Preparation of amorphous CN_x thin films by pulsed laser deposition using a radio frequency radical beam source

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Amorphous CN_x thin films were deposited by pulsed laser deposition (PLD) combined with a nitrogen rf radical beam source which supplies active nitrogen species to the growing film surface. The dominant active nitrogen species are excited N_2 molecules and nitrogen atoms. The deposited films were characterized by scanning electron microscope, x-ray photoelectron spectroscopy (XPS), Raman scattering, and Fourier transform infrared (FTIR) spectroscopy. Nitrogen content of the deposited films increased with increasing rf input power and N_2 pressure in the PLD chamber. The N/C ratio 0.23 was obtained at 400 W of rf input power and 1.3 Pa. XPS N 1*s* spectra shows the existence of N–sp^2C and N–sp^3C bonds in the deposited films. The fraction of the N–sp^3C increased with increasing of N_2 pressure in the PLD chamber during the operation of radical beam source. FTIR and Raman spectra of the deposited films indicated that N≡C bonds in the films were few as compared to the other carbon and nitrogen bonds. © *1999 American Institute of Physics.* [S0021-8979(99)02216-1]

I. INTRODUCTION

The deposition of carbon nitride, CN_x, thin films has been of much interest recently since the theoretical calculations by Liu and Cohen.[1,2] They predicted that β-C_3N_4, similar in structure to β-Si_3N_4, was comparable with or even harder than diamond. These reports sparked the studies on the preparation of carbon nitride thin films. Conversely, Guo and Goddard III showed from *ab initio* calculation, that β-C_3N_4 is about half as hard as diamond.[3] More recently, Teter claimed that the hardness of C_3N_4 should not exceed that of *c*-BN, let alone diamond.[4] He reported that the shear modulus is a better predictor of hardness than the bulk modulus, and C_3N_4 have shear moduli of only 60% that of diamond. Apart from its predicted hardness, carbon nitride, even N/C ratio is less than 1.33 (stoichiometric value of C_3N_4), may prove to be useful in the fields of material science, tribological and wear-resistant coating, optical, and electronic engineering. Amorphous carbon nitride is expected to be an interesting new material because it exhibits outstanding properties such as high value of hardness, low friction coefficient, chemical inertness, and variable electrical conductivity and optical band gap. Amorphous carbon nitride has been considered as an electronic material whose electronic structure and band gap are controllable by controlling the composition.

Many experimental efforts have been made to prepare carbon nitride thin films. Various deposition techniques have been adopted. They include dc and rf magnetron sputtering deposition,[5–9] ion beam and ion beam assisted deposition,[10–12] nitrogen implantation,[13] pulsed laser deposition,[14–18] and chemical vapor deposition.[19–21] Recently, we have prepared crystalline carbon nitride thin films with a high nitrogen content by the electron cyclotron resonance (ECR) sputtering deposition.[22]

In this study, we tried to prepare amorphous carbon nitride, *a*-CN_x (x is less than 1), thin films by the pulsed laser deposition technique. Pulsed laser deposition (PLD) is a suitable method in order to fabricate a large variety of thin films with high quality.[23] There are some attempts to prepare carbon nitride thin films by PLD. The specific feature of PLD technique is that the average energy of the particles in the laser-evaporated species is higher than the thermal evaporation energy. From observations of formation of amorphous carbon films, it has been shown that the high energetic particles are favorable to form sp^3-bonded carbon compared to sp^2-bonded carbon, since the high energetic particles injected onto the film produce high compressive stress in the film.[24–26] Most of the efforts to prepare carbon nitride thin films by PLD using a graphite target are carried out under nitrogen containing atmosphere.[14–16] Preparation of carbon nitride thin films by PLD using a graphite target under atomic or ionic nitrogen beam irradiation has also been reported.[17,18]

In this article, amorphous carbon nitride thin films were prepared by PLD by use of a graphite target, utilizing nitrogen radical beam source during the deposition process. We investigate the effects of nitrogen pressure in the PLD chamber and rf input power during the operation of radical beam source on the film characteristics. Composition and chemical bonding of the deposited films were studied using x-ray photoelectron spectroscopy (XPS). Fourier transform infrared (FTIR) and Raman scattering spectroscopy were also utilized in order to characterize the deposited films.

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II. EXPERIMENT

A. Preparation of films

The PLD system consists of a KrF excimer laser, nitrogen radical beam source, and vacuum chamber. The background pressure of the PLD chamber before deposition was less than 1×10^{-6} Pa using a turbomolecular pump. A Lambda Physics COMPex 102 laser charged with KrF (248 nm) is operated at 5 Hz and 200 mJ pulse^{-1}, which is focused onto a rotating graphite target surface to give $\sim$3 J cm^{-2} energy density. A Si (100) wafer was used as the substrate. Before deposition, the substrate was ultrasonically cleaned in acetone. The distance between a substrate and a graphite target was kept at 3.5 cm. The substrate temperature was room temperature.

A 13.56 MHz rf radical beam source (Vacuum Products Corporation) was used for providing an active nitrogen beam. An active nitrogen plasma is generated in a pyrolytic boron nitride (PBN) discharge tube supplied with N_2 gas. The active nitrogen species diffuse out of the discharge tube towards the substrate inside the PLD chamber through the small holes at one end of the discharge tube. The rf radical beam source was operated at various background N_2 pressures in the PLD chamber in the range of 1.3×10^{-3}–1.3 Pa and rf input powers in the range of 200–400 W, respectively. In order to characterize the active nitrogen species, optical emission spectroscopy of the generated nitrogen plasma in the discharge tube were performed using a Otsuka Electronics MCPD-2000 multichannel photodetector. Spectra were recorded through the quartz window at the end of the discharge tube. The obtained spectra showed several emission bands which are attributed to the second positive N_2 band ($C\ ^3\Pi_u \rightarrow B\ ^3\Pi_g$), the first positive N_2 band ($B\ ^3\Pi_g \rightarrow A\ ^3\Sigma_u^+$), and the first negative system of N_2^+ molecules ($B\ ^2\Sigma_u^+ \rightarrow X\ ^2\Sigma_g^+$).[27–30] The emission intensity of first negative system of N_2^+ was very weak as compared to the first positive and second positive N_2 bands. From the obtained spectra, we inferred that the dominant active nitrogen species generated by the radical beam source utilized in this study are excited N_2 molecules (N_2^*) and atomic nitrogen (N), although some quantity of N_2^+ were also emanated.

B. Characterization of films

XPS was carried out on a Rigaku XPS-7000 by using Mg $K\alpha$ 1253.6 eV x-ray source to characterize the electronic and compositional properties of the deposited films. Prior to XPS measurements, films were sputter cleaned by 0.5 keV Ar^+ etching for 1 min. All obtained spectra were calibrated using the signal from the implanted Ar $2p_{2/3}$ peak at 241.3 eV. FTIR was carried out on a JEOL IR-8300. FTIR spectra were recorded in transmission mode for the films deposited on Si substrates. Absorption of bare Si was subtracted as background from the obtained spectrum. Raman spectroscopy was performed using a micro-Raman instrument, RENISHAW JRS-System2000, with an argon laser (514.5 nm). Surface morphology of the deposited films was observed by scanning electron microscope (SEM), JEOL JSM-6301F.

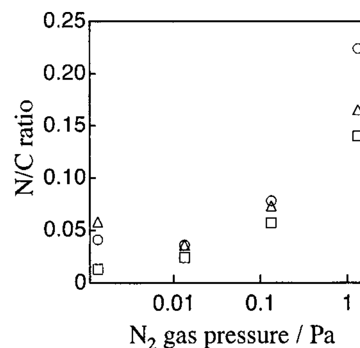

FIG. 1. N/C atomic ratio of the films as a function of N_2 pressure with different rf input power. □: 200, △: 300, and ○: 400 W.

III. RESULTS AND DISCUSSION

From SEM observation of the surface of the deposited films, the surface was plane and very smooth without any cracks, and any particulates were not observed. Similar surface morphology were observed for the films deposited at another deposition conditions. Surface morphology was not affected by the deposition condition in the present experimental condition range.

The N/C atomic ratio evaluated from XPS measurements of films deposited under nitrogen radical beam irradiation at various rf input powers as a function of N_2 gas pressure are shown in Fig. 1. The N_2 gas pressure in the PLD chamber was varied in the range from 1.3×10^{-3} to 1.3 Pa. The N/C atomic ratio increased with increasing N_2 gas pressure and increasing rf input power. The highest N/C ratio in the present experiment was 0.23 for the film deposited under nitrogen radical beam irradiation at 1.3 Pa and 400 W rf input power. The following experiments were carried out at 400 W rf input power.

Figure 2 shows C 1s electron spectra of films deposited under nitrogen radical beam irradiation at various N_2 gas pressure accompanied with spectrum of amorphous carbon film which deposited without radical beam irradiation. The peak positions of all films were almost constant at 284.6 eV. It can be considered that the main bonding configuration of

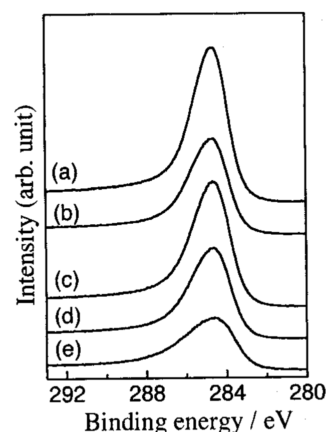

FIG. 2. C 1s electron spectra of amorphous carbon film (a) and the films deposited under nitrogen radical beam irradiation at various N_2 pressure. (b) 1.3×10^{-3}, (c) 1.3×10^{-2}, (d) 1.3×10^{-1}, and (e) 1.3 Pa.

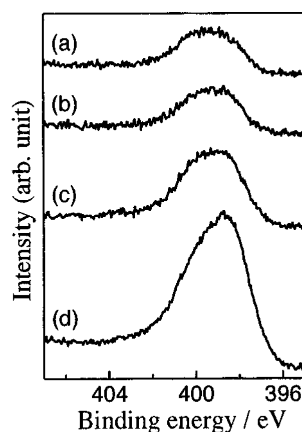

FIG. 3. N 1*s* electron spectra of the films deposited under nitrogen radical beam irradiation at various N_2 pressure: (a) 1.3×10^{-3}, (b) 1.3×10^{-2}, (c) 1.3×10^{-1}, and (d) 1.3 Pa.

TABLE I. Results of deconvolution of N 1*s* electron spectra of films deposited under nitrogen radical beam irradiation at various N_2 pressure.

Pressure/Pa	Peak position/eV	FWHM/eV	Relative area	I/II
1.3×10^{-3}	398.3	1.7	0.31	0.46
	399.8	2.3	0.68	
	402.0	1.0	0.02	
1.3×10^{-2}	398.4	1.7	0.42	0.81
	399.9	2.1	0.52	
	402.0	2.1	0.05	
1.3×10^{-1}	398.5	2.0	0.52	1.41
	400.1	2.0	0.37	
	402.0	3.4	0.11	
1.3	398.4	2.2	0.62	2.00
	400.1	2.0	0.31	
	402.0	2.6	0.07	

carbon atoms in the film is C–C. The C 1*s* spectra of carbon nitride films, spectra (b)–(e), are broad and asymmetric at the higher binding energy side as compared with the spectrum of carbon film, spectrum (a). It suggests that there are several bonding configurations related to carbon atoms in the films. The peaks had tails located on the higher energy side, and the component of higher energy side increased with increasing N_2 gas pressure. According to the published data[6,8,16,31–33] it is considered that the component of higher energy side is related to C–N bonds. We have not performed further quantitative analysis of C 1*s* electron spectra, because it is very difficult to separate into each peak due to complexity of the bonding of carbon atoms.[14]

N 1*s* electron spectra of films deposited at various N_2 pressure are shown in Fig. 3. N 1*s* spectra had a broad peak corresponding to some bonding configurations related to nitrogen atoms. All spectra can be deconvolved into three Gaussian-type peaks as illustrated in Fig. 4. The deconvolved peaks were located at ~398, ~400, and ~402 eV. We present these peaks as I, II, and III, respectively. The results of the peak deconvolutions are presented in Table I, where peak positions, full width at half maximums (FWHM), peak areas, and peak area ratios of the peaks I–II are summarized. These deconvolutions are in good agreement with previous reported CN_x films.[6–10,14,31–33] The small peak located at higher binding energy side, the peak III, can be assigned to N–O bonds, and it probably related to incorporation of oxygen into the sample due to exposure to the atmosphere before XPS measurement and/or accidental incorporation during the deposition. We discuss the other two peaks afterward. The two main peaks, I and II, are generally ascribed to C–N covalent bonds. The chemical shifts between these two peaks are 1.5–1.7 eV, they reflect two different bonding configurations of C–N in the films. We assigned these peaks according to the previous published N 1*s* electron spectra of CN_x films.[6–10,14,31–34] Peak I can be assigned to N bonded to sp^3-hybridized carbon (N–sp^3C) and peak II contributed to N bonded to sp^2-hybridized carbon (N–sp^2C).

The area ratios of peak I to peak II, I/II, changed according to the N_2 pressure in the PLD chamber. Figure 5 shows the relationship between the peak I/II as a function of N_2 pressure. The peak area ratio of the I to the II increased monotonously with increasing N_2 pressure. The I/II peak area ratio reached 2.0 for the film deposited at 1.3 Pa. The ratio indicates that the amount of nitrogen bonded to sp^3-hybridized carbon in the films increased with N_2 pressure during the operation of the radical beam source. From these XPS results, it can be concluded that the amount of N–sp^3C increases accompanied by an increase in the total amount of nitrogen in the films with increasing N_2 pressure in the PLD chamber. A similar tendency was observed for the films deposited by the reactive dc magnetron sputtering method,[9] which indicates that the nitrogen atoms tend to

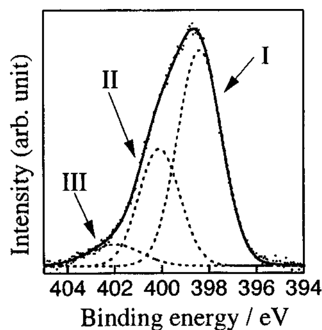

FIG. 4. Typical deconvolved N 1*s* electron spectra of the film. The spectra are presented after background subtraction and using Gaussian fits.

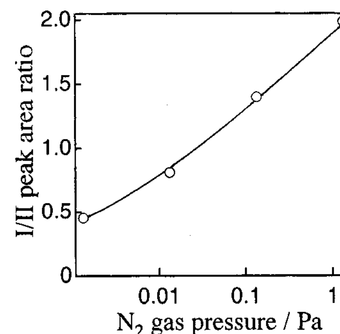

FIG. 5. Relationship between I/II peak area ratio obtained from deconvolution of N 1*s* electron spectra as a function of N_2 pressure.

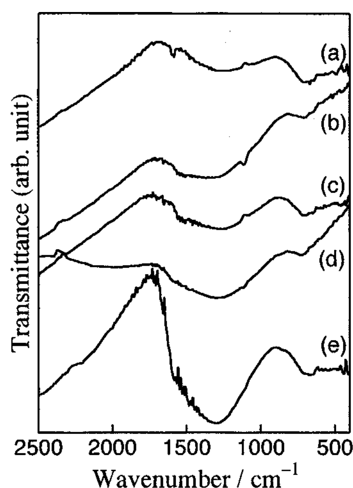

FIG. 6. FTIR spectra of the carbon film (a) and the films deposited under nitrogen radical beam irradiation at various N_2 pressure (b) 1.3×10^{-3}, (c) 1.3×10^{-2}, (d) 1.3×10^{-1}, and (e) 1.3 Pa.

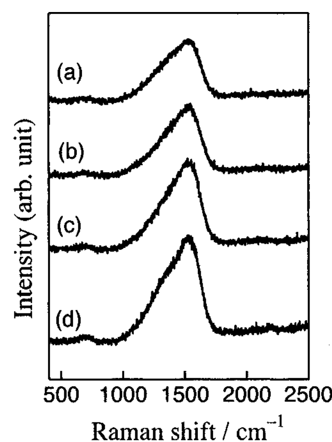

FIG. 7. Raman spectra of the deposited films under nitrogen radical beam irradiation at various N_2 pressure: (a) 1.3×10^{-3}, (b) 1.3×10^{-2}, (c) 1.3×10^{-1}, and (d) 1.3 Pa.

bound to sp^2-hybridized carbon atoms at the initial stage and then some of the nitrogen atoms will be bound to sp^3-hybridized carbon atoms when more nitrogen atoms are incorporated.[9]

The bonding states of carbon and nitrogen atoms were also observed by FTIR measurements. Figure 6 shows FTIR transmission spectra of the films deposited under nitrogen radical beam irradiation at various N_2 pressure, accompanied with amorphous carbon film which deposited without nitrogen radical beam irradiation. The absorption band around 700 cm^{-1} can be assigned to the out-of-plane bending mode for graphite-like domains.[5] FTIR spectrum of the CN_x film, spectrum (b)–(e) in Fig. 6, showed broad absorption band from 1000 to 1700 cm^{-1}. The absorption of this broad band increased with increasing N_2 pressure during the operation of radical beam source, because of the increasing nitrogen content of the films. This broad absorption band is related to the Raman active *G* and *D* bands. According to Kaufman *et al.*,[35] these Raman active *G* and *D* bands become IR active due to incorporation of nitrogen atoms into the carbon network and the resulting symmetry breaking of carbon network. They reported broad absorption band with absorption maximum at around 1570 and 1370 cm^{-1}. Wixom[36] proposed that N–sp^3C stretching frequency is estimated to be in the range of 1212–1265 cm^{-1}. Lu *et al.*[37] assigned the absorption maximum at 1222 cm^{-1} as the N–sp^3C bond and at 1689 cm^{-1} as the N–sp^2C bond. González *et al.*[15] reported an absorption band centered at 1625 cm^{-1} attributed to N–sp^2C vibration and an absorption band between 1500 and 1350 cm^{-1} to be assigned to N–sp^3C bond. From these interpretations of IR spectra for carbon nitride, the broadband from 1000 to 1700 cm^{-1} in our obtained spectra can be assigned to some C–N bonds. In our spectra, absorption at smaller wave numbers increased with increasing N_2 pressure, and the component of the spectrum at smaller wave numbers is larger than that at larger wave numbers for the film obtained at 1.3 Pa. This observation indicates that the contribution of the component at smaller wave numbers is large. The increasing contribution of the smaller wave number component with increasing N_2 pressure may suggests that the fraction of N–sp^3 bonds increased with increasing nitrogen content of the films. This result is in accord with the XPS analysis mentioned earlier. This interpretation, however, is tentative, because of the overlap with the sp^2 carbon vibrations.[31] The obtained spectra of the CN_x film showed very weak absorption at ~2200 cm^{-1} which is assigned to the N≡C stretching mode.[35] It was found that N≡C bonds are a minority in the film. Any other absorption bands, such as N–H and C–H stretching modes, were not observed in the range of 2500–4000 cm^{-1}.

Raman spectroscopy is a suitable tool for the characterization of carbon related materials. Figure 7 shows Raman spectra of films deposited at various N_2 pressure in the PLD chamber. The spectra showed broad asymmetric bands at about 1500 cm^{-1}. The obtained spectra are similar to that of diamond-like carbon (DLC). It was found that the films have mainly a diamond-like structure. These Raman spectra deconvolved into two Raman active bands; one at ~1550 cm^{-1} and another at ~1380 cm^{-1}. These bands are characteristics of graphitic (*G*) and disordered (*D*) bands, respectively.[38,39] The *G* band corresponds to graphite-like sp^2 carbon and *D* band corresponds to disordered sp^2 carbon induced by the linking with sp^3 carbon atoms.[38,39] Significant changes of the intensity ratio of *D* and *G* bands, I_D/I_G, with N_2 pressure (i.e., N/C ratio in the deposited film) were not observed. The ratios I_D/I_G were ~2.0 for all films. For the film deposited under nitrogen radical beam irradiation at 1.3 Pa, spectrum showed a very weak peak at ~2250 cm^{-1}. This peak can be assigned to nitrogen bonded to sp^1–hybridized carbon, i.e., N≡C.[40] The peak assigned to the N≡C is very weak, indicating that the amount of N≡C is small as compared to the other carbon and nitrogen bonding states. This result coincides with FTIR analysis.

IV. CONCLUSIONS

CN_x thin films have been deposited on a Si (100) wafer at room temperature by pulsed laser deposition using KrF excimer laser and graphite target combined with rf radical

beam source. From optical emission spectroscopy of the generated nitrogen plasma in the discharge tube of the radical beam source, it was found that the dominant active nitrogen species were excited N_2 molecules (N_2^*) and ground state (^4S) nitrogen atoms. Some quantity of N_2^+ ions also emanated from the radical beam source. The N/C ratio determined by XPS depended on rf input power and N_2 background pressure in the PLD chamber. The N/C ratio increased with increasing rf input power and N_2 pressure in the PLD chamber. The N/C ratio 0.23 was obtained for the film deposited at rf input power 400 W and 1.3 Pa. XPS C 1*s* peaks of the deposited films were located at 284.6 eV, indicating that the main bonding configuration of carbon atoms are C–C. The C 1*s* peaks were asymmetric and had tails at higher energy side. The result suggests the presence of several different types of bonds of carbon atoms. N 1*s* electron spectra were deconvolved into N–sp^2C and N–sp^3C signals. The fraction of N–sp^3C increased with increasing N_2 background pressure in the PLD chamber, i.e., increasing of N/C ratio in the deposited film. FTIR and Raman spectra indicated that the films have mainly a diamond-like structure and N≡C bonds are small as compared with other carbon and nitrogen bonding states.

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