Feasibility of Etching Studies on BCN Thin Films
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Investigation of wet chemical etching Boron carbon nitride (BCN) thin films deposited by dual magnetron sputtering of B$_4$C (DC) and BN (RF) targets was conducted. BCN, a low-k dielectric material, is a potential candidate as inter-layer dielectric (ILD) in VLSI process. A common aluminum etchant consisting of phosphoric acid (H$_3$PO$_4$), nitric acid (HNO$_3$) and acetic acid (CH$_3$COOH) was tested for its feasibility as a good etchant for BCN thin films. The etching studies were performed on BCN films that were deposited at room temperature, 200°C and 300°C as a function of various N$_2$/Ar gas flow ratios in an rf sputtering technique. It was found that the film deposited at higher temperatures shows lower etching rate trends. Activation energies ($E_a$) were calculated for each N$_2$/Ar gas flow ratio with the help of Arrhenius plots and compared with respect to elemental compositions of the films.

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Manuscript submitted March 11, 2016; revised manuscript received May 5, 2016. Published May 17, 2016. This article is a version of Paper 2028 from the San Diego, California, Meeting of the Society, May 29-June 2, 2016.

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Boron carbon nitride compounds are emerging as the potential candidates of great technological importance due to their favorable electronic, mechanical and optical properties for various device applications.1,2 BCN films with high density, hardness and Young’s modulus in the range of 2.0–2.5 g/cm$^3$, 5–13 GPa and 110–150 GPa, respectively, have been reported.3 Umeda et al. were able to deposit non-porous BCN films with dielectric constant as low as 1.9.3 A low dielectric constant (i.e. low-k value) of 2.13 with high breakdown strength, resistivity of 3.4 MV/cm and 3E12 Ω·cm have been reported which strengthens the claim that BCN is a robust material in terms of device processing in VLSI integration.6 The burning issue is integration of uniform, thin low-k film into manufacturing processes like damascene structures for interconnections.7 It has been demonstrated that copper diffuses faster in oxide based low-k films.8 This creates serious reliability problems. Recently, we demonstrated that BCN is robust to the copper diffusion even at higher annealing temperatures of 400°C.9 BCN with high density and low porosity is expected to be a good candidate as ILD.

The technique, etch rate and selectivity to the low-k materials and the characterization of the by-product during etching have attracted great attention to researchers and scientists recently. BN was tested as a potential low-k material before BCN. BN films deposited by plasma-assisted chemical vapor deposition (PACVD) have reliability issues like cracking and film peeling off the substrate as reported by Kimura et al. It has been found that adding carbon atoms to BN films is effective in resolving these issues.10 Device processing requires steps such as wet chemical etching, electro chemical plating and chemical mechanical polishing (CMP). Watanabe et al. established that BCN film with lower oxide composition is robust to such wet chemical influences.11 In case of similar materials like boron nitride (BN), the sp$^3$ bonds are preferentially etched than the sp$^2$ ones.12

Based on the limited knowledge of different etching techniques of BN and B$_4$C thin films, further investigation of BCN etching is conducted. Various dry etching techniques have been employed like using C$_2$F$_6$ gas to etch methyl-BCN.7 Samuel et al. reports advancements in dry etching techniques like inductively coupled plasma using SF$_6$ on h-BN films.17 Nguyen et al. demonstrated the magnetron enhanced reactive ion etching (MRIE) system using Freon/Oxygen (CF$_4$/O$_2$) and found out that the etch rate increases with increase in CF$_4$ gas flows.14 Dry plasma etching technique like reactive ion etching (RIE) is highly anisotropic and furthermore it can cause surface damage by ion-bombardment.15,16 While fluorinated plasma etching is still the mainstay for plasma etching for patterning applications in the semiconductor industry, sometimes wet chemical etching complements dry etching for processes like cleaning and complete removal of deposited BCN on undesirable places. Further, for some specific applications where large line width patterning is required, the wet etching process may be more viable and inexpensive. Watanabe et al. studied the influence of wet chemicals which are used for interconnection integration on BCN. They studied the effects of various chemicals used for post CMP processing and cleaning after dry etching like oxalic acid, dilute hydrofluoric acid (HF), trimethylammonium hydroxide (TMAH) and ammonium hydroxide on BCN thin films with different oxide concentrations.15 However, they did not focus holistically on the wet chemical etching techniques for BCN films. As BCN thin film wet etching studies are seldom reported elsewhere, the present work reports a detailed analysis on wet etching of BCN thin films.

As BCN thin films are not so reactive, a very good oxidizing agent with better standard reduction potential (SRP) should be considered. The etchant candidate should be such that it should seldom interfere with other components of fabrication like silicon, silicon dioxide or silicon nitride. One of the good candidates would be the common Piranha etch, which is comprised of sulfuric acid (H$_2$SO$_4$) and hydrogen peroxide (H$_2$O$_2$) in the ratio of 7:3 respectively. Our group tried using freshly prepared Piranha etch at 50°C, 60°C and 70°C and found that it barely etched BCN even for 15–20 minutes duration. Also, the preparation of Piranha etch is highly exothermic which may result in high temperatures of around 110–130°C.17 The SRP’s of H$_2$O$_2$ (in acidic medium), and H$_2$SO$_4$ are 1.776 V and 0.172 V respectively.18 The high oxidizing characteristics and high reaction temperatures can sometimes oxidize the silicon, silicon nitride and other components during device interconnect fabrication. The SRP of nitric acid (HNO$_3$) is 0.934 V,19 which is a moderate one compared to other acids. Nitric acid oxidizes BCN into B$_2$O$_3$. A good solvent is needed to dissolve B$_2$O$_3$ so as to increase the overall etching rate. Phosphoric acid (H$_3$PO$_4$) is good at dissolving B$_2$O$_3$ and acetic acid (CH$_3$COOH) provides a good buffer for wetting the substrate. Hence a common aluminum etchant comprising of HNO$_3$, H$_3$PO$_4$ and CH$_3$COOH is used as a viable, safe, economic option and an effective etchant at various temperatures.

**Experimental**

BCN thin films were deposited by dual target sputtering of B$_4$C (DC gun) and BN (RF gun) in an ultra-high vacuum (UHV) system. The system base pressure was approximately 4 × 10$^{-7}$ torr. Nitrogen was used as the reactive gas in the ambient of argon. Powder pressed targets of 99.5% purity and diameter of three inches were used. The N$_2$ to Ar gas flow ratio was increased from 0.25 to 1, in steps of 0.25. Individual gas flow rates were varied, while total gas flow was kept constant at 20 sccm at a deposition pressure of 5 mTorr. Room temperature, 200°C and 300°C were different substrate deposition temperatures.
temperatures used for each gas flow ratio. The R. F. power for the BN target was maintained at 250 W while the DC power of 20 W was used for B$_4$C target during sputtering. The thicknesses of the deposited films were measured by Veeco Dektak-150 profilometer.

P-type (100) silicon was used as the substrate. The substrates were cleaned by rinsing with acetone, methanol, deionized water (DI) and dried with nitrogen. These samples were loaded in the sputtering system to deposit BCN films. The target was placed at an angle of 45° facing the substrate holder. The rotation speed of the substrate was set around 20 rpm to get uniform films. The sputtering duration for all the samples was 1 hour. The BCN thicknesses ranged from 900 Å–1500 Å. After film deposition, the substrates were cut into pieces of 1 cm × 1 cm area each.

A basic aluminum etchant was used for the etching studies of BCN thin films. The etchant consisted of 16 parts of phosphoric acid (H$_3$PO$_4$), 1 part of nitric acid (HNO$_3$), 1 part of acetic acid (CH$_3$COOH) and 20 parts of DI water. 40 ml of freshly prepared aluminum etch was filled into a small glass beaker of 100 ml capacity. The glass beakers were submerged in a water bath to control the uniformity of the etching temperature. Etching was performed first at room temperature, but the etching was very slow. Hence the temperature was increased and etching was performed at three different temperatures, i.e. a) 50 °C, b) 60 °C and c) 70 °C. The temperatures were monitored with the help of a glass thermometer. Duration of complete etching of the film was monitored by stopwatch. As BCN is hydrophilic in nature, the DI water was used to rinse the etched samples to check whether the sample would turn completely hydrophobic and silver-gray in color denoting the silicon surface.

XPS measurements were performed using a PHI 5400 ESCA system to ascertain the chemical composition of the freshly prepared BCN films. The base pressure during analysis was 10$^{-9}$ Torr. The Mg K$\alpha$ X-ray source (hv = 1253.6 eV) at a power of 350 watts was used for the analysis. The survey and high-resolution spectra were recorded with an electron pass energy of 44.75 eV and 35.75 eV, respectively, to achieve the maximum spectral resolution. Any charging recorded with an electron pass energy of 44.75 eV and 35.75 eV, respectively, to achieve the maximum spectral resolution. Any charging was carefully removed by using C (1s) line shapes. The oxygen composition was found to be less than 5%.

The survey and high-resolution spectra were used for all the samples. Therefore, all the elemental compositions were used for the analysis. The base pressure was 10$^{-9}$ Torr. The Mg K$\alpha$ X-ray source (hv = 1253.6 eV) at a power of 350 watts was used for the analysis. The survey and high-resolution spectra were recorded with an electron pass energy of 44.75 eV and 35.75 eV, respectively, to achieve the maximum spectral resolution. Any charging was carefully removed by using C (1s) line shapes. The oxygen composition was found to be less than 5%.

Results and Discussion

XPS analysis.—Table I shows the percentage elemental compositions of B, C, N and O in BCN films that were deposited at various N$_2$/Ar gas flow ratios and substrate deposition temperatures. During the deposition process of the BCN films, B and C can form compounds reacting with N or with each other. Therefore, the process of N integration and the reciprocity among B, C and N during the deposition become the main factors, which affect the composition and properties BCN films. According to the literature,20,21 many XPS analyses report that, the BCN chemical composition can either be a stable ternary BCN and/or a mixture of CN and BN phases.22,23 Lei et al. reports that B-C can exist in two different phases namely, B$_4$C and BC$_3$, depending on the film’s boron and carbon compositions.24 The BCN films deposited at room temperature, in Table I show an overall carbon rich film with low boron concentration. Hence, we can deduce that as-deposited BCN films may be dominated by a mixture of CN, BN and BC$_3$ phases. The films deposited at 200 °C show boron rich films with low nitrogen concentration. These films can be assumed to consist of CN, BN, and BC$_3$ phases.

Usually, wet etching involves oxidation of the surface and dissolution of the resulting oxides.25 The CH$_3$COOH helps in buffering and wetting. It is considered as a good industrial solvent. HNO$_3$ is a strong oxidizing agent which reduces BCN into B$_2$O$_3$. The proposed reaction is given in equation 1. According to literature, hot H$_3$PO$_4$ is said to dissolve BN.26 Hence, it can be considered as an additional oxidizing agent to reduce BCN as well. DI water is used for dilution. It is also found that, hot water dissolves B$_2$O$_3$ into metaboric acid (HBO$_2$) as shown in the Equation 2. Films deposited by sputtering may result in BCN and boron carbide (B$_4$C) and boron nitride (BN). According to the literature, B$_4$C is insoluble in acids but soluble in fused salts and alkaline hydroxides like potassium hydroxide (KOH).27 There is a limited solubility of BN in hydrochloric acid (HCl), hydrofluoric acid (HF), sulfuric acid (H$_2$SO$_4$), phosphoric acid (H$_3$PO$_4$) and hydrogen peroxide (H$_2$O$_2$). Therefore, by choosing the combination of nitric acid and phosphoric acid with good wetting solvent like acetic acid, the BN part of BCN can be attacked to yield B$_2$O$_3$. Hence the above said etchant is chosen to perform the etch studies of the BCN films.

\[
\text{BC}_n\text{N}_x + \text{HNO}_3 \Rightarrow \text{B}_2\text{O}_3 + \text{N}_2 + \text{CO}_2 + \text{H}_2\text{O}
\]  

\[
\text{B}_2\text{O}_3 + \text{H}_2\text{O} \Rightarrow 2\text{HBO}_2
\]

Etch rate studies.—Figure 1 shows the BCN etch rate as a function of N$_2$/Ar gas flow ratio at different etching temperatures. It is observed that the etch rate is lower for films deposited at higher temperatures. The X-ray diffraction (XRD) studies performed on the films indicated that, the films deposited at higher temperatures improved their grain sizes. Several literatures also report the increase in grain size with the increase in deposition temperature. Mannan et al. reports significant increase in grain size for RF sputtered BCN films with increase in deposition temperatures.29 Similar work by the same group on microwave plasma CVD deposition of BCN has deduced that, the BCN films can be found in crystalline forms of b-BCN in short-range order when deposited at 300 W and 400 W process power.30 In this study, the BCN was deposited at 250 W BN target power and 20 W B$_4$C target power. This higher target power and increase in deposition temperatures might have assisted increase in grain size. Hence, lower temperature deposited films etched faster thereby having the highest etch rate.

| N$_2$/Ar | Room Temperature | 200 °C | 300 °C |
|---------|------------------|-------|-------|
|         | B C N O          | B C N O | B C N O |
| 0.25    | 3.1 74.2 19.3 3.4 | 63.2 27.2 5.1 4.5 | 12.2 43.2 40.0 4.6 |
| 0.5     | 13.1 48.1 31.2 7.6 | 59.6 34.3 2.5 3.7 | 12.0 42.5 40.1 5.4 |
| 1       | 8.8 53.7 27.2 10.3 | 74.2 10.9 4.6 7.3 | 19.6 25.3 50.3 4.8 |
Figure 1. Etch rate as a function of N2/Ar gas flow ratio at various etching temperatures for films deposited at a.) Room temperature, b.) 200° C, c.) 300° C.

For the room temperature deposited films, it shows an overall decreasing trend with respect to increase in N2/Ar gas flow ratio. The films etched at 70° C show the highest overall etch rate. It is interesting to note that, the etch rate is lowest and similar for all the films deposited at N2/Ar = 1. A similar trend can be found in the films deposited at 200° C. The etch rate remains almost same for films deposited at N2/Ar = 0.25 and 0.5 but decreases by a great factor at N2/Ar = 1. A carbon rich film is deposited at N2/Ar = 0.25, with a low amount of boron. Hence, most of film might be comprised of elemental carbon with traces of BC3 and CN phases. HNO3 is known to dissolve carbon to form carbon dioxide (CO2) and nitrous oxide (N2O).28 Therefore, initially the etch rate is high. At N2/Ar = 0.5, there is a decrease in carbon concentration and increase in both boron and nitrogen concentration, which gives rise to more BC3 and C-N phases, thereby decreasing the etching rate by a small factor. The etch rate slightly decreases further at N2/Ar = 1. This is due to further increase in BC3 and C-N phases as a result of small increase in the carbon composition. Hence, a marginal decrease in the etch rate is observed for films deposited at N2/Ar = 1. The etch rates for films etched at 60° C and 70° C, have similar trends with respect to increase in gas flow ratios but they have higher etch rates than films etched at 50° C. Increase in the reaction temperature, increases the frequency of collision of particles, thereby providing the necessary activation energy for successful collisions. This increases the rate of the reaction and such reaction follows the Arrhenius equation.31 Further work on Arrhenius plots are discussed in the next subsection.

From Table I, it can be observed that for films deposited at 200° C, the carbon composition is highest at N2/Ar = 0.5 whereas the etch rate at N2/Ar = 1 is the lowest. The overall nitrogen composition is low in films deposited at 200° C. From this it is assumed that the films deposited at N2/Ar = 1 has more B-C phase in the films with B4C being the dominant bonding characteristic. Literatures have reported that carbon forms stronger networks when singly bonded with elements compared to doubly or triply bonded cases.32,33 Hence the films in which carbon is found in B4C forms are difficult to etch. B4C is fairly insoluble in acids28 and therefore the etch rate further decreases for films deposited at 200° C when compared to room temperature deposited film. As the film deposited at N2/Ar = 0.25 has considerable amount of carbon and boron, it forms more B4C phases. Hence, the etch rate is high. For films deposited at N2/Ar = 0.5, the boron and carbon compositions do not vary much when compared to N2/Ar = 0.25 deposited film, hence the etch rate does not seem to differ by a great amount. The N2/Ar = 1 deposited film consists of significant amount of boron with considerable amount of carbon and traces of nitrogen. Hence, this film is assumed to consist of phases of B4C and traces of elemental boron. Boron can be etched effectively by concentrated HNO3.28 As the aluminum etchant consists of dilute HNO3 in lower quantity, the overall etch rate of the film decreases.
Hence the films deposited at N$_2$/Ar = 1 has the overall lowest etch rate.

The films deposited at 300 °C were observed to have constant etch rate from N$_2$/Ar = 0.25 to 0.5 but show an increase in etch rate at N$_2$/Ar = 1. From Table I, the percentage compositions of B, C and N were similar for films deposited at N$_2$/Ar = 0.25 and 0.5. Hence their etch rates showed similar behavior. For films deposited at N$_2$/Ar = 1, the compositions of both boron and nitrogen increased significantly while the carbon composition decreased. As the boron bonds more preferentially with nitrogen than carbon, it was easier to etch away films with higher h-BN characteristics than B$_4$C or BC$_3$. Hence the overall etch rate becomes high for films deposited at N$_2$/Ar = 1.

**Activation energy.**—The activation energy (E$_a$) and high temperature limit of etch rate or pre-exponential factor (R$_0$) can be calculated according to the Arrhenius law: 34

$$R = R_0 \exp \left(-\frac{E_a}{kT}\right)$$

Wherein, R = etch rate, $k$ = Boltzmann’s constant and T is temperature in Kelvin. Figure 2 shows the Arrhenius plot of BCN films deposited at different N$_2$/Ar gas flow ratios and substrate deposition temperatures.

In Figure 2, the etch rates follow a linear Arrhenius plot as shown. Arrhenius plots can be used to find the activation energies (E$_a$) and their high temperature etch rate limit factor (R$_0$). Table II lists all the values of E$_a$ and R$_0$. In case of room temperature deposited films, the etch rate is highest for films deposited at N$_2$/Ar = 0.25 and 0.5. The lesser the etch rate for a reaction, the higher will be the activation energy needed to complete the etching.

The E$_a$ and R$_0$ are dependent on several reaction parameters like concentration of the solution, nature of etchant and substrate. 35 Activation energy of a reaction is a direct indicator to its high temperature etch limits. Hence, both these terms can be used interchangeably to explain the reaction kinetics. In this study, the activation energies obtained for BCN etching using aluminum etchant ranges from as low as 0.375 eV to 0.939 eV and their corresponding R$_0$ values range from 1.24 × 10$^8$ Å/min to 1.94 × 10$^{16}$ Å/min, respectively. Some literatures report on the activation energies of closely related high temperature ceramic materials such as silicon carbide (SiC) and silicon nitride (SiN). Gelder et al. reports the activation energy of 0.99 eV for silicon nitride, using phosphoric acid (94.5%) etchant. 36 D H van Dorp et al. conducted the anodic wet etching of SiC using 0.1 M KOH solution and were successful in achieving activation energy as low as 45.8 kJ mol$^{-1}$ (0.47 eV). 36 In this present work, it was possible to achieve lower activation energies in the range of 0.38 eV–0.506 eV for room temperature deposited BCN films.

Films deposited at 200 °C, yielded the highest activation energy range despite the fact that films deposited at 300 °C had the slowest etching rates. Hence, the films deposited at 200 °C were more temperature dependent than other films. The pre-exponential factors of as-deposited films and films deposited at 300 °C are in the orders of 10$^0$ to 10$^{12}$, whereas the films deposited at 200 °C have pre-exponential factor in the range of 10$^{16}$. From Table I, it is noted that, the percentage compositions of boron and carbon are very high for films deposited at 0.25 and 0.5 N$_2$/Ar ratio and 200 °C deposition temperature. The ratio of boron to carbon composition is around 2:1. Hence, we can deduce that the films rich in boron and carbon compositions in particular,

![Arrenius plot of films deposited at various gas flow ratios and substrate deposition temperatures](image)

**Table II. Activation energy and pre-exponential factor of BCN thin films deposited at various gas flow ratios and substrate deposition temperatures.**

| N$_2$/Ar | Room Temperature | 200°C | 300°C |
|---------|-----------------|-------|-------|
|         | E$_a$ (eV) | R$_0$ (Å/min) | E$_a$ (eV) | R$_0$ (Å/min) | E$_a$ (eV) | R$_0$ (Å/min) |
| 0.25    | 0.38   | 1.97e08    | 0.939   | 1.94e16   | 0.379   | 1.02e07    |
| 0.5     | 0.375  | 1.24e08    | 0.932   | 1.71e16   | 0.482   | 3.95e08    |
| 1       | 0.506  | 2.64e09    | 0.683   | 4.16e11   | 0.753   | 7.44e12    |

Figure 2. Arrhenius plot of films deposited at a.) Room temperature, b.) 200 °C, c.) 300 °C.
yield \( B_2 C \) rich films. As \( B_2 C \) has a very limited solubility in acids, it solely depends on the high temperatures and takes longer to break its bonds. This is the reason why the pre-exponential factor and its subsequent reaction activation energy is very high for the films deposited at 200 °C. Figure 3 shows the activation energy of BCN films for various \( N_2/Ar \) gas flow ratios and substrate deposition temperatures. The films deposited at room temperature and 300 °C show an increase in trend whereas the films deposited at 200 °C show a decreasing trend. From Table I, it can be observed that the percentage compositions of nitrogen in the as-deposited and 300 °C deposited films, is considerably high when compared to films deposited at 200 °C.

The activation energies of BCN films deposited at room temperature and at 300 °C show an increase in trend with increase in gas flow ratio. Their overall activation energy is lower than the 200 °C deposited films. It is probably because of higher nitrogen and lower boron concentration in these films with significant carbon concentrations. According to the chemical properties of B, C and N during the deposition process, B and C can exist both as elements and as compounds reacting with N or each other, while N could exist in the films only as compound by reacting with either B or C. Therefore, the process of nitrogen integration and the reciprocity among B, C and N during the deposition become main factors, which affect the composition and properties of BCN films. These films can be considered as carbon rich films with elemental carbon and traces of C-N, and B-N bonds. Hence, it is easy to dissolve these carbon rich films and therefore their subsequent activation energy for the reaction is less.

Conclusions

BCN films were deposited by dual target sputtering of \( B_2 C \) and BN targets at various substrate deposition temperatures and \( N_2/Ar \) gas flow ratios. A common aluminum etchant was used to etch these samples at different temperature conditions and their subsequent activation energies (\( E_a \)) and pre-exponential factors (\( R_o \)) were calculated using Arrhenius plots. Room temperature deposited BCN film at \( N_2/Ar = 0.25 \) was found to have the highest etch rate of 231.6 Å/min. Boron rich films with significant amount of carbon showed \( B_2 C \) like characteristics, hence the overall etch rate decreased drastically and they became more temperature dependent reactions yielding very high values of \( R_o \) and \( E_a \). BCN films deposited at 200 °C showed the highest temperature dependent reaction among others, and they have a high \( E_a \) (~0.9 eV) and \( R_o \) (10^16 Å/min). Films deposited at 300 °C showed the lowest etch rate in the range of (~11–13 Å/min). BCN has a wide range of reaction kinetics with respect to different elemental compositions due to the various deposition parameters involved. This work accounts for workability of common aluminum etchant as an economic, effective reagent for BCN etching and cleaning in a fabrication process.

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