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Nano-crystalline Diamond Films for X-ray Lithography Mask

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1. Introduction

At present, the integrated circuits have developed from only dozens of devices on a chip in 20th century 60's to today's one billion devices on each chip. And lithography technology has played a crucial role in its rapid development. As the X-ray wavelength is very short, it meets the needs of the development of ultra-large scale integrated circuit and has drawn a lot of attention in recent years. X-ray lithography is the first alternative to optical lithography in the future

In the X-ray band, the material reduction of atomic absorption coefficient decrease with the atomic number. Although the diamond is consisted of carbon element and the atomic number is greater than the conventional X-ray window materials beryllium, it can be prepared very thin (less than 1 micron), and is also able to withstand 1 atm pressure because of its high strength. Thus, compared to the traditional 8-micron beryllium window, the thickness of prepared diamond films can be nearly 1/20, and a higher X-ray transmission rate can be obtained. In particular, in the longer wavelength soft X-ray band, beryllium window, basically, is not translucent. By this work, it shows that the prepared free-standing diamond film X-ray window has a transmission rate as high as 52.7% in the vicinity of E = 258eV. Thus, compared to a highly toxic beryllium window, the thin film has many application advantages, such as high transmission rate, high strength, high radiation resistance and nontoxicity.

In this paper, hot filament assisted chemical vapor deposition (HFCVD) method was applied to prepare diamond film. While maintaining the concentration of carbon source and changing the deposition parameters, such as reaction pressure, substrate temperature, nano-crystalline diamond (NCD) film with low surface roughness was deposited on silicon substrates. On this basis, hydrogen etching process was applied to further improve the quality and structure of the films. X-ray lithography mask-based material, whose optical transmittance and surface roughness meet the requirements of large integrated circuits (VLSI) was obtained.

2. Preparation of nano-crystalline diamond films

The preparation of nano-crystalline diamond films lies in: 1, the increase of the growth of nucleation density; 2, a very high secondary nucleation rate to suppress the growth of diamond nuclei and obtain films with nanometer-sized grain. In this study, HFCVD

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apparatus was used to prepare NCD films, with tantalum wire as a heat source, acetone and hydrogen as a reactant, 2 cm × 2 cm p-type polished silicon as substrate. The detailed deposition parameters were shown in Table 1. By changing the deposition temperature, pressure, hydrogen plasma treatment during the process of the hot-filament chemical vapor deposition (HFCVD), we studied the new technology for the preparation of NCD films. The microstructure and quality of the NCD film were characterized and analyzed.

Pressure (kpa)	Gas flow rate (acetone: hydrogen)	substrate temperature (°C)	Substrate bias/current (V/A)
1-3	50:180	600-700	+18V/4A

Table 1. HFCVD deposition parameters

It is noteworthy that, tantalum wire was polished with sand papers to clean the surface before the experiment. Pretreatment was carried out by importing hydrogen and acetone about 30 minutes to remove the surface oxide layer impurities of tantalum wire and form a layer of carbide coating in the surface to prevent pollution of prepared diamond film surface because of the evaporation of the tantalum.

In addition, before the preparation of diamond films, a series of pretreatments of Si substrate was conducted: first, Si substrate was placed in HF solution for 5 minutes to remove the oxide layer, then handly grinded for 15 minutes in the mixture of ultra-fine diamond powder with 100 nm particle size and glycerol. After these, the substrate was ultrasonically cleaned in baths of acetone and DI water, for 15 and 10 minutes, respectively. The above cleaning steps were repeated until the substrate surface was very clean.

2.1 The effects of deposition temperature on the quality of nano-crystalline diamond films

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Generally, the deposition temperature and pressure of NCD films are lower than the microcrystalline diamond films. In this experiment, the substrate temperature is 600-700°C. If the temperature is too low, the catalysis of hydrogen and hydrocarbons doesn't work and it's difficult to form diamond film. If the temperature is too high, the carbon tantalum alloys formed on the surface are volatile, resulting in the pollution of the matrix. In this study, while other conditions (acetone and hydrogen flow ratio of 50/180, pressure of 1 kPa) were maintained, the deposition temperatures were changed (detailed parameters shown in Table 2).

Samples	T1	T2	T3	T4
Temperature/°C	600	640	680	700

Table 2. Deposition temperature of diamond films

Figure 1 shows the surface morphology of the samples observed under scanning electron microscopy (SEM). It can be found that as the temperature increases, the grain size increased

slightly, but not significantly, and all the grain sizes are basically below 50nm. In this study, a low deposition pressure (1 kPa) was applied and in the temperature range it was easy to achieve conditions beneficial for secondary nucleation. However, with the increase of temperature, the surface roughness trend to become larger.

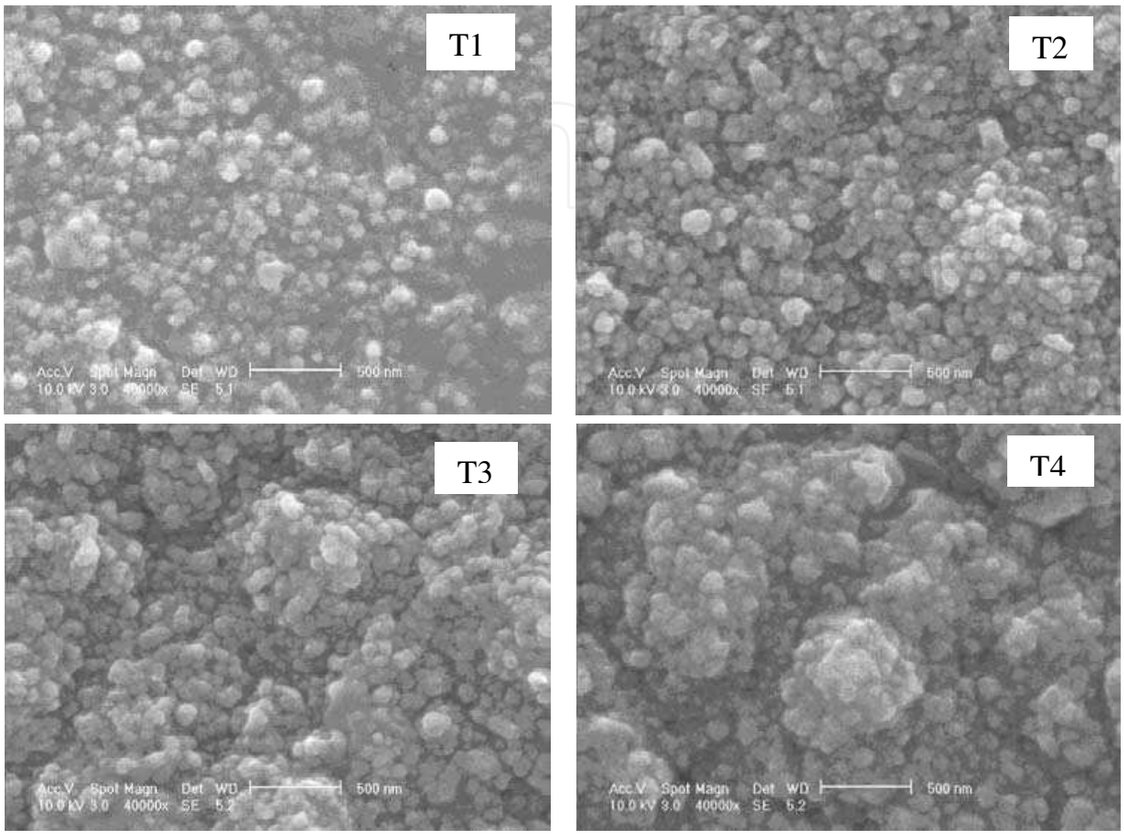


Fig. 1. SEM images of NCD films deposited under different temperature
(T1. 600°C,T2. 640°C ,T3. 680°C ,T4. 700°C)

The atomic force microscope (AFM) images shows the surface root mean square roughness (RMS), which are 35 nm, 36.1 nm, 45 mm and 50.2 mm for the sample T1, T2, T3 and T4, respectively. Compared with the surface of micron diamond films, NCD films' surface is very smooth with RMS only in tens of nanometers, which not only solves the issue of diamond films polishing, but also greatly reduced light scattering of the film surface. And with the decreasing of temperature, the surface roughness of the samples decreased rapidly.
Raman spectroscopy (HORIBA JOBIN YVON HR800 UV) was applied to characterize Raman spectra of the films. The excitation wavelength is 514.5 nm(Ar^+) with spectral measurement range of 1000~1800cm^{-1}.
The Raman spectrum of the samples given in figure 2 revealed several features that may be linked to NCD characters. First, the diamond peak at 1332cm^{-1} was very weak and significantly broadened, which was well known to be caused by the decrease of the grain size to the nanometer scale. Second, the bands at approximately 1140cm^{-1} and 1480cm^{-1} were presented, which had been assigned to trans-polyacetylene lying at grain boundaries in recent works and always been observed in chemical vapor deposition(CVD) NCD films as

well as in hydrogenated amorphous carbon(a-C:H) containing nanocrystalline diamond. They were often regarded as a marker of NCD phase. Third, the scattering intensity at approximately 1350cm^{-1} and 1560cm^{-1},corresponding to sp^2- carbon(graphite D and G bands, amorphous carbon),was obviously enhanced.

As the temperature increased, the diamond peaks reduced and broadened. When the temperature gets close to 700°C, the intensity of characteristic peaks of diamond and graphite are very close, indicating that the increase of temperature leads to the increase of disordered graphite in the thin films. When the temperature is too high, the growth rate is great, and the first nucleation of grains grow soon which restrains the formation of other diamond nuclei, making the nucleation density on the matrix not high and more greater holes between grains. Thus the quality of the film declines. At 640°C, characteristic peaks of diamond of the samples is more obvious and diamond/graphite peak intensity reaches the maximum. It's considered that at this temperature there are a relatively small number of impurities and defects in the films. Above 600°C, the film quality declines because low deposition temperature makes the activity of atoms H decrease, and etching effect not strong. Meanwhile the gasification of graphite and other ingredients weakened, leading to an increase of non-diamond phase.

As the temperature increases, the proliferation and resettlement of various types of activated free radicals, quickens, the effect of active hydrogen enhances, which increase the diamond growth rate and the electric performance. On the contrary, the high deposition temperature makes the distortion from sp^3 carbon bond to sp^2 carbon bond combined group (i.e., graphitization). Moreover, because of the high deposition temperature, the activity of hydrogen atoms is too strong, resulting in the etching of sp^3 hybrid carbon bonds which form the structure of diamond while etching sp^2 carbon bonds. In short, too high or too low substrate temperature will change the effects of the atoms of hydrogen and carbon combined groups on the substrate, affecting the diamond film deposition processes and making amorphous carbon in the thin films increase.

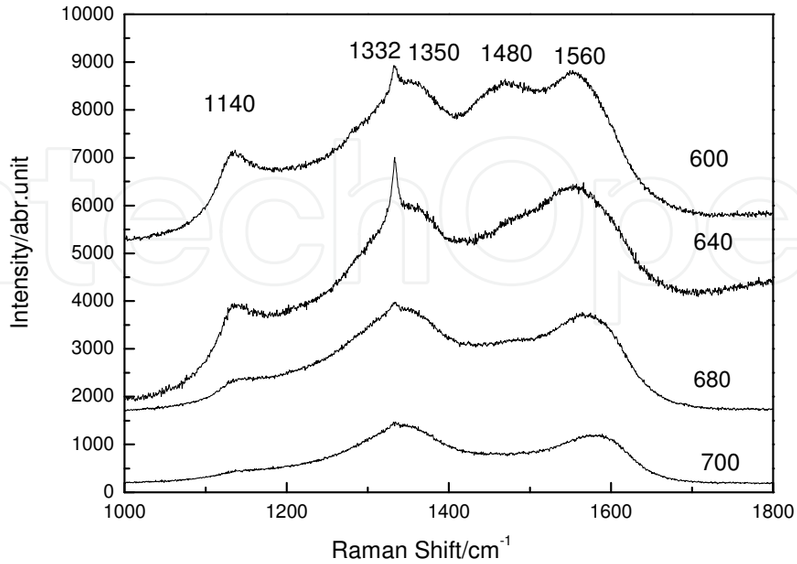


Fig. 2. Raman spectra of nano-crystalline diamond films under different deposition temperature

Optical transmittance of diamond films mainly lies on optical absorption of the film and light scattering on the film surface, grain boundary and film-substrate interface. The optical absorption of diamond films is caused by the imperfection of crystal lattice and impurities in the films, such as non-diamond-like carbon, hydrogen, nitrogen, etc, which can improve by adjusting the growth parameters and processes of CVD. Light-scattering loss mainly depends on the surface roughness of the films.

2.2 The effects of deposition pressure on the quality of NCD films

Selecting the appropriate pressure range is also one of the keys to prepare high-quality diamond films. To prepare NCD films, the reactant gas pressure is usually reduced. This is because, under the setted growth conditions, the nano-diamond particles are difficult to grow when becoming nuclei and the secondary nucleation rate in the film is very high. When preparing nano-crystalline diamond films, high concentrations of carbon source will make more carbon groups. At the same time, high H/H_2 is easy for nucleation. In addition, due to the low gas pressure, free path of the atoms is longer and the atoms will have higher energy. Their impacts with the growing diamond grains surface cause defects, leading to secondary nucleation while impeding normal growth of the original grains.

In this work, the deposition temperature is 640 °C.The NCD films were prepared by maintaining the constant gas flow rate (50/180 sccm) and changing the deposition pressure. SEM photographs of the diamond films surface synthesized at different pressure were depicted in Figure 3. It can be seen from Figure 3, with the gas pressure changes, the surface morphology of the films change significantly. When the pressure is 3kPa, the film surface is rough and the diamond crystals are well developed, indicating a low rate of secondary nucleation of diamond under this deposition condition. At the pressure of 2.5 kPa, the crystal grains of diamond film are no longer complete and there are many large grains surrounded by small particles. And compared with 3 kPa, the secondary nucleation rate increased significantly. When the pressure is down to 2 kPa, the film has a marked decline in grain size and film is nano-scale grain based, while its surface structure reaches nanometer scale. When the gas pressure is further reduced below 1.5kPa, the diamond grain size decreases even more evidently.

The above experimental results show that the reactant gas pressure have a very strong impact on the morphology and structure of the films. The concentrations of hydrogen atoms and carbon combined groups such as CH_2, CH_3, C_2H_2 in the reaction atmosphere as well as the energy state of these groups, play very crucial roles on the structure of the films. When the reactant gas pressure is high, the active hydrogen atoms and carbon-combined groups have short free path and low energy. They collide with the substrate and the generation of secondary nucleation is not easy, leading to the easy growth of diamond particles. When the reactant gas pressure is low, it will increase substrate temperature and H_2 dissociation rate. Meanwhile, the free path of various particles in the reaction chamber increase as well. These two factors will increase the velocity and energy that the particles reach the substrate. When the particles collide with the substrate surface, the energy are transferred to the surface. Thus the activity of the adsorbed particles is enhanced, making the carbon clusters form quickly on the substrate surface and improve the secondary nucleation rate. Besides, since the impacts of the particles on substrate reinforce, it's difficult for diamond grains to grow, resulting in increase of secondary nucleation rate.

Figure 4 shows RMS changes of NCD films under different deposition pressure. When deposition pressure is below 2.5 kPa, the surface roughness changes little. But when the

deposition pressure increase to 3 kPa,the surface roughness of the film increases. As the pressure increases, the growth conditions come close to micron diamond growth conditions. The grain size of the film gradually become larger and the surface become rough.

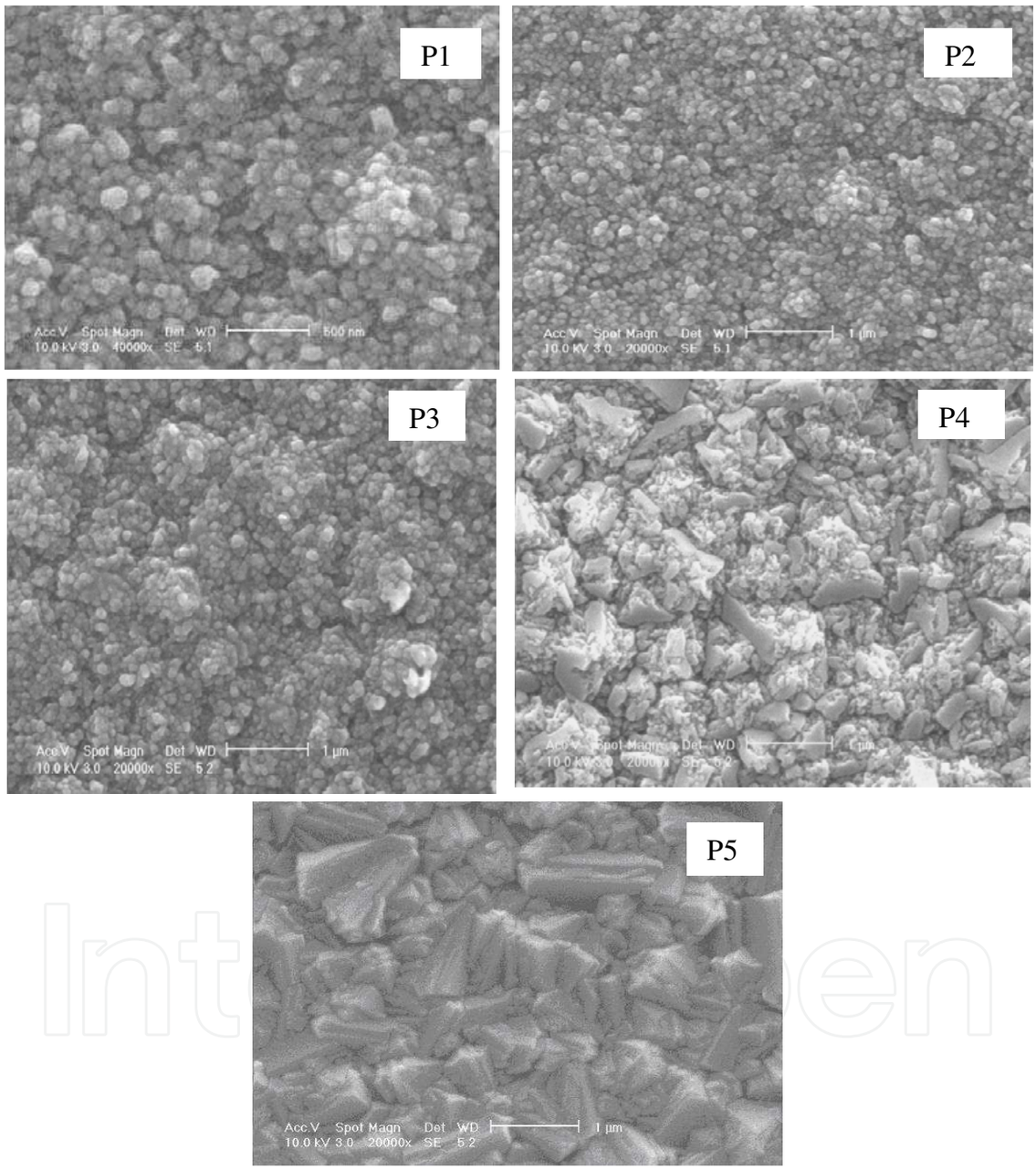


Fig. 3. SEM images of nano-crystalline diamond films under different deposition pressure (P1. 1 kPa, P2. 1.5 kPa, P3. 2 kPa, P4. 2.5 kPa, P5. 3 kPa)

Figure 5 is Raman spectra of diamond films deposited under different pressure. In the spectra of low-pressure synthesized nano-crystalline diamond film, there are two weak broadband scattering peaks around 1350 cm^{-1} and 1580 cm^{-1}, which are D peak and G peak.

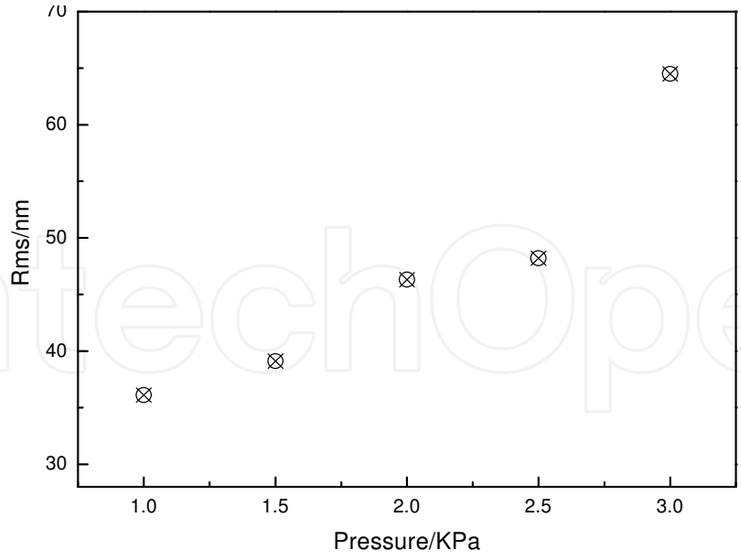


Fig. 4. RMS changes of nano-crystalline diamond films under different deposition pressure

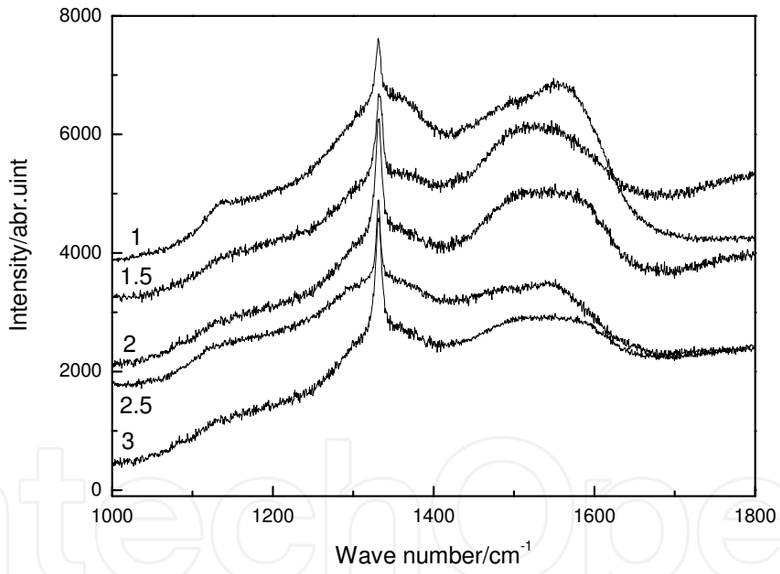


Fig. 5. Raman spectra of nano-crystalline diamond films under different deposition pressure

The diamond peak intensity of 1332cm^{-1} is small and wide. With the increase of gas pressure in the chamber, the scattering intensity of D peak and G peak gradually weakens while the the diamond peak at 1332 cm^{-1} becomes sharp. In addition, when the pressure reaches 2.5kPa, the band scattering background diminishes greatly in the range of 1100~1600cm^{-1}. The peak intensity of graphite decreases while the diamond peak is more clear, which shows good quality of the film.

In the 2 cm × 2 cm sample surface, paraffin was coated as a protective layer. In the middle, a circular window about 1 cm in diameter was left. Afterwards, it was put into the solution with HF: H_2NO_3 = 1:1. The silicon substrate was the corroded and a free-standing structure

of diamond film was formed. During the corrosion process, the uniformity and thickness of the paraffin coat and the ratio of etching solution must be noted.

Optical transmittance of NCD films under different deposition pressure are shown in figure 6. In Figure 6, when the deposition pressure is 2.5kPa, the transmittance of the thin film on has reached 42.37% at 632.8 nm. By the above analysis of the surface roughness, we can see that, when the pressure is below 2.5 kPa, the surface roughness of the samples changes little. Here the quality of the thin film is the main factor that affects the transmittance. At 3kPa, although the samples shows good quality in the Raman analysis and the absorption of impurities weakens, the roughness increases while the transmittance decreases slightly. Therefore, transmittance of the film lies on the quality and surface roughness.

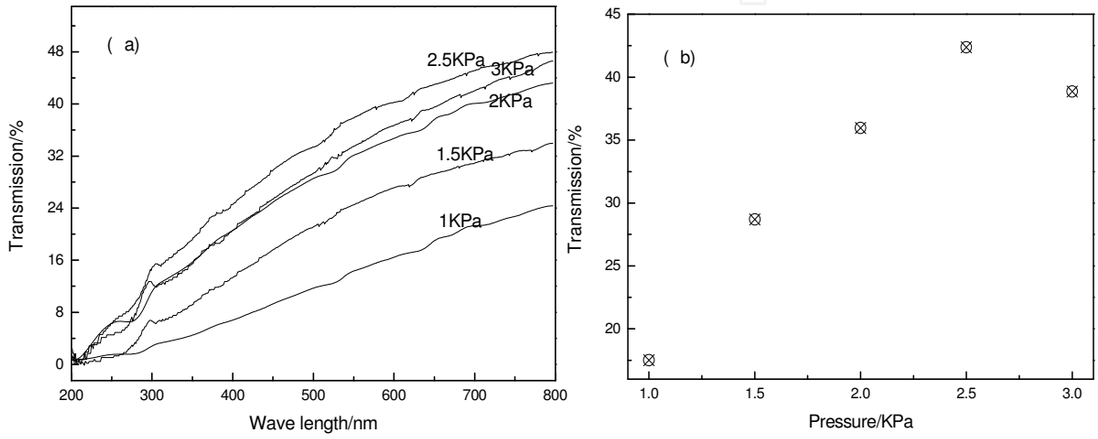


Fig. 6. Optical transmittance of NCD films under different deposition pressure
(a)The UV and visible band (b) Optical transmittance of the film at 632.8nm

2.3 The effects of etching on the electrical and optical properties of thin films

In CVD, the atomic hydrogen plays an important role in the diamond film growth process has been confirmed: atomic hydrogen stabilizes carbon with diamond structure while etching the carbon with graphite structure. The existence of hydrogen contributes to the growth of high-quality diamond films. It's commonly believed that hydrogen has a very important role which is the selective etching of graphite phase. Etching of H atoms for graphite and amorphous carbon are much stronger than for diamond. And this selective etching results in the non-equilibrium gas phase growth of diamond.

In this experiment, intermittent import of acetone gas and strengthening of hydrogen etching of graphite phase were applied to study improvement of the film quality during the process of the chemical vapor deposition (CVD) diamond film deposition. According to the parameters of the samples in previous experiments, the closing time and frequency of acetone were changed to increase the etching time.

In this work, the sample was deposited at pressure of 2.5Kpa, temperature of 640°C and unchanged total growth time. Sample A wasn't hydrogen etched throughout the deposition process. Sample B was etched for 15 minutes every half an hour and the total etching time was 1.5 hours. Sample C was etched for 15 minutes after 15 minutes and the total etching time was 3 hours.

Figure 7 shows surface morphology of the thin films under different eching time. Observed by SEM, without hydrogen etching, the film formed into a flake with large grain of about 120 nm. And with the increase of etching time, the grain of the film decrease. After etching for 3 hours, the grain of the film was reduced to 55 nm. As the grain size decreased, the surface roughness of the film increased. The SEM cross-section morphology of the films shows that the thickness of the samples is about 1μm, so it's unnecessary to consider the impact of film thickness on the optical properties.

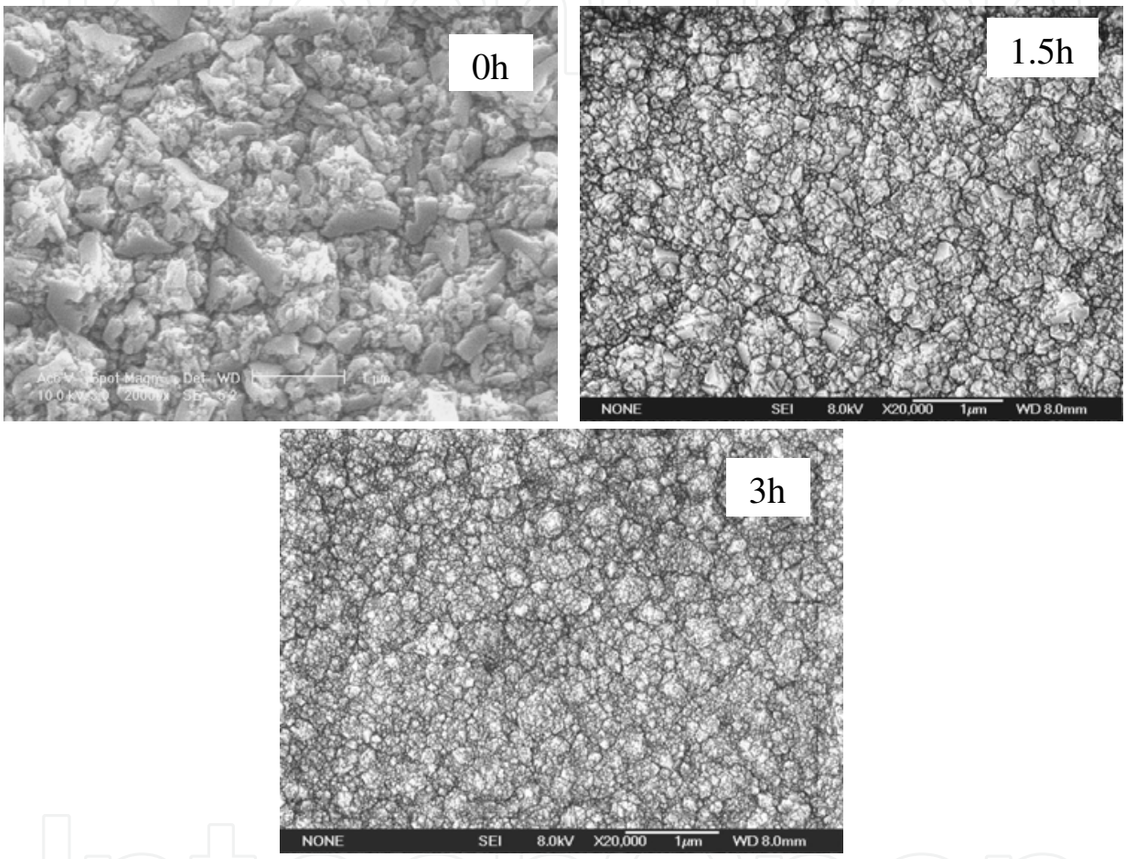


Fig. 7. Surface morphology of the samples under different eching time
(the etching time are 0 h, 1.5 h, 3 h, respectively)

The AFM images also shows that as the etching time increases, the diamond particles decreases, while the surface roughness of the film decreased gradually as well, from 47.2 nm to 14.8 nm. The phenomenon can be explained from the deposition mechanism: in the growth process of thin film, the positive ions and neutral groups in the chamber moves towards the substrate surface where the surface adsorption and reaction happens. Meanwhile, the hydrogen atoms in the chamber will etch the film. In the growth process, the energy of hydrogen atom is relatively low and the etching effect on the film is relatively small. Therefore the film obtained has more organic phase with relatively loose and rough surface. After strengthening the etching, the bombardment time of hydrogen atom is

enhanced, leading to the proliferation of surface atoms and the surface etching of the loose structure in the film-forming process. Thus the surface of the film is smooth and dense.

As can be seen from figure 8, after the strengthening of etching, the diamond characteristic peak begins to widen and the FWHM increases from 5.31 cm^{-1} to 27.88 cm^{-1}. By etching, the grain size of the film becomes smaller and smaller. In addition, as the etching time increases, the atoms bombardment increases the deposition temperature, prompting the transformation from sp^2 bonds to the sp^3 bonds. The intensity of the diamond peak increases with graphite peak decreasing. Id/Ig increases and content of sp^2 decreases. As the etching time increases, a scattering peak emerges at ~1130 cm^{-1}, which is usually referred to as the signature of NCD films. And this is widely cited in the literature. However, some recent studies show that this may be wrong as the location of this peak changes with the excitation photon energy used and the scattering intensity decrease with the increase of incident photon energy. This indicates the typical sp^2 hybrid characteristics and was identified as the C-C bond stretching (stretching) and twist (wagging) mode of polyacetylene (trans-polyacytylene) by Ferrari and Robertson.

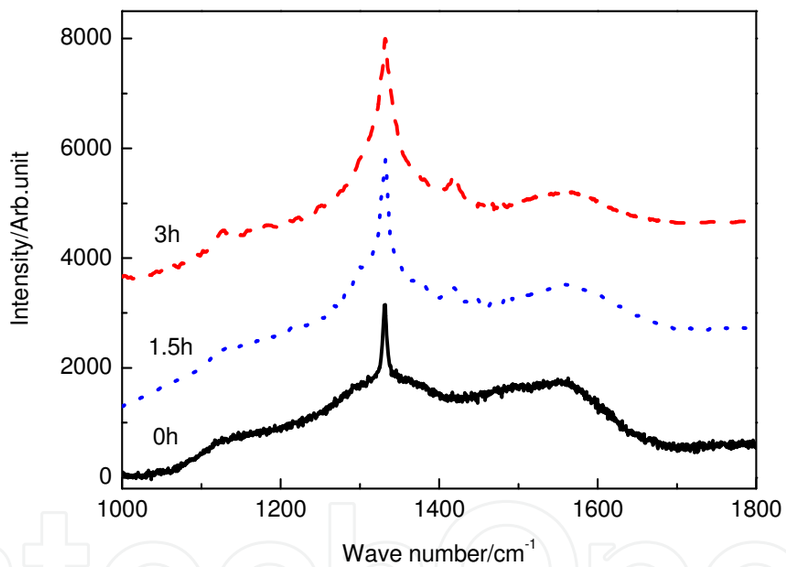


Fig. 8. Raman spectra of diamond thin films under different etching time

The prepared diamond thin films were processed into free-standing diamond films. Infrared spectrometer (LAMDA900) was used to measure the infrared spectra of the samples, which were shown in Figure 9. The measuring wavelength range is 500 ~ 2500nm.

Sample C with 3 h etching time, has the maximum transmittance and its transmittance is greater than the other two samples' in the whole band (500~2500nm). The transmittance curve is an oscillation curve in the low frequency due to the interference effect. This effect is caused by the multiple reflection beams interference, leading to a series of mini/max change on the transmittance spectra. Three samples have relatively large oscillation amplitudes in the whole wavelength region, indicating relatively smooth surface of the samples. In the short wavelength band, infrared light transmittance of the films decreases with the decrease

of the wavelength because of the scattering effects caused by surface roughness. However, in the long wavelength band, the scattering losses are not large and the losses of transmittance are mainly due to the absorption within the film, such as absorption of graphite.

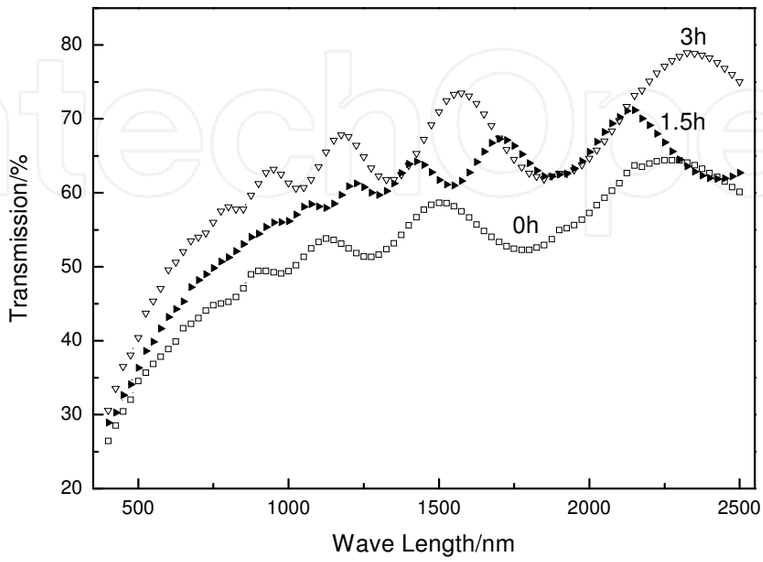


Fig. 9. Infrared transmittance of diamond thin films under different etching time

Sample A has the minimum infrared light transmittance and the diamond-related peak is also the weakest in the Raman spectra, indicating the absorption of non-diamond-like carbon in the film has a large impact on the infrared light transmittance of the sample. After 3 hours of hydrogen etching, the optical transmittance of the thin film at 632.8 nm reaches 51%. By etching, the quality of the thin film has been greatly improved.

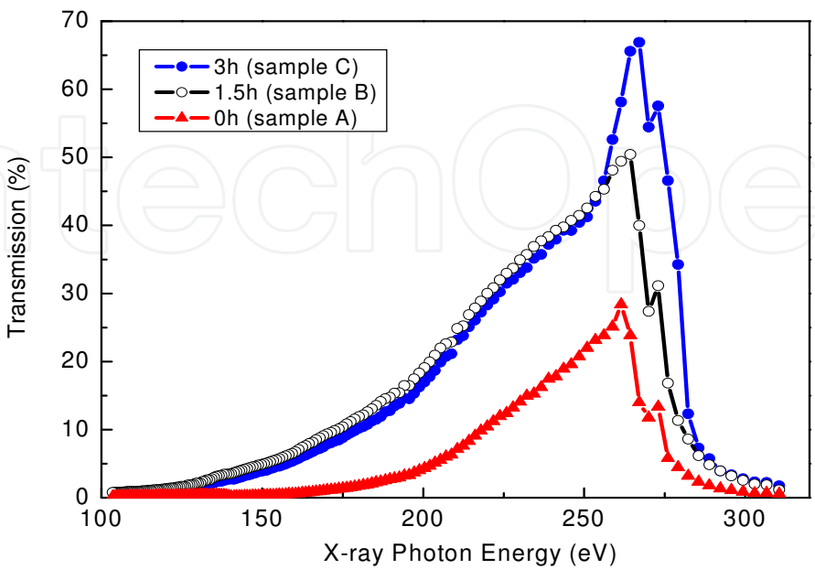


Fig. 10. Transmittance of diamond thin films in the soft x-ray under different etching time

Figure 10 shows the x-ray transmittance of diamond thin films under different etching time. The x-ray photon energy range is 100 ~ 310eV (a wavelength of 4 ~ 12nm). Taking the impact of the limited absorption on the film transmittance into account, we have chosen the transmittance at 258eV to measure the X-ray transmission characteristics. The transmittances of the three samples at 258eV are 52.7%, 47.8% and 24.8%, respectively while the traditional 8μm thick beryllium window in this energy is completely opaque. X-ray transmittance is not only related to the surface roughness, but also to the quality, structure and grain size of the film. By hydrogen etching process, the compactability of the film structure can be improved. Moreover, the internal defects (especially graphite content) and surface roughness decreases while the transmittance increases. However, etching can't make unlimited changes in quality of the film. The improvement of the film's performance is limited and the growth conditions are key factors in the quality of the film.

3. Conclusions

In this work, NCD films are prepared by HFCVD method. While maintaining the concentration of carbon source and changing the deposition parameters, such as reaction pressure, substrate temperature, NCD film with low surface roughness and high quality is deposited on silicon substrates. On this basis, hydrogen etching process was applied to further improve the quality and structure of the films. X-ray lithography mask-based material, whose optical transmittance and surface roughness meet the requirements of large integrated circuits (VLSI) was obtained. The main conclusions of the work are as below:

1. With the decreasement of deposition temperature (600^{o}C~700^{o}C) in the process of diamond thin films deposition, the secondary nucleation increased and the thin films grain size decreased. At the deposition temperature of 640^{o}C, NCD films with the higher diamond quality, the lower surface roughness and the higher optical transmittance in wavelength about 632.8nm was achieved.
2. The influence of deposition pressure variation on the NCD quality, structure and optical properties under the deposition temperature of 640^{o}C is investigated. With the increasement of deposition pressure, grain size of diamond grown up. At relative low deposition pressure, variation of surface roughness of thin films are not so obviously, but increased intensely at 3kPa. The transmittance reaches 42.37% at the wavelength of 632.8nm when deposition pressure is about 2.5kPa.
3. By using the fixed the deposition parameter, such as reaction pressure, substrate temperature, etc, and modifying the duration of hydrogen etch, the NCD films with low roughness and high optical transmittance are successfully deposited on silicon substrate. With the increasement of the duration of hydrogen etch (0~3h), the grain size decreased, and the surface roughness (RMS) reduced from 47.2nm to 14.8nm. The Raman scattering spectra indicated that as the etch duration increased, the contents of impure phase in the diamond thin films such as graphite decreased, the existence of hydrogen in thin films is propitious to the stability of sp3 bond, and could improve the quality of thin films in some extent.
4. The NCD films' transmittance for soft X-ray band at wavelength 4~12 nm has been measured using a synchrotron radiation device. It reveals that when grain sizes are

about nanometer scale, the transmittance of thin films is increased much intensely with the increasement of the particle size, and is slightly lowered with the variation of surface roughness. By modified the frequency and duration of hydrogen etch process, the influence of hydrogen etch on thin film transmittance of X-ray has been studied. With the increasement of etch duration, the transmittance of thin films are increased. After processed by hydrogen etch for 3 h, the transmittance of thin film at 258eV reaches to 52.7%.

5. In this work, the NCD film with low surface roughness and high transmittance of X-ray, which can meet the requirements of large integrated circuits (VLSI), is developed. However, for the optical transmittance of diamond films are depend on both surface roughness and film quality, there still need to further optimize the deposition process to simultaneously obtain smoothed surface and lower content of carbon sp2 bond, and then achieve the preparation of high quality optical window.

4. Acknowledgments

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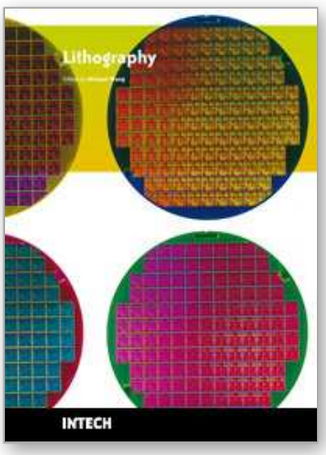
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Lithography, the fundamental fabrication process of semiconductor devices, plays a critical role in micro- and nano-fabrications and the revolution in high density integrated circuits. This book is the result of inspirations and contributions from many researchers worldwide. Although the inclusion of the book chapters may not be a complete representation of all lithographic arts, it does represent a good collection of contributions in this field. We hope readers will enjoy reading the book as much as we have enjoyed bringing it together. We would like to thank all contributors and authors of this book.

How to reference

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