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Characterization of SiC Thin Films Deposited by HiPIMS

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In this work thin films of silicon carbide (SiC) were deposited on silicon wafers by High Power Impulse Magnetron Sputtering (HiPIMS) technique varying the average power of the discharge on a stoichiometric SiC target. X-ray diffraction, Raman spectroscopy, scanning electron microscopy and profilometry were used to analyze the films. It was observed that high values of the average electric power favors the formation of C-C bonds, while low values of the power promote the formation of Si-C bonds. At high power, we have also observed higher deposition rates, but the samples present surface imperfections, causing increase in the roughness and decrease in the film uniformity.

Keywords: *HiPIMS, thin film, silicon carbide*

1. Introduction

Silicon carbide (SiC) has properties such as chemical, thermal and electrical stabilities that allow its use in harsh environments[1]. SiC thin films have a wide range industrial applications, such as in coating tools[2] and in micro-electro-mechanical systems (MEMS)[3-5]. For applications in MEMS, the piezoresistive properties of SiC generate great interest because they have stable behavior at temperatures above 600 °C, while silicon is stable at temperatures below 175 °C.

Despite the use of crystalline silicon carbide (c-SiC) be more widespread in microelectronics, the amorphous form (a-SiC) is being increasingly studied[3,6-10], in order to reduce process steps, reduce costs and promote greater integration with microelectronics. However, to generate crystalline SiC films, high deposition temperature or thermal annealing after the deposition is required, and this can damage previous deposited materials, being impracticable the production of devices[11].

There are several methods for deposition SiC thin films, such as chemical vapor deposition (CVD)[12], plasma enhanced chemical vapor deposition (PECVD)[13], RF magnetron sputtering[14] or DC magnetron sputtering[15]. CVD and PECVD techniques have problems since the deposited films generally have higher oxygen contamination than films deposited by sputtering[6] and also the films are typically hydrogenated because the precursor gases (SiH_4 and CH_4) containing hydrogen[16]. Both DC and RF magnetron sputtering techniques, proved to be efficient for thin film deposition for application in pressure sensors[3,6]. In the last years, a new promising technique has been studied and presents interesting results for the production of SiC films[17]. In this technique, called High Power Impulse Magnetron Sputtering (HiPIMS), a low frequency (and low duty cycle) pulsed signal is applied to the target producing plasmas with high rate of gas ionization[18], and can be categorized as an ionized physical vapor deposition (IPVD) technique.

In this work studies about silicon carbide thin films deposited by HiPIMS at different values of average electric power were performed. The objective of the study is to establish deposition parameters that optimize the HiPIMS technique in order to produce SiC films useful in MEMS devices.

2. Experimental

2.1. *Samples*

SiC thin films were deposited on (100) n-type 1-10Ω. cm silicon wafers. The wafers were cleaned on hydrofluoric acid and then placed in the deposition reactor (Figure 1).

A high purity (99.95%) SiC target was used and the distance between the target and the substrate was maintained at 50mm. The depositions were performed at a pressure of 5mTorr and argon flow of 20sccm. Before each deposition, the gas line purge was carried out and previous argon sputtering of the target was done by 15 minutes. The HiPIMS power supply operated at 500Hz and pulse width of 100ms. The deposition parameters are shown in Table 1.

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Table 1. List of parameters of samples.

Samples	Averagepower (W)	Deposition time (min)	Peakcurrent (A)	Peakvoltage (V)
H1	100	60	0,2	652
H2	200	60	3,2	577
H3	300	60	6,9	583
H4	400	60	11,5	587

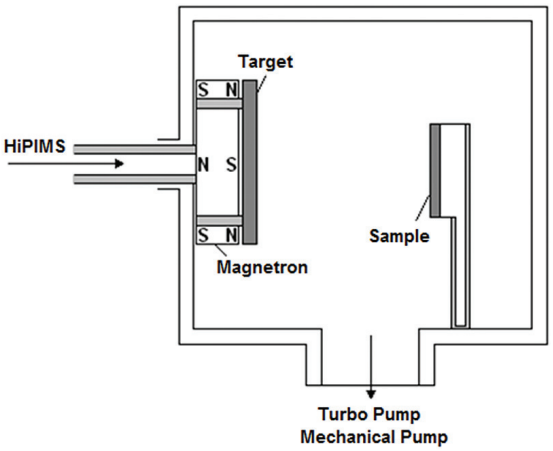

Figure 1. Schematic drawing of the deposition reactor.

2.2. *Characterization*

The films were characterized by X-ray diffraction (XRD), Raman spectroscopy, scanning electron microscopy (SEM) and profilometry.

X-ray diffraction was performed to determine the crystallinity of the film with a Philips PW1840 diffractometer with a l=1.54060 Å.

Chemical bonds of the film were analyzed by using a micro-Raman spectroscopy Renishaw 2000 with an Ar laser line of 514 nm.

A scanning electron microscope, performed with a SEM model EVO-MA10 (Zeiss) was necessary to observe the morphology of the films.

During the depositions, a piece of silicon wafer was placed on the sample in order to produce a step to determine the thickness of the deposited films with a mechanical profilometer model Tencor Instruments Alpha-Step 500.

3. Results and Discussion

SiC films deposited on the Si wafers were firstly analyzed by X-ray diffraction (XRD), as can be seen in Figure 2. It can be observed the presence of four peaks in all samples, identical to the silicon sample. Using Bragg's law and the l=1,54060Å and the lattice parameter of silicon (a=5,430Å) the 2θ values of silicon were calculated and compared with experimental diffraction angles, as indicated in Table 2. We can observe that the values in this table are very close to those shown in Figure 2, indicating that the observed peaks are related to crystalline silicon, thus there is a great indication that the deposited film is amorphous SiC. In the range of 10-30 degrees of the diffraction spectra a swelling could be possibly related to the amorphous nature of the deposited material.

Table 2. Results of diffraction angles of crystalline silicon calculated with Bragg's Law and the experimental values of diffraction angle.

h k l	n	2θ (Bragg's Law)	2θ (Experimental)
1 0 0	2	32,945°	32,84°
3 2 0	1	61,486°	61,77°
0 0 4	1	69,099°	69,77°
3 3 0	1	73,957°	74,32°

Table 3. List of positions of the Raman peaks of amorphous SiC films.

Line	Wavelength found	Wavelength of literature	Bonds
A	435 cm^{-1}	470 cm^{-1}[19]	Si-Si (a)
B	528 cm^{-1}	521 cm^{-1}[20]	Si-Si (c)
C-D	577-906 cm^{-1}	550-1000 cm^{-1}[21,22]	Si-C
E-F	931-1103 cm^{-1}	750-1100 cm^{-1}[20]	Si-Si (c)
G	1288 cm^{-1}	1300 cm^{-1}[23]	C-C (D band)
H	1480 cm^{-1}	1500 cm^{-1}[23]	C-C (G band)

In Figure 3 we can see the Raman spectra of the samples and in Table 3 the indication of the Raman peaks. From Figure 3 we can see the existence of the crystalline silicon band in the direction of the line B, which disappear with the increase of the average power of the discharge. This band is related to the silicon substrate and increasing the thickness of the films the peaks are no more visible. Analyzing the spectrum of the H4 sample we can observe the presence of D and G bands of the carbon located in the direction of the lines G and H, respectively, that are higher than the Si-C band, located between the lines C and D. Decreasing the power, we can see a decrease in relation between C-C and Si-C bonds, to almost the same value observed in the sample H1 spectrum. This means that the amount of carbon-carbon bonds is favored at higher powers, and then the intensity of the band corresponding to the silicon-carbon bond is greater for lower powers. The increase in the band of C-C bonds with the average power is probably a result of higher energy transfer occurred in the film deposition process. If a thermal annealing for crystallization of the sample was done, probably the samples deposited with smaller average power would require a smaller temperature to crystallize SiC. The Raman spectra also show the validity of the X-ray diffraction analyses about the amorphous SiC, since the degree of organization of the film is directly related to the peak width in the Raman[23] and in the case of the SiC peak, observed in all samples, can be verified that all of them have considerable width.

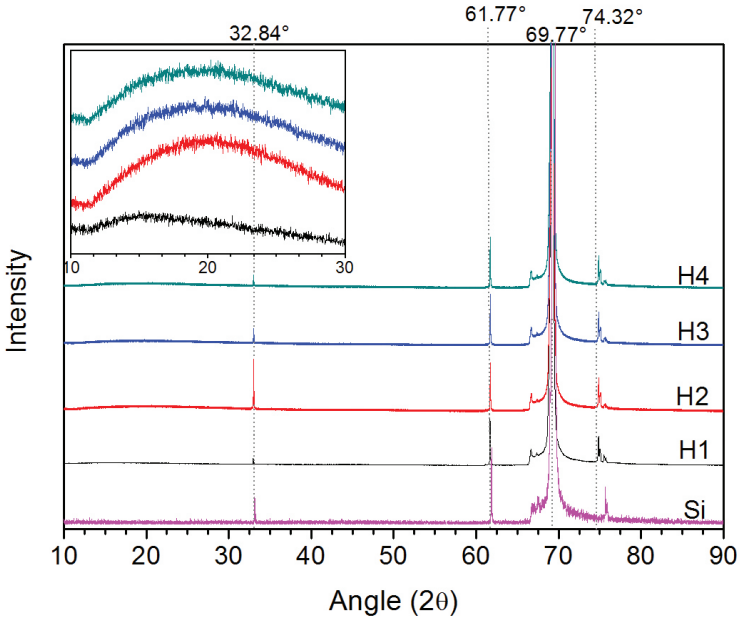

Figure 2. X-ray diffraction of H1, H2, H3, H4 and silicon sample.

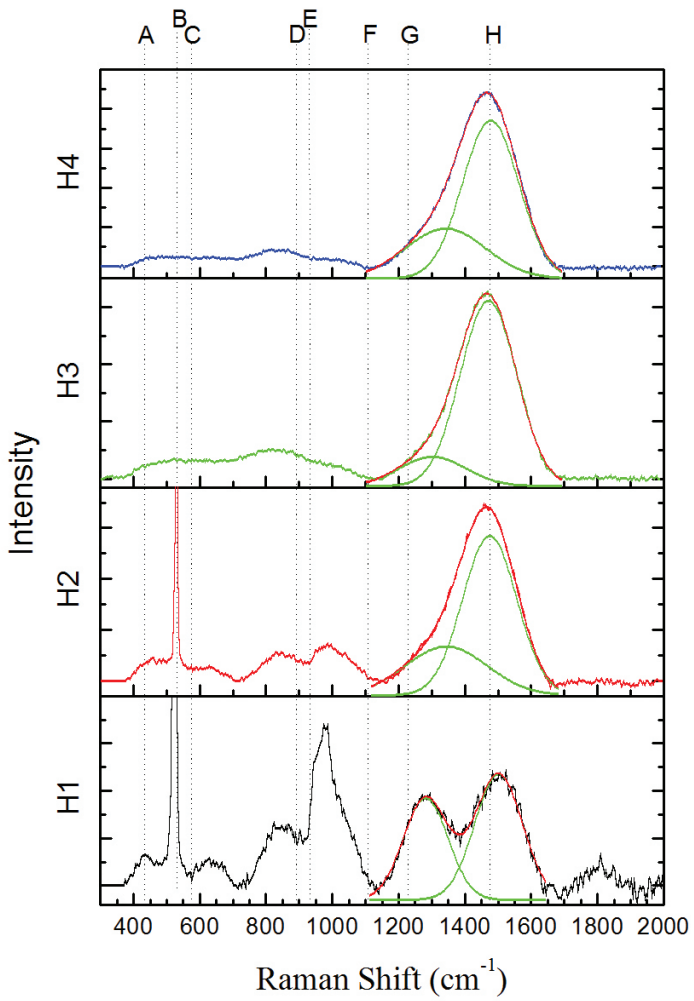

Figure 3. Raman spectra of H1, H2, H3 and H4 samples.

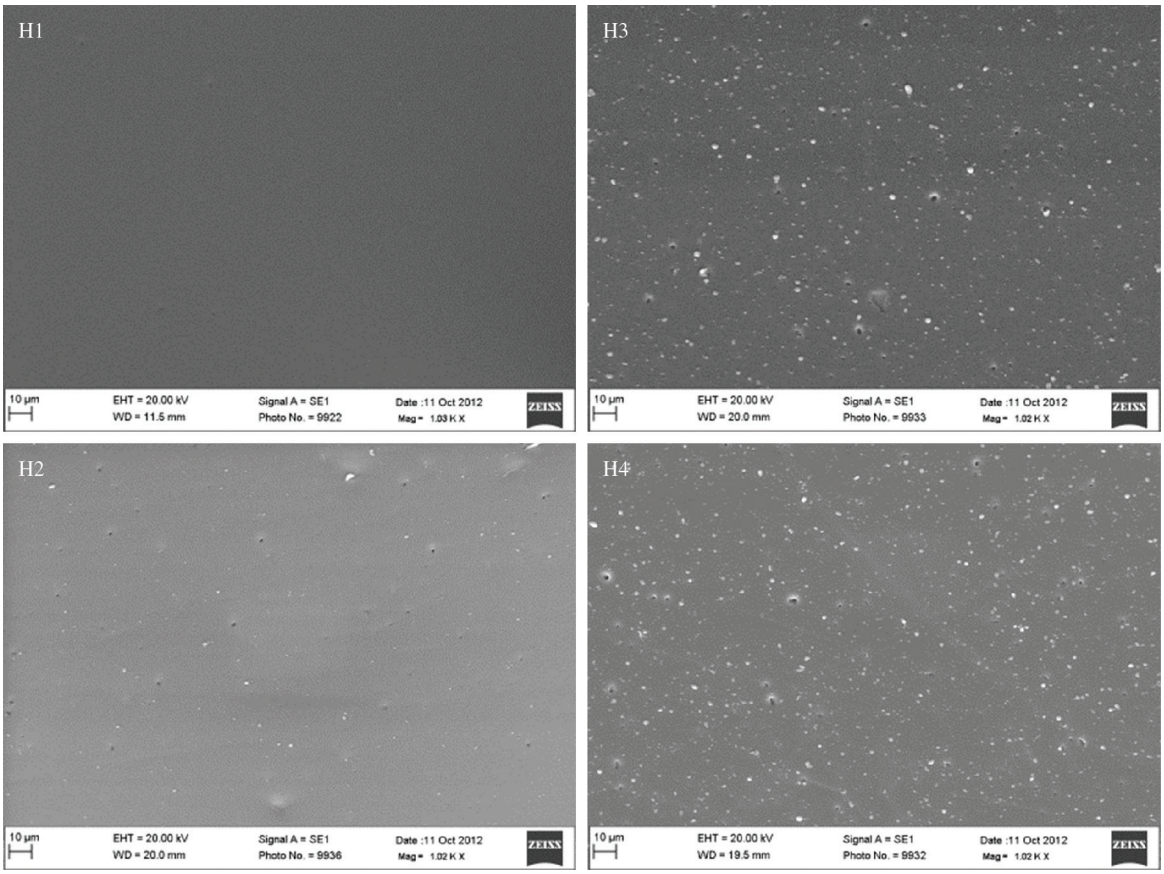

Figure 4. Scanning electron microscopy images of H1, H2, H3 and H4 samples, with 1000x magnification.

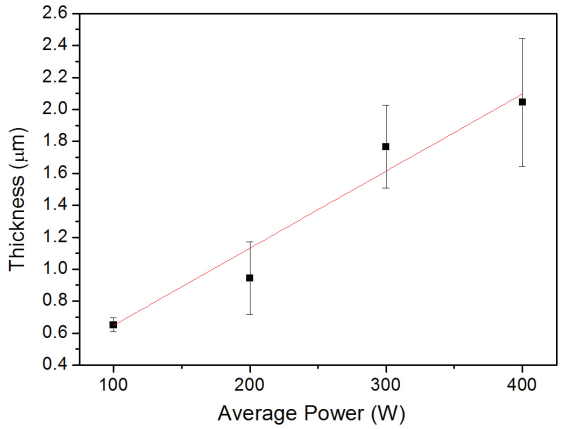

Figure 5. Graphic of films thickness by the average power deposition.

Figure 4 shows the images of the samples obtained with 1000x magnification by Scanning Electron Microscope. As can be seen, the increase in the average power promotes the formation ofnon-uniform films. It is possible to identify small "bubbles" on the surface of the films deposited at high power (300-400 W). This is probably caused by the high energetic particles that attack the film surface, causing the surface imperfections.

The profilometry results, Figure 5, show that the increase in average power causes a linearly increase in film thickness due to the increase in the sputter of the target. It can be also verified that the error of the analysis is higher for higher powers, indicating a rise in non-uniformity of the film. This variation in thickness was confirmed by the images shown in Figure 4.

4. Conclusion

According to the profilometry measurements, the film thickness increase almost linearly with increasing of the average power applied. By XRD and Raman analysis it can be seen that all the films are amorphous. According to the Raman spectra, lower power favors the formation of Si-C bonds while higher power is more favorable to the formation of C-C bonds. Furthermore, it was verified that imperfections arise on the surface of the film deposited at high power (300-400 W). Then, we can conclude that the films produced at lower values of average power are probably more appropriated to be used in MEMS devices.

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