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(Citation)
Optics Letters,38(20):4224-4227

(Issue Date)
2013

(Resource Type)
journal article

(Version)
Accepted Manuscript

(URL)
https://hdl.handle.net/20.500.14094/90002572

Low-temperature growth of near-infrared luminescent Bi-doped SiO_xN_y thin films

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Received Month X, XXXX; revised Month X, XXXX; accepted Month X, XXXX; posted Month X, XXXX (Doc. ID XXXXX); published Month X, XXXX

Bi-doped siliconoxynitride (SiON:Bi) thin films were prepared by a sputtering method and the photoluminescence (PL) properties were studied. Without any thermal treatments, broad Bi-related luminescence was observed in the near infrared (NIR) range. The luminescence efficiency depended strongly on the film composition. It was found that N atoms play a crucial role for the formation of Bi NIR luminescence centers. The effect of annealing on the luminescence efficiency was also studied. The optimum annealing temperature to have the largest number of Bi NIR luminescence centers depended strongly on the film composition and it was lower for the films with lower N concentration. The PL excitation spectra revealed that two different Bi NIR luminescence centers exist in the films.

OCIS Codes: (160.4670) Optical materials, (160.4760 Optical properties, (300.6280) Spectroscopy, fluorescence and luminescence.
http://dx.doi.org/10.1364/OL.99.099999

Since the discovery of near-infrared (NIR) luminescence from Bi-doped aluminosilicate glasses [1], Bi doped NIR luminescent materials have been attracting great attention as a candidate material to achieve broadband near-infrared (NIR) optical amplification in the optical telecommunication window [2, 3]. A variety of Bi-based NIR luminescent materials, i.e., oxide glasses and fibers [1-6], chalcogenide glasses [7], chloride glasses [8], crystals [9, 10], molecular crystals [11-13], ionic liquids [14], zeolites [15], Bi clusters in solid neon matrix [16], etc., have been developed. In the case of Bi-doped silicate glasses, which are practically the most important because of the easy coupling with silica optical fibers, doping of additional elements such as Al, Ge, P, etc., had been believed to be crucial for the formation of Bi NIR luminescent centers. However, Razdobreev, et al., [17, 18] demonstrated that pure silica can also exhibit efficient Bi-related NIR luminescence if nano-sized pores are formed in silica glasses. Similar result was observed in Bi-doped oxidized porous Si [19, 20]. Although the diversity of materials and structures to have Bi NIR centers are still expanding, the luminescent centers are not clearly identified [21, 22]. To fully understand the origin of NIR luminescence from Bi-doped materials, not only detailed analyses of existing materials but also development of a new type of Bi-doped materials is indispensable.

In previous work, we developed NIR luminescent Bi-doped silica-based thin films by sputtering [23-25]. Xu et al. also demonstrated the formation of Bi-doped thin films by pulsed laser deposition [26]. The growth of Bi-doped thin films by a standard semiconductor technology opens a rout for the development of waveguide-type broadband optical amplifiers, which can be integrated on a Si-based optoelectronic device. For the application, it is not favorable to use elements which are not employed in a standard semiconductor technology. In previous work [23, 24], we demonstrated that pure silica films doped with excess Si atoms (Si-rich silica) can have Bi NIR centers without addition of other elements. However, for the activation of Bi NIR luminescent centers in Si-rich silica films and to obtain the spectral shape comparable to those of bulk glasses, very high temperature treatments, typically at temperatures higher than 900°C and sometimes at 1300°C, were necessary [23-25]. This is a significant drawback for the integration of the material to a Si-based device.

The purpose of this work is to develop a technology to grow NIR luminescent Bi-doped thin films with a very small thermal budget, ideally without any thermal treatments. We employed SiO_xN_y as a host of Bi NIR centers. The principle reason to choose SiO_xN_y as host materials for Bi NIR centers lies in its widely-tunable compositions in the non-stoichiometric region, making it possible to give rise to suitable micro-environment for Bi NIR centers at low temperatures or even without undergoing any thermal treatments. Another reason to study Bi-doped SiO_xN_y (SiON:Bi) is that, to our knowledge, nitrides have not been considered as a candidate host material of Bi NIR centers. In this work, we show that SiON:Bi thin films prepared by sputtering exhibits NIR luminescence with the maximum around 1330nm without any thermal treatments. We also demonstrate that the luminescence efficiency can be improved by annealing at relatively low temperatures and the effect of annealing depends strongly on the film compositions.

SiON:Bi thin films were deposited on fused quartz substrates by simultaneously sputtering SiO_2 and Bi doped silica glass (Bi_2O_3:SiO_2=50:50mol.%) targets [24] in Ar and N_2 mixture gas by using a multi-target rf magnetron sputtering system. Substrates were not intentionally heated during sputtering. The total pressure was fixed to 4mtorr, and the partial pressure ratio of N_2 gas was changed from 0 to 100%. The sputtering power of each target is fixed and thus the cation ratio is the same for all the samples (Bi:Si=1:99at.%). The films are amorphous and exhibit a broad IR absorption band around 1000cm^{-1}. The spectrum depends on the film composition and from the peak wavenumber, the composition can be estimated [27]. In this work, when the N_2 gas partial pressure ratio is changed from 0 to 100%, x/(x+y) in SiO_xN_y is changed from 1 to 0.4. The film thickness was fixed to 500 nm. To study the influence of annealing on the PL properties, some samples were annealed in N_2 gas atmosphere for 30 min at temperatures from 500°C to 1100°C. The films are amorphous even after annealing.

The PL spectra were measured by using a spectrometer equipped with a liquid N_2 cooled InGaAs diode array (OMA-V-SE, Roper Scientific). The excitation sources were a 488nm line of an Ar ion laser or 785 nm light from a diode laser. The contour maps of excitation and emission wavelengths were obtained by a spectrofluorometer (Flourolog, Horiba Jobin Yvon). The spectral response of the detection system was corrected with a reference spectrum of a standard halogen lamp. Photoluminescence decay curves were measured using a NIR photomultiplier (R5509-72, Hamamatsu Photonics). PL was measured at temperatures from 6 to 300K. However, since the spectral shape was independent of the temperature, we only discuss the data at room temperature.

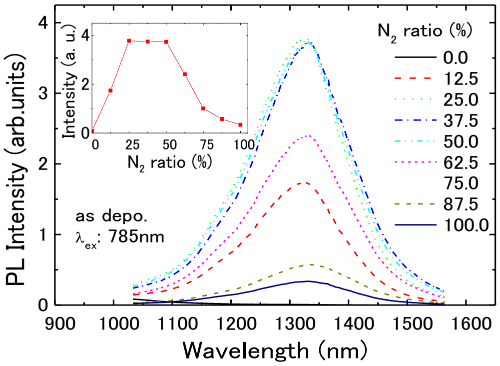

Fig. 1 PL spectra of as-deposited SiON:Bi films. N_2 partial pressure ratio is changed from 0 to 100 %. Inset is the PL peak intensity as a function of the N_2 partial pressure ratio.

Figure 1 shows PL spectra of as-deposited SiON:Bi thin films deposited in different N_2 partial pressure ratios. When the N_2 partial pressure is 0%, i.e., sputtering in Ar gas, no NIR PL is detected. This is consistent with previous work that NIR PL of Bi-doped pure silica is weak and difficult to detect in thin film samples [23, 24], although it is observable in bulk glasses and fibers [6, 21]. When sputtering is performed in Ar and N_2 mixture gas, a PL with the full width at half maximum (FWHM) of about 200nm appears around 1330nm. The observed PL spectra are very similar to those reported for Bi-doped multicomponent glasses [1-6]. In the inset of Fig. 1, the PL peak intensity is plotted as a function of the N_2 partial pressure ratio. The intensity depends on the ratio and is the highest when it is in the range from 25 to 50%. The observation of Bi NIR PL from Si-based thin films without any thermal treatments is an important step for the development of waveguide-type optical amplifiers integrated in Si optoelectronic devices.

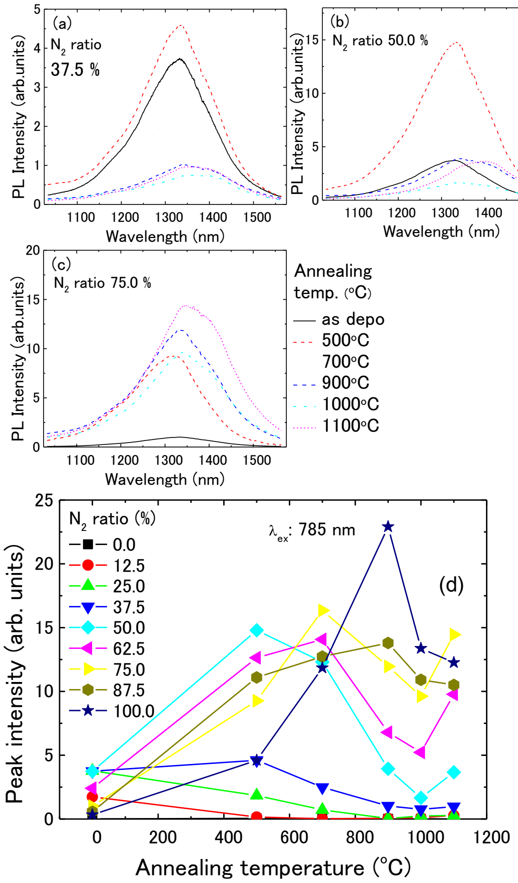

Fig. 2 PL spectra of SiON:Bi films annealed at different temperatures. N_2 partial pressure ratios are (a) 37.5%, (b) 50.0% and (c) 75.0%. (d) PL peak intensity as a function of annealing temperature for SiON:Bi films deposited in different N_2 partial pressure ratios.

Figure 2(a) shows PL spectra of SiON:Bi annealed at different temperatures for the sample deposited in the N_2 partial pressure ratio of 37.5%. By annealing at 500°C, the PL intensity increases slightly. However, further increase of the annealing temperature results in the quenching of the PL. On the other hand, for the sample deposited in the N_2 partial pressure ratio of 50% (Fig. 2(b)), it increases about four times by annealing at 500°C. The effect of annealing becomes more prominent for the samples deposited in higher N_2 partial pressure. When the partial pressure ratio is 75% (Fig. 2(c)), it increases nine times by annealing at 500°C and reaches the maximum when the annealing temperature is 700°C. In Fig. 2(d), annealing temperature dependence of the PL intensity of samples deposited in different N_2 partial pressure ratios is summarized. When it is below 25%, annealing results in the quenching of the PL, while it enhances the PL efficiency when the partial pressure ratio is above 37.5%. The optimum annealing temperature to maximize the PL intensity increases with increasing the N_2 partial pressure and reaches 900°C when the partial pressure ratio is 87.5 and 100%. Note that, when the N_2 partial pressure ratio is 0%, no NIR PL is detected in the whole annealing temperature range.

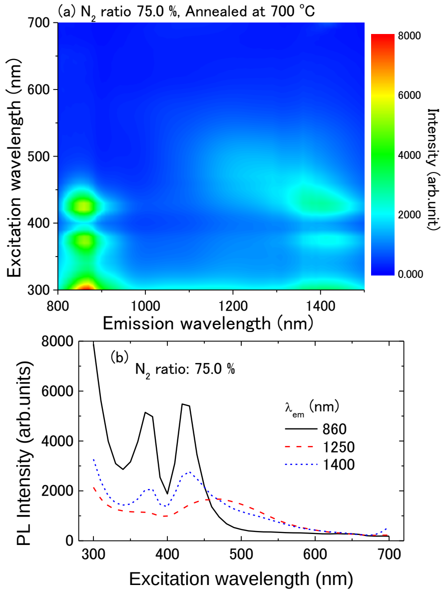

Fig. 3 (a) Contour plot of PL intensity of SiON:Bi films (N_2 partial pressure ratio: 75%, annealing temperature 700°C) as functions of excitation and emission wavelengths. (b) PL excitation spectra detected at different wavelengths.

In Fig. 2(a)-(c), annealing does not strongly affect the spectral shape below 700°C. A bump appears on the longer wavelength side of the peak around 900°C and it grows with increasing the annealing temperature. This results in the shift of the PL maximum to at maximum 1400nm. The change of the spectral shape suggests that different types of Bi NIR centers co-exist and the number ratio depends on the annealing temperature.

To further examine Bi NIR centers in these films, we took a contour plot of the PL intensity as a function of excitation and emission wavelengths. Figure 3(a) shows an example for the sample deposited in the N_2 partial pressure ratio of 75% and annealed at 700°C. Three broad PL bands can be seen around 860, 1250 and 1400 nm. Figure 3(b) shows the PL excitation spectra detected at the maxima of the three bands. The 1250 nm PL has a broad excitation band centered at 470nm. The excitation spectrum is very similar to those reported for Bi doped multicomponent glasses, which shows luminescence in the 1100 to 1250nm range [6, 28]. On the other hand, the PL around 860 and 1400nm has excitation bands around 370 and 430nm. The PL and excitation spectra resemble very much to those of Bi-doped nanoporous silica [17-20] Similar PL excitation spectra were obtained for other samples deposited in different N_2 partial pressure and annealed at different temperatures (not shown here).

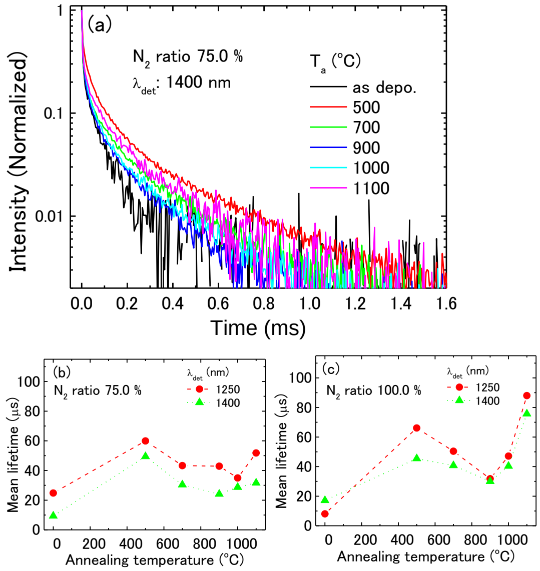

Fig. 4 (a) PL decay curves of SiON:Bi films (N_2 partial pressure ratio: 75%) annealed at different temperatures. (b) and (c) PL mean lifetime of SiON:Bi films detected at different wavelengths as a function of annealing temperature. N_2 partial pressure ratio: (b) 75%, (c) 100%.

Figure 4(a) shows PL decay curves of the samples deposited in the N_2 partial pressure ratio of 75% and annealed at different temperatures. The detection wavelength is 1400nm. The decay curves are not single exponential and consists of several components with different lifetimes. The lifetime of the slowest component for the sample annealed at 500°C is about 565 μsec, which is close to those reported for Bi-doped silicate glasses [1-3]. In Fig. 4(b) and (c), the mean lifetime defined by

$$\tau = \int_{t_0}^{\infty} \left[I(t) / I_0 \right] d(t),$$

where $I(t)$ is the PL intensity as a function of time t and I_0 is the initial intensity at time t_0, for the samples deposited in N_2 partial pressure ratio of 75% and 100% are plotted, respectively, as a function of annealing temperature. The data obtained at different detection wavelengths are shown. The lifetime slightly depends on the detection wavelength. In both cases, the mean lifetime of as-deposited samples is from ten to several tens of μsec. It increases drastically by annealing at 500°C and reaches ~60μsec. Further increase of the annealing temperature results in the shortening of the lifetime. Therefore, optimum annealing temperature for curing the nonradiative centers is very low in SiON:Bi films. Above 1000°C, the lifetime again increases. This is probably due to the growth of another type of Bi NIR centers, because the lifetime lengthening is accompanied by the emerging of a new PL band at 1400nm. By further increasing the annealing temperature, e.g., to 1300°C, the lifetime is considered to be longer. However, it is not practical for applications in integrated optoelectronic devices.

It is interesting to note that the annealing temperature dependence of the PL lifetime is very much different from that of the intensity shown in Fig. 2. This discrepancy indicates that the number of Bi NIR centers is a major factor to determine the PL intensity. For example, when the N_2 partial pressure ratio is 87.5%, the PL intensity increases with increasing the annealing temperature till 900°C, although the lifetime has the maximum at 500°C, implying that the number of Bi NIR centers continue to increase till 900°C. As discussed above, the annealing temperature to achieve the highest concentration of Bi NIR centers increases with increasing the N_2 partial pressure. This is probably due to different diffusion coefficients of Bi in SiON with different compositions. When N concentration is low, the diffusion coefficient of Bi in SiON is relatively large and Bi NIR centers are effectively formed by low temperature annealing or even without annealing. However, large diffusion length of Bi at higher temperature results in the formation of Bi metal clusters that do not contribute to NIR PL. On the other hand, when N concentration is high, diffusion length of Bi is not large enough for the formation of Bi NIR centers during sputtering and annealing at low temperatures. Higher temperature annealing is necessary to maximize the number of Bi NIR centers.

In summary, SiON:Bi thin films deposited by sputtering shows Bi-related NIR PL without any thermal treatments. To our knowledge, this is the first successful formation of Bi NIR centers in thin films at room temperature, which paves the way for their applications in integrated photonics. The data clearly show that N plays a crucial role for the activation of Bi NIR centers at low temperature. We also show that the optimum annealing temperature to maximize the PL intensity depends on the composition of SiON films. The present results suggest that SiON can be a promising candidate material as a host for Bi NIR active centers.

This work is supported by KAKENHI (23310077).

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