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PHOTOLUMINESCENCE OF NANOCRYSTALLINE SiC FILMS PREPARED BY RF MAGNETRON SPUTTERING*

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Amorphous SiC films are deposited on Si (111) substrates by rf magnetron sputtering and then annealed at 1200°C for different times by a dc self-heating method in a vacuum annealing system. The crystallization of the amorphous SiC is determined by Raman scattering at room temperature and X-ray diffraction. The experimental result indicates that the SiC nanocrystals have formed in the films. The topography of the as-annealed films is characterized by atomic force microscopy. Measurements of photoluminescence of the as-annealed films show blue or violet light emission from the nanocrystalline SiC films and photoluminescence peak shifts to short wavelength side as the annealing time decreases.

Keywords: luminescence, SiC, nanocrystalline film, rf sputtering
PACC: 7855

I. INTRODUCTION

Silicon carbide (SiC) has many excellent properties, such as wide band gap, high breakdown voltage, high saturated electron drift velocity, and high thermal conductivity. It is stable at high temperature and can be used at high frequency.[1] Its wide band gap also renders it applicable for blue light emitting devices.[2] Unfortunately, its indirect band gap makes SiC hardly suited for optical device application although it has been used for blue light emitting diodes. The discovery of porous Si emitting strong light inspired research on the blue light emission of porous SiC. However, little work has been carried out so far on the photoluminescence (PL) of nanocrystalline SiC films.[3,4] In this work, we prepare nanocrystalline SiC films by rf magnetron sputtering and annealing treatment, and study the dependence of the photoluminescence of the SiC films on the annealing procedure.

II. EXPERIMENT

The n-type polished Si (111) wafers of 8mm× 18mm with a resistivity of 8–13Ωcm were used as substrates. Before deposition of an amorphous SiC film, the wafers were soaked into a solution of 5% hydrofluoric acid for 1min to remove the native oxide layer and then rinsed by deionized water. The specimen preparation consisted of two steps: deposition of the amorphous SiC film, then annealing of the specimens by a dc self-heating method.

An rf (13.65MHz) magnetron sputtering system was used to deposit amorphous SiC film. The target was a 99.9% SiC disc with 70mm in diameter. After the wafers had been put into the sputtering chamber and the base pressure had reached 2×10^{-3}Pa in the chamber, which was obtained by a rotary pump and a turbomolecular pump, highly pure Ar (99.999%) was introduced into the chamber as sputtering gas. The pressure was maintained at 3Pa by a mass flow controller. The sputtering power was 300W and the growth time of the films was 1 min and 5 min, respectively.

Amorphous films were crystallized in a vacuum annealing system. The as-deposited specimen was grasped tightly by Mo pieces at both ends and heating was conducted by a dc current passing through the specimen when the vacuum reached 1.2×10^{-4}Pa in the annealing system. During annealing, the speci-

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men temperature was measured with a pyrometer facing the rear of the wafer. The annealing temperature was 1200°C and the time was 1 min and 1 hour, respectively.

The Raman scattering spectra of the as-annealed SiC films were recorded at room temperature (RT) using an Ar ion laser with the 488 nm line as incident light. The crystallization of the amorphous SiC films was determined by X-ray diffraction (XRD) with the Cu$K\alpha$ radiation. Surface topography of the specimens was characterized using atomic force microscopy (AFM). Measurements of the photoluminescence of the films were performed with a He-Cd laser.

III. RESULTS AND DISCUSSION

During deposition of the SiC films, the sputtering power is maintained constant. Only the deposition time is changed in order to obtain different thick films. Table 1 gives the deposition and annealing conditions of the SiC films.

Table 1. Deposition and annealing conditions.

Specimens	Deposition conditions		Annealing conditions	
	Power	Time	Temperature	Time
AN5	300W	1min	1200°C	1min
AN7	300W	5min	1200°C	1min
BN7	300W	5min	1200°C	1h

Figure 1 shows the Raman scattering spectra of the as-annealed films AN5 and BN7 measured at room temperature. In the spectra the two peaks at 796 cm^{-1} and 967cm^{-1} are the transverse optical (TO) phonon peak and longitudinal optical (LO) phonon peak of SiC, respectively.[5] The Raman spectroscopic result suggests that the amorphous SiC films can be crystallized by annealing at 1200°C within 1min. However, for the AN5 specimen, the peak at 796cm^{-1} does not appear; this is due to the SiC film being too thin. Feng *et al.*[6] showed that no SiC phonon can be detected for samples with film thickness less than 0.4μm, and only when the film thickness increases to 0.6μm, the SiC LO phonon is recognizable above the background. The penetration depth of the 488 nm radiation for the SiC is about hundreds of μm and all penetrating light is quickly absorbed within about 1μm in the Si substrate. Hence, the very thin SiC film on the top of Si substrate absorbs very slight penetrating light, so that it cannot give a relatively strong Raman scattering light and some weak Raman scattering light is submerged in the background.

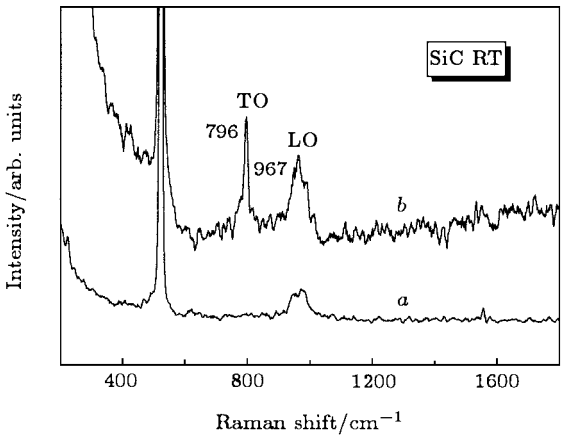


Fig.1. Raman scattering spectra of the SiC specimens AN5 (curve *a*) annealed at 1200°C for 1min and BN7 (curve *b*) annealed at 1200°C for 1 h.

The X-ray diffraction result of the BN7 specimen is shown in Fig.2. It is obvious that the SiC peak is at 35.7° (2θ). In terms of Scherrer formula, the average grain size in the BN7 specimen was calculated to be about 33nm.

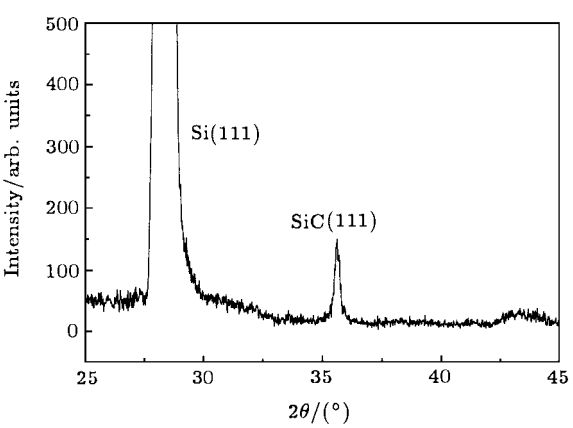


Fig.2. XRD result of the BN7 specimen annealed at 1200°C for 1 h.

Figures 3(a), 3(b), and 3(c) show the AFM surface images of the AN5, AN7 and BN7 specimens, respectively. From Figs.3(a) and 3(b), it can be seen that by annealing the amorphous SiC films for 1min, a large amount of little nanocrystalline islands have formed on the specimen surfaces. It can be also noticed that on the surface of the AN5 specimen nanocrystalline grain sizes are not uniform and two kinds of nanocrystalline grains are formed. On the surface of the AN7 specimen only one kind of nanocrystalline grain size appears. By annealing the BN7 specimen at 1200°C for 1 h, the labyrinth islands appeare due to the growth and connection of some islands. These islands have a

larger size than those on the surfaces of the AN5 and AN7 specimens, as shown in Fig.3 (c).

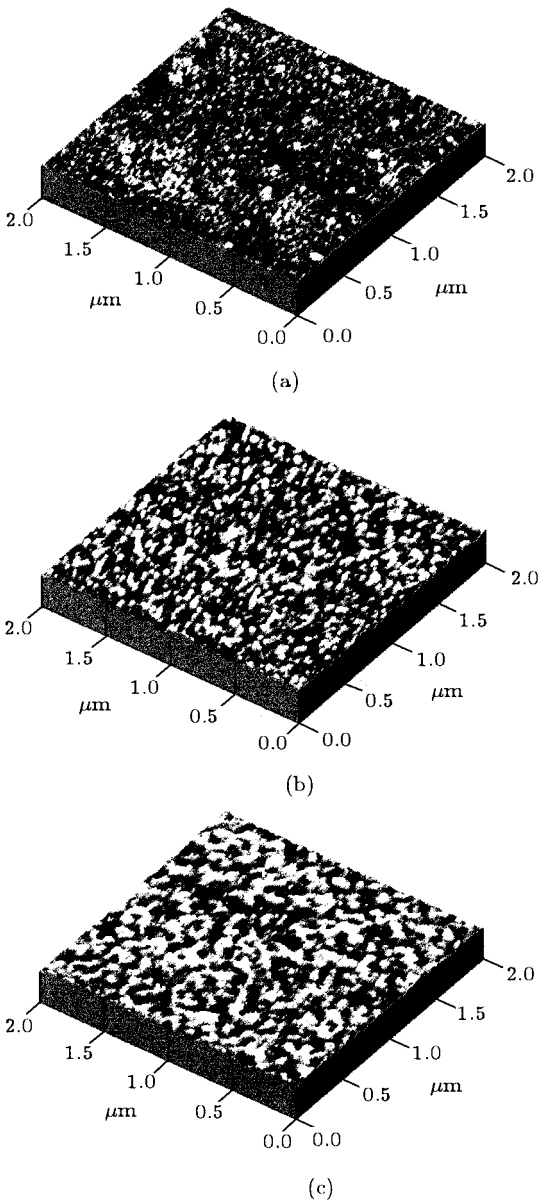


Fig.3. AFM images of the specimens AN5 (a) and AN7 (b) annealed at 1200°C for 1min and the BN7 specimen (c) annealed at 1200°C for 1 h.

Figure 4 shows the PL spectra of the as-annealed SiC films at room temperature. From the AN5 and AN7 specimens, we can observe violet light emission. But the PL spectra of the two specimens are different. For the AN5 specimen there are two peaks in its PL spectrum, one at 417nm and the other at 438nm. The PL spectrum of the AN7 specimen has one peak at 430nm. The BN7 specimen emits blue light and its PL peak is at about 474nm. It's PL intensity is weak and the spectral range becomes broad as compared with that of the AN5 and AN7 specimens. The origin of the blue or violet light emission from the different specimens is inferred to be the quantum confinement effect of nanocrystalline grains of SiC films because the SiC polycrystalline grains emit no visible light at room temperature. The occurrence of the two peaks in the PL spectrum of the AN5 specimen may be due to its nanocrystalline grains of two sizes, and the single-size nanocrystalline grains of the AN7 specimen are possibly responsible for the one peak in its PL spectrum. For the BN7 specimen the decrease in the PL intensity and the widening of the PL spectral range are considered to be connected with grain growth and the non-uniformity of grain size with increasing annealing time. It can be noticed that the PL peak shifts to short wavelength side, from blue to violet, with decreasing annealing time. The blue-shifting phenomenon may be attributed to the decrease of grain size.

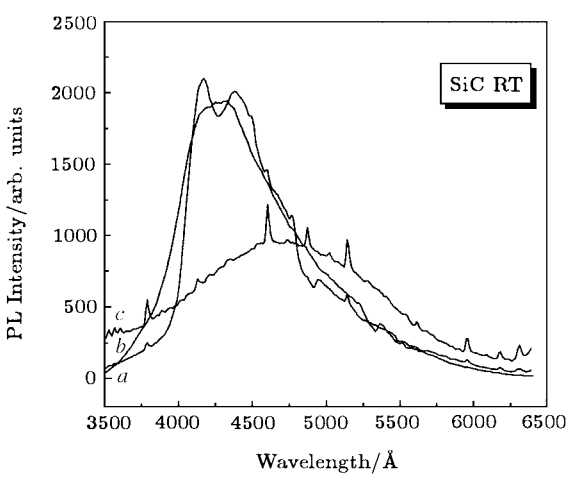


Fig.4. PL spectra of the as-annealed SiC specimens AN5 (curve *a*), AN7 (curve *b*) and BN7 (curve *c*).

IV. CONCLUSION

We have prepared nanocrystalline SiC films on Si (111) substrates by an rf magnetron sputtering and annealing treatment process. The as-annealed SiC films exhibit photoluminescence from blue to violet at room temperature with decrease of annealing time.

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