodes. Initially, silicon nitride films with large excess of Si atoms were of great interest to synthesize Si nanocrystals which emit in the full range of visible spectrum [1–3]. Afterwards, it was reported that such emission is not related to radiative recombination in Si nanocrystals [4–5]. Nevertheless, Si-rich films with slight excess of Si atoms (<10 %) are more preferable as active layers in light-emitting structure due to better conductivity. Thereby, Si-rich silicon nitride films with ~5 % Si excess were chosen as an object of investigation in present work.

Heat treatment (furnace, rapid thermal or laser annealing) is one of the widely applicable ways to enhance light output from silicon nitride. In our earlier works [6–7] it was demonstrated that the optimal regime of annealing differs for the SiN_x films with different stoichiometry. So, a photoluminescence (PL) intensity of Si-rich silicon nitride film substantially increases after annealing at 600 °C in Ar ambient, while the PL intensity of the N-rich film increases after annealing at 800 °C in air, though to a lesser extent. The regime of effective annealing is assumed to be different for the SiN_x films deposited by different techniques. Plasma-enhanced (PECVD) and low-pressure (LPCVD) chemical vapor depositions are common techniques for the silicon nitride films deposition for different application. LPCVD process is characterized with high deposition temperatures (700−800 °C) and low deposition rate, while PECVD depositions are carried out at relatively low temperatures (~300 °C) with higher deposition rate [8]. Therefore, the different effect of annealing is expected for PECVD and LPCVD films.

Despite the fact that laser annealing is non-commonly used annealing technique, it has several advantages over furnace annealing, such as low thermal budget, ultra-fast annealing, diffusionless activation and others. Applied to silicon nitride films, laser annealing was used to crystallize amorphous silicon cluster formed inside them [9] as well as to increase a density and etch selectivity of SiN_x films [10]. In this paper, we discuss the effect of furnace and laser annealing on emission of Si-rich SiN_x films grown by PECVD and LPCVD techniques.

Experimental. Si-rich silicon nitride films were deposited on the (100)-oriented Si-substrates by PECVD and LPCVD techniques. The PECVD film was deposited using a gaseous mixture of silane (SiH_4) and nitrogen (N_2) as precursors ($SiH_4/N_2 = 1/3$) at 350 °C. The LPCVD film was deposited using silane and ammonia gas mixture ($SiH_4/NH_3 = 1/4$) at 785 °C. The thickness of both nitride films was ~250 nm according to the ellipsometry data. The samples of 1 × 1 cm^2 were cut out from the SiN_x/Si structures, and a part of them was annealed in Ar ambient at 600–1100 °C for 60 min in a resistance furnace. Pulsed laser irradiation of silicon nitride films was carried out by a ruby laser ($\lambda = 694$ nm). The laser pulse was of a nearly Gaussian temporal shape. Its full width at half maximum (FWHM) amounted to 70 ns. Pulse energy densities were ranging at 0.45–2 J/cm^2. In our experiment, the densities of the laser radiation energy in a pulse were either higher or lower than the threshold density that was required for the origination of macroscopic molten regions.

The surface morphology of as-deposited silicon nitride films as well as the laser irradiated areas was studied by optical microscope Nikon Eclipse LV150. The composition of as-deposited SiN_x films was determined by Rutherford backscattering spectrometry using 1.3 MeV He^+ ions from the HVE AN-2500 accelerator. The parameter “x” (the ratio of the numbers of N and Si atoms) is known to be 1.3 for the stoichiometric silicon nitride Si_3N_4. In our experiment, this parameter is equal to 1.1 for both types of the samples. Additionally, Raman scattering (RS) measurements were performed in backscattering geometry with a Ramanor U1000 micro-Raman spectrometer (Jobin Yvon) using a 532-nm laser beam as the excitation source. PL spectra excited by UV He–Cd laser (325 nm) were recorded at room temperature in the spectral range of 350–800 nm.

Results and discussion. It is known that physical properties are differed for PECVD and LPCVD grown silicon nitride films. LPCVD films are characterized with higher density, electrical resistivity and are dissolved by chemical etchants more slowly than PECVD ones. As our experiments revealed, laser annealing affected these two types of films differently, too. The LPCVD films sustained the laser pulses with an energy density up to 2 J/cm^2 without any change of surface morphology. At the same time, surface morphology of PECVD nitride films was changed sufficiently after laser annealing. Fig. 1 depicts the images of the surface and Raman spectra of the PECVD silicon nitride films before and after laser annealing.

One can see from Fig. 1, *a*, a partial and full ablation of PECVD films occurred after laser irradiation with pulse energy density of 1.1 and 1.4 J/cm^2, respectively (Fig. 1, *a*). Besides, the surface of silicon substrate exposed via laser ablation has a rough texture. A phase composition of this structured Si subsurface layer was investigated by Raman spectroscopy. We used $Z(XY)\bar{Z}$ polarization configuration and a sample rotation to suppress the signal from the Si (100) substrate. In the case of the samples irradiated by laser pulses with the energy densities of 0.45−0.9 J/cm^2, the Si phonon band at 520 cm^{-1} was supressed as well as for as-deposited sample. In contrast, bands with maximum at 515 and 520 cm^{-1} were exhibited in the Raman spectra from areas irradiated by pulses of 1.1 and 1.4 J/cm^2, respectiely. An appearance of these bands is the evidence of breaking selection rules due to a polysilicon layer formation. It should be noted a shift of Si line position (515 cm^{-1}) to long wave region for the area irradiated by 1.1 J/cm^2 laser pulse in comparison with the area irradiated by 1.4 J/cm^2 pulse (520 cm^{-1}). Moreover, the FWHM (9 cm^{-1}) of the Si line for the 1.1 J/cm^2-irradiated area exceeds the one for the 1.4 J/cm^2 – irradiated area. Taking it into account we can suggest that compressive stress in polycrystalline silicon layer formed after irradiation by 1.1 J/cm^2 laser pulse is higher than after irradiation by 1.4 J/cm^2 laser pulse.

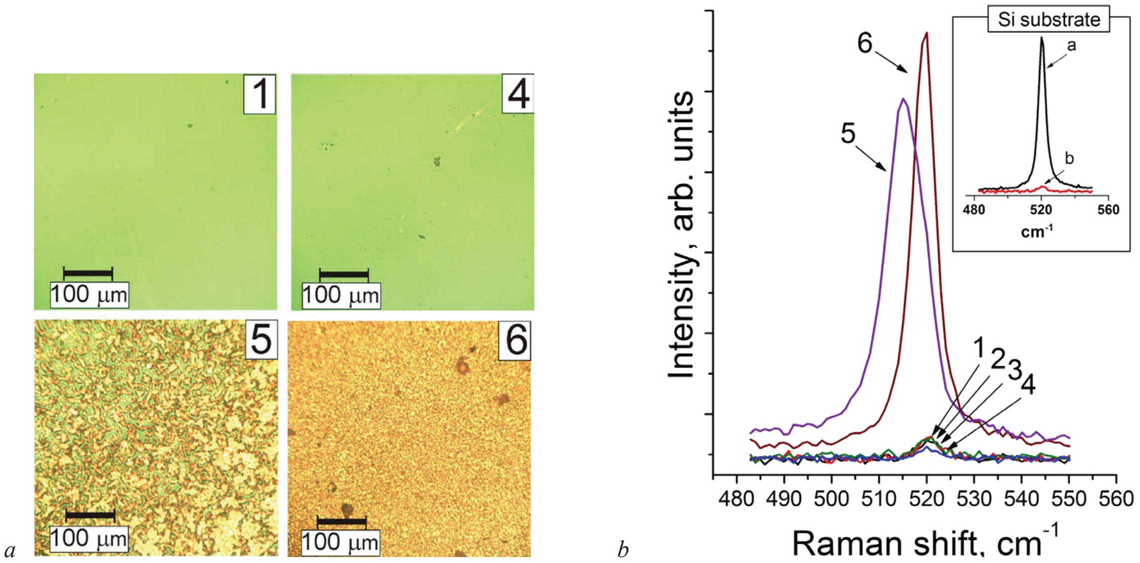


Fig. 1. Surface morphology (*a*) and Raman spectra in “forbidden” polarization geometry (*b*) of the PECVD silicon nitride film before (*1*) and after laser annealing by pulses with energy densities of 0.45 (*2*), 0.7 (*3*), 0.9 (*4*), 1.1 (*5*), 1.4 J/cm^2 (*6*). The insert in Fig. 1, *b* shows the Raman spectra of the deposited samples in “allowed” (*a*) and “forbidden” (*b*) polarization geometries

Рис. 1. Морфология поверхности (*a*) и спектры комбинационного рассеяния в «запрещенной» геометрии поляризации (*b*) PECVD-пленок нитрида кремния до (*1*) и после лазерного отжига импульсами с плотностями энергии 0,45 (*2*), 0,7 (*3*), 0,9 (*4*), 1,1 (*5*), 1,4 Дж/см^2 (*6*). На вставке (рис. 1, *b*) представлены спектры комбинационного рассеяния исходного образца при «разрешенной» (*a*) и «запрещенной» (*b*) геометрии поляризации

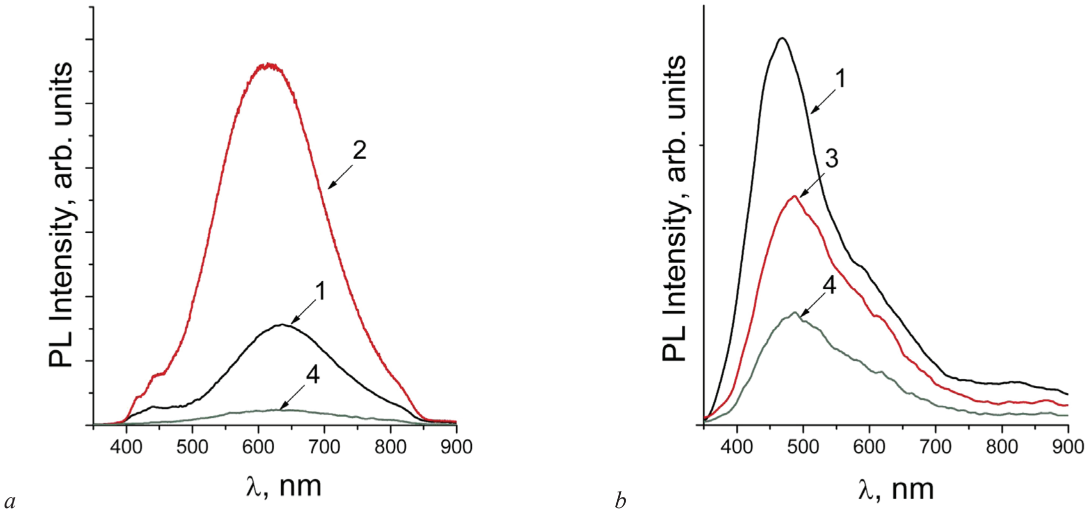


Fig. 2. PL spectra of the PECVD (*a*) and LPCVD (*b*) silicon nitride films before (*1*) and after furnace annealing at 600 (*2*), 800 (*3*) and 1100 °C (*4*)

Рис. 2. Спектры фотолюминесценции PECVD- (*a*) и LPCVD-пленок (*b*) нитрида кремния до (*1*) и после печного термического отжига при температурах 600 (*2*), 800 (*3*) и 1100 °C (*4*)

Fig. 2 shows the PL spectra of as-deposited and furnace-annealed PECVD and LPCVD silicon nitride films.

In spite of similar chemical composition for both types of films ($SiN_{1.1}$), a location of emission maxima of the as-deposited films is different. In the PL spectrum of PECVD film a broad band of complicated shape with maximum in red spectral range at ~640 nm dominates. The as-deposited LPCVD film emits in violet-green range with maximum at ~470 nm.

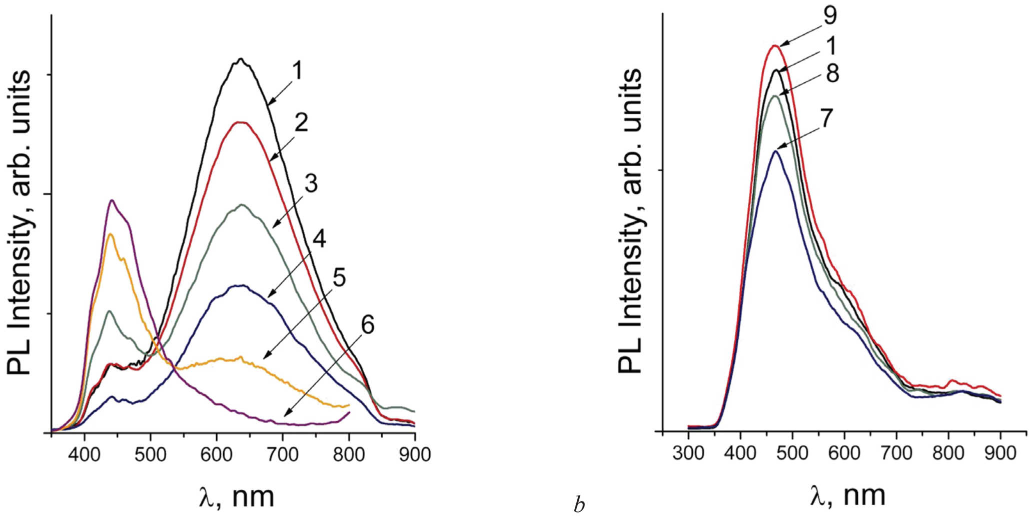
In order to improve light-emitting properties of the nitride films, furnace annealing was tested at first. For the sake of comparison with our previous results on furnace annealing [7], the annealing temperature was chosen 600 °C and 1100 °C for the PECVD-samples. Taking into account a high deposition temperature of LPCVD-nitride film, in this case annealing was carried out at 800 °C and at 1100 °C. As can be seen from Fig. 2, the effect of furnace annealing on light-emitting properties differs for PECVD and LPCVD films. PECVD film annealing at 600 °C results in significant (3.5 times) increase of PL intensity, while annealing at high temperature (1100 °C) quenches an initial emission. In the case of LPCVD films, annealing results in emission suppression for two tested temperatures. Moreover, PL intensity of the LPCVD film decreases with annealing temperature increasing. Different positions of PL maxima for as-deposited films can be explained by structural peculiarities PECVD and LPCVD nitrides. Due to higher deposition temperature, the LPCVD films are characterized with higher density and lower concentration of hydrogen than PECVD films [8]. Bonding distortions and/or compositional fluctuations induce a continuum of band-tail states around the mobility edges. These band tail states play a crucial role in radiative recombination of amorphous Si-rich silicon nitride films [4–5]. The presence of tails of the density of states results in effective narrowing of optical band gap. It is obvious that the PECVD $SiN_{1.1}$ films should exhibit a more significant tailing than LPCVD ones due to a more disordered structure. In turn, band tail states result in the broadening of PL spectrum and its maximum red shift. Thus, various levels of structural inhomogeneity are the reason of difference in PL spectra for the as-deposited PECVD and LPCVD films.

As has been mentioned above, PECVD films are deposited at relatively low temperatures and contain a large amount of hydrogen (~20 %) in a form of Si–H and N–H bonds. Furnace annealing at 600 °C results in breaking these bonds, hydrogen evaporation and a formation of Si-dangling bonds (K-centers). These K-centers take part in radiative recombination. Thus, we can conclude that the increase of PL intensity of PECVD films after annealing at 600 °C is due to the Si–H bonds dissociation and the formation of K-centres. Then, the PL quenching after annealing at 1100 °C can be explained by a reduction of K-center concentration due to formation of Si–N bonds via connection of Si and N dangling bonds during high-temperature treatment. In addition, the decrease of structural disorder after annealing leads to the reduction of the band tail states density. LPCVD films are deposited at elevated temperatures (700–800) °C and initially characterized with a better structural order and low concentration of Si–H bonds (<8 %). Therefore, we could expect that LPCVD film annealing results in further structure ordering only, and the effect of K-centers formation/dissociation is negligible in this case. It is known that crystal silicon nitride does not exhibit any light-emitting properties. Hence, one can expect that the improvement of film perfection should result in the PL intensity decrease. This approach allows us to explain a negative effect of furnace annealing on light emission of LPCVD films in our experiment.

Fig. 3 shows PL spectra of the PECVD and LPCVD as-deposited films before and after laser irradiation. In the case of PECVD film, laser annealing leads to PL quenching. Besides, the intensity of emission at red spectral range decreases linearly with increasing energy density of laser pulses from 0.45 to 1.4 J/cm^2. However, laser annealing results in increase of blue emission for the same samples. A blue band becomes dominant in PL spectra of partly and fully ablated films irradiated by pulses 1.1 and 1.4 J/cm^2. In the case of LPCVD film, laser annealing at density energy of pulses less than 1.8 J/cm^2 does not result in any changing of PL spectra. Irradiation of LPCVD films by pulses 1.8 and 2 J/cm^2 results in PL decrease, but no so significantly than in the case of furnace annealing. Moreover, the intensity of emission increases with increase of energy density of laser pulses from 1.8 to 2 J/cm^2. In this regard, the double laser pulse (1.4 J/cm^2 + 2 J/cm^2) has been tested for LPCVD film. As can be seen form Fig. 3 b such a double impact leads to PL signal light gain what could not be achieved by furnace annealing.

It can be assumed that the quenching of the dominated PL band with maximum at red range for PECVD film is also associated with K-center concentration decrease, as occurs during high-temperature (~1100 °C) furnace annealing. Increase of the blue band intensity with increasing energy density of laser pulses can be related to the ablation of nitride films and formation of polysilicon layer. Similar blue emission from polysilicon films was observed in Ref. [11]. It was attributed to the quantum size confinement effect of the ultrafine Si grains passivated by native oxide and recombination in the near interface states at Si/SiO$_2$. Thus, laser annealing is not suitable to enhance the emission of PECVD silicon nitride film, but it can potentially be used for the creation of red-green-blue silicon-based full color display.

In the case of LPCVD silicon nitride, the positive tendency of PL intensity increasing with increase of the pulse's energy density indicates that laser annealing is reasonable to use for these films. Taking into account the absence of ablation and good adhesion of LPCVD silicon nitride film to substrate after laser irradiation, the next stage of our work will involve multiple laser irradiations of LPCVD silicon nitride films by pulses with energy the density 2 J/cm^2.



a *b*

Fig. 3. PL spectra of the PECVD (*a*) and LPCVD (*b*) silicon nitride films before (*1*) and after laser annealing by single pulses with energy densities of 0.45 (*2*), 0.7 (*3*), 0.9 (*4*), 1.1 (*5*), 1.4 (*6*), 1.8 (*7*), 2 J/cm^2 (*8*) and by a double pulse (1.4 +2) J/cm^2 (*9*)

Рис. 3. Спектры фотолюминесценции PECVD- (*a*) и LPCVD-пленок (*b*) нитрида кремния до (*1*) и после лазерного отжига одиночными импульсами с плотностями энергии 0,45 (*2*), 0,7 (*3*), 0,9 (*4*), 1,1 (*5*), 1,4 (*6*), 1,8 (*7*), 2 (*8*) Дж/см^2 и двойным импульсом (1,4 + 2) Дж/см^2 (*9*)

Conclusions. The light-emitting properties of Si-rich silicon nitride films deposited on Si by PECVD and LPCVD techniques have been studied. It has been shown, in spite of similar stoichiometry ($SiN_{1.1}$), two types of films emit in different spectral range. PECVD-$SiN_{1.1}$ emits in red (640 nm) spectral range, and LPCVD-$SiN_{1.1}$ emits in blue (470 nm) one.

The effects of equilibrium furnace annealing and laser pulse annealing on the PECVD- and LPCVD-$SiN_{1.1}$ emission have been studied. It has been shown, the resulted PL spectra of annealed films depend both on a deposition method and on a type of thermal treatment. In the case of PECVD film, furnace annealing at 600 °C results in the increase of red PL band intensity. However, the intensity of red PL decreases after laser annealing. Furthermore, the irradiation by laser pulses with high energy densities (1.1 and 1.4 J/cm^2) results in ablation nitride film, formation of textured polysilicon layer as well as in blue emission enhancing.

In the case of the LPCVD film, a PL signal decreases as furnace annealing temperature increases. Laser annealing also results in the decrease of PL intensity of LPCVD film. However, the PL intensity tends to increase with increasing the laser energy density. The irradiation by double laser pulse (1.4 + 2 J/cm^2) leads to increase of PL signal. Thus, local laser annealing is reasonable to use for improvement of light-emitting properties of LPCVD silicon nitride $SiN_{1.1}$ films.

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