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# STUDY OF THE MECHANICAL PROPERTIES OF a-SiC AND a-$SiC_xN_y$ THIN FILMS

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## ABSTRACT

*This work describes the mechanical properties of a-SiC and a-$SiC_xN_y$ thin films prepared by RF magnetron sputtering of a SiC target at different $N_2$/Ar gas flow ratios. Four different film compositions were studied. The elastic modulus and hardness of the films were determined by nanoindentation. Fourier transform infrared spectrometry (FTIR), Raman spectroscopy and atomic force microscopy (AFM) were used in order to investigate the influence de nitrogen content on the chemical bonds and morphology of the films.*

## 1. INTRODUCTION

Silicon carbide (SiC) and silicon carbon nitride (SiCN) thin films have been recognized as a promising material for high-temperature micro-electro-mechanical (MEMS) systems due to it attractive properties such as high hardness, high chemical inertness and large band gap [1-2]. These properties are strongly dependent of Si, C and N contents in the films and can be altered by modifying the deposition conditions. Several tehniques have been used to produce amorphous and crysttaline forms of SiC and SiCN thin films, including plasma enhanced chemical vapor deposition (PECVD) [3], sputtering [4], atmospheric pressure chemical vapor deposition (APCVD) [5], and pulsed laser deposition (PLD) [6].
As shown in previous studies, the area of research in mechanical properties of SiCN ternary material and of the binary such as SiC, SiN, CN compounds is fairly open [7-8]. Mechanical properties of these thin films have been quite studied due to facilities provided by nanoindentation technique [9]. This technique offers capabilities to measure hardness and elastic modulus of films with few nanometers thickness. In addition, in this technique the influence of the substrate is eliminated since the maximum indentation deep remains lower than 10-15% of the film thickness [10].
Although some studies have been published on the mechanical properties of $SiC_xN_y$ films, more investigations are needed to better understand the influence of nitrogen content on these properties. In this study, a-SiC and a- $SiC_xN_y$ films have been deposited on p-type Si (100) substrates by RF magnetron sputtering at different $N_2$/Ar flow ratios (r= 0, 0.1, 0.2, 0.3).
Correlations between mechanical properties and the nitrogen content in the films have been evaluated by nanoindentation. We also studied the influence of nitrogen content on the morphological and structural properties of the films.

## 2. EXPERIMENTAL PROCEDURE

Amorphous SiC and $SiC_xN_y$ films were deposited onto p-type (100) Si substrates by RF (13.56 MHz) magnetron sputtering of a SiC target (99.5% purity) in an argon-nitrogen plasma. The target-substrate distance was fixed at 75 mm. Different film compositions were obtained by varying the nitrogen flow rate while the other parameters were kept constant**.** Table 1 summarizes the deposition conditions.

**Table 1 – Deposition conditions of the films**

| Parameter | Value |
|---|---|
| $N_2$ flow (sccm) | 0 to 2,1 |
| Ar flow (sccm | 7,0 |
| RF power (W) | 200 |
| Working pressure (Torr) | $2 \times 10^{-6}$ |
| Time (min) | 120 |

The chemical bonding of the films have been investigated by Fourier transform infrared spectrometry (FTIR) and Raman spectroscopy. Infrared spectroscopy was performed by a Perkin Elmer spectrum 2000 Fourier transform infrared spectrometer in the range 400–4000$cm^{-1}$. A (100) silicon wafer was used as reference. Raman spectroscopy was performed with a Renishaw 2000 system using an $Ar^+$-ion laser ($\lambda$ = 514 nm) with power 0.6 mW. Raman spectra were obtained at room temperature in the range of 400 to 2000 $cm^{-1}$. AFM images were obtained by a SPM-9500J3 system in dynamic mode. The thickness of each sample was obtained by measuring step heights using profilometry (TENCOR Alpha-Step 500 profilometer).
Elastic modulus and hardness of the films were determined by nanoindentation technique using a Hysitron triboindenter. In this technique, a controlled load is applied to a diamond tip in contact with the surface. The continuos mea-

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surement of the tip displacement as the applied allowed the construction of load-unload curves.

## 3. RESULTS AND DISCUSSION

### 3.1 CHEMICAL BONDING

Figure 1 shows transmission FTIR spectra of a-SiC and a-$SiC_xN_y$ thin films in the range of 400 – 4000 $cm^{-1}$. As can be observed, the a-SiC film (sample B0) presents bands at around 841 $cm^{-1}$ and 1108 $cm^{-1}$ that associated to Si-C and Si-O stretching vibration modes respectively.

These vibrational modes also appear in the spectra of all samples of a-$SiC_xN_y$ films. In addition, the sample B3 deposited under higher nitrogen flow also present bands at around 1200 $cm^{-1}$ and 1540 $cm^{-1}$ corresponding to Si-N and C-N bonds respectively.

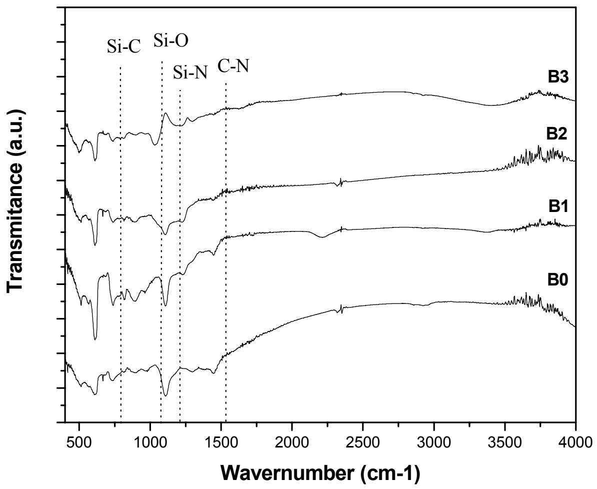



**Figure 1 - FTIR spectra of the SiC film (sample B0) and a-$SiC_xN_y$ films deposited at different $N_2$/Ar flow ratios.**

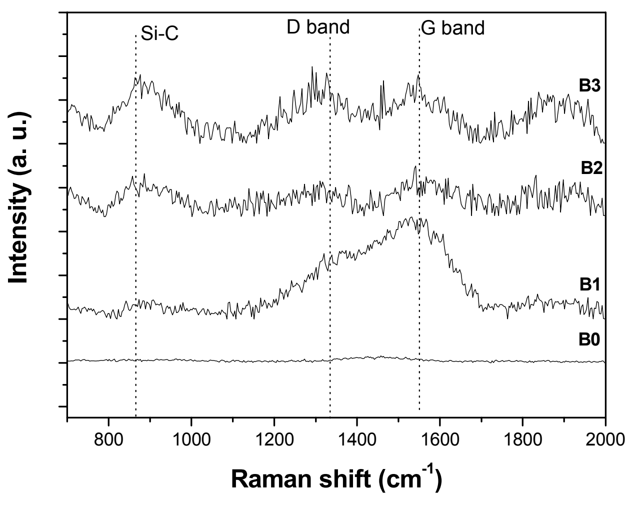



**Figure 2 - Raman spectra of the SiC film (sample B0) and a-$SiC_xN_y$ films deposited at different $N_2$/Ar flow ratios.**

Additional information about chemical bonds can be obtained from Raman spectra of the films shown in Figure 2. Raman spectrum of the SiC film (sample B0) does not present evidences of any peak corresponding to Si-C or C-C bonds. On the other hand, the $SiC_xN_y$ films present a relatively weaker band around 830 $cm^{-1}$ which is typical of amorphous SiC structure and show intense and broader bands, centered around 1350 $cm^{-1}$ and 1580 $cm^{-1}$, that are characteristics of C-C bonds recognized as D and G band, respectively.

Some studies [8,11] show that the existence of G band in $SiC_xN_y$ films indicate the formation of $CN_x$ phases.

### 3.2 THICKNESS, ROUGHNESS AND SURFACE MORPHOLOGY

The thickness and roughness of the a-SiC and $SiC_xN_y$ films were determined by profilometry and atomic force microscopy (AFM) respectively. Figure 3 shows the results obtained as a function of nitrogen flow rate.

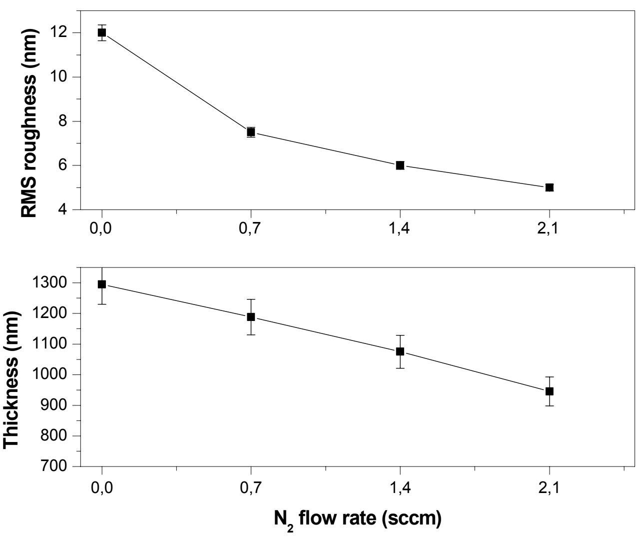



**Figure 3 - RMS roughness and thickness of the a-SiC and a-$SiC_xN_y$ films as a function of $N_2$ flow rate**

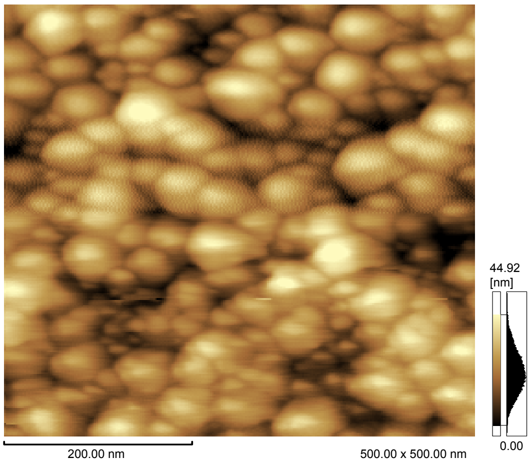
It was observed that the addition of nitrogen in the sputtering process promotes a reduction in the RMS roughness and an increase in the thickness of the films.

AFM images of the films (see Figure 4) indicate that the $SiC_xN_y$ films are much smoother than SiC film. This can be understood by the fact that the films are formed by a reactive process. Therefore, higher nitrogen flow rate increases the reactive process allowing the formation of denser films with fewer voids.

### 3.3 MECHANICAL PROPERTIES

The nanoindentation technique is widely used approach for the determination of hardness and elastic modulus of thin films, which can be obtained through an interpretation of experimentally obtained load–displacement curves during the indentation load /unload cycle [12–13].

**(a)**




**(b)**

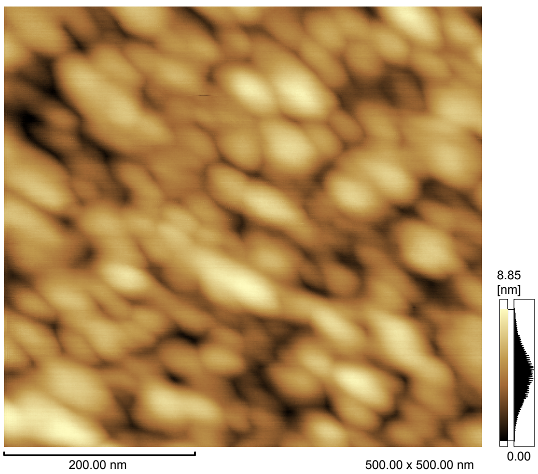



**Figure 4 - AFM images of the surface of: (a) a-SiC film (sample B0) and (b) a-SiC$_x$N$_y$ films (sample B2).**

In this work, nanoindentation testing with a Berkovich diamond indenter (a three-sided pyramid) was used to measure the reduced elastic modulus (E) and hardness (H) of the a-SiC and a-SiC$_x$N$_y$ films. The physical principles to determine E and H are based on the Oliver-Pharr theory [14].
The fundamental relations to calculate hardness and reduced elastic modulus are:

$$H = \frac{P_{\max}}{A} \tag{1}$$

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \tag{2}$$

where $P_{max}$ is the peak load, S is the parameter known as the elastic contact stiffness and A is the projected contact area at that load that for a Berkovich tip is defined by:

$$A = 24.5 h_c^{\,2} \tag{3}$$

where $h_c$ is the final plastic contact depth.

The elastic modulus of the material, E, is then calculated from $E_r$ by the expression:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^{\,2}}{E_i} \tag{4}$$

where $\nu_i$, $\nu$, $E_i$ and E are the Poisson's ratio and Elastic modulus of the indenter and sample, respectively. In this study, we used a diamond identer with $E_i$ = 1141 GPa and $\nu_i$ = 0,07.

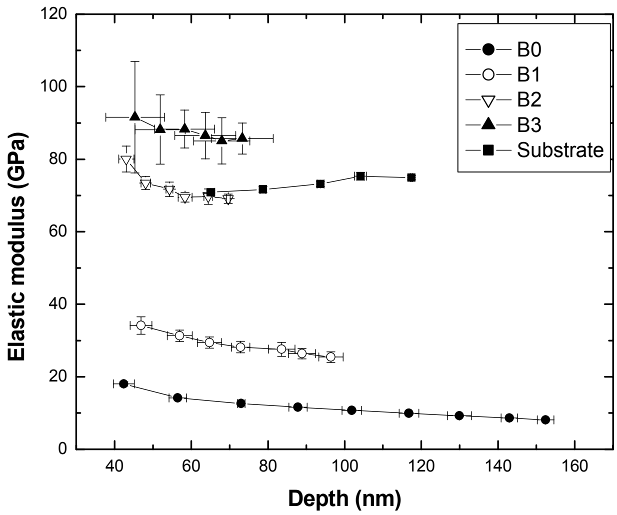



**Figure 5 - Elastic modulus of of SiC film (sample B0) and a-SiC$_x$N$_y$ films as a function of indentation depth.**

Figures 5 and 6 show the elastic modulus and hardness values of the a-SiC and a-SiC$_x$N$_y$ films as a function of indentation depth. The values obtained indicate that the hardness and elastic modulus of the films increase with nitrogen flow rate increases. This increase is due to formation of C-N and Si-N bonds in the films.

## 4. CONCLUSION

The mechanical properties of a-SiC and a-SiC$_x$N$_y$ thin films deposited by RF magnetron sputtering have been investigated. Hardness and elastic modulus were determined by nanoidentation, an increase in their values was observed as a function of nitrogen flow rate increases. The results indicate that the films had a range from 3.8 to 9.2 GPa for the hardness and 17 to 92 GPa for the elastic modulus when the $N_2$

flow rate is varied from 0 to 2.1 sccm. FTIR and Raman spectra indicated the existance Si-C, Si-N and C-N phases in the films. AFM analysis showed that the films become smoother as the nitrogen flow rate ratio increases.

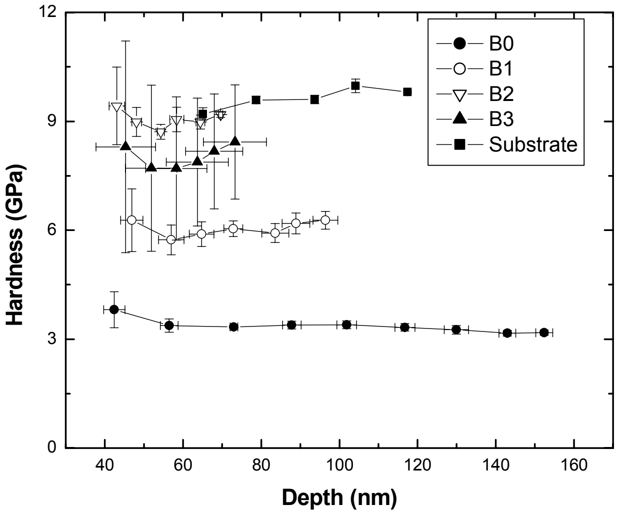



**Figure 6 - Hardness of SiC film (sample B0) and a-$SiC_xN_y$ films as a function of depth.**

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