

A Brief Review of Plasma Enhanced Atomic Layer Deposition of Si₃ N₄

저자 (Authors)	You Jin Ji, Ki Seok Kim, Ki Hyun Kim, Ji Young Byun, Geun Young Yeom
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Review Paper

A Brief Review of Plasma Enhanced Atomic Layer Deposition of Si₃N₄

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You Jin Ji^a, Ki Seok Kim^a, Ki Hyun Kim^a, Ji Young Byun^a, and Geun Young Yeom^{a,b,*}

^aSchool of Advanced Materials Science and Engineering, Sungkyunkwan University, Suwon 16419, Republic of Korea

^bSKKU Advanced Institute of Nano Technology (SAINT), Sungkyunkwan University, Suwon 16419, Republic of Korea

*Corresponding author E-mail: gyyeom@skku.edu

ABSTRACT

Silicon nitride (SiN_x) thin films have attracted interest as an important material for use in next-generation devices such as a gate spacer in 3D fin field-effect transistors (finFETs), charge trap layers, etc. Many studies using the SiN_x plasma enhanced atomic layer deposition (PEALD) method have been conducted, owing to its advantages over other SiN_x deposition methods. In this review, the recent studies on PEALD of SiN_x thin films are summarized, and the effects of some process parameters including plasma power, frequency, and process temperature on the material properties of SiN_x are discussed. In addition, some properties of SiN_x thin films such as conformality, wet etch rate, and others are reviewed.

Keywords: Silicon nitride (Si₃N₄), Plasma enhanced atomic layer deposition (PEALD), Process temperature, Step coverage, Wet etch rate

I. Introduction

Recently, silicon nitride (SiN_x) has attracted considerable interest owing to its diverse range of applications [1-10]. For instance, SiN_x is used as a permeation barrier for flexible organic light emitting devices [7-10] or as a charge trap layer for logic and memory devices [4]; SiN_x gate spacers have also been studied extensively [1-3,6]. High quality and excellent conformality are critical requirements for various applications of SiN_x thin films. In addition, lowering the deposition temperature is an important factor for devices employing low temperature substrates such as polymer substrates. To satisfy such a requirement for employing SiN_x thin films, many studies have used various deposition techniques such as chemical vapor deposition (CVD), low pressure chemical vapor deposition (LPCVD), plasma enhanced chemical vapor deposition (PECVD), atomic layer deposition (ALD), plasma enhanced atomic layer deposition (PEALD), and so on [11-43].

Low pressure chemical vapor deposition is the most common technique used to fabricate SiN_x thin films because of its simple method and low cost. However, it is difficult to achieve a conformal layer on a high aspect ratio structure. In addition, the deposition needs to be conducted at high temperature (> 700 °C) [17,18]. Plasma enhanced chemical vapor deposition can deposit films at temperatures lower than that by using thermal LPCVD. Unfortunately, this leads to poor step coverage and low film quality [3,15,16]. To address issues related to conformality, the ALD technique has been studied extensively [19-26]; ALD is a cyclic process that offers atomic scale thickness control of the material that is being deposited. In addition, ALD methods can deposit thin films with high quality in terms of low wet etch rate and high conformality at low process temperatures [25,26]. However, ALD methods have several challenges such as a relatively

high thermal budget for actual device application and low throughput (GPC > 2 Å/min) that hinders the industrialization of ALD methods [22,26]. By assisting with a plasma for dissociating reactive gases during ALD with PEALD processes, a thin film with a good step coverage on a high aspect ratio structure can be deposited at low temperatures. A plasma with reactant molecules can be used instead of exposure to reactant molecules only during the reactant exposure step; highly reactive species are formed during the reactant exposure step, which allows the deposition of high quality films with a high growth rate while lowering deposition temperatures [15,27-42].

In this paper, we briefly reviewed the recent work of PEALD SiN_x and related process parameters that could determine film characteristics. Furthermore, this review will discuss properties of the film that are dependent on deposition conditions. A schematic of a PEALD cycle is shown in Fig. 1. Each SiN_x PEALD cycle can be divided into four steps. In the first precursor adsorption step, similar to ALD, a Si precursor is introduced into the deposition chamber. Si precursors are chemisorbed on the surface through self-limiting reactions followed by a purge step. In the following plasma exposure step (for ALD, reactant exposure step), plasma-generated reactive species react with the adsorbed precursor on the surface. As a plasma source, capacitively coupled plasma (CCP) or inductively coupled plasma (ICP) sources are commonly used along with N₂, NH₃, or N₂/H₂ to generate reactive plasmas [5,15,27-40]. To optimize PEALD processes, various parameters should be adjusted to meet the material properties of SiN_x required for the application.

II. Process parameters

As mentioned above, in PEALD, there are various process parameters including reactant gas, plasma source, precursor, precursor



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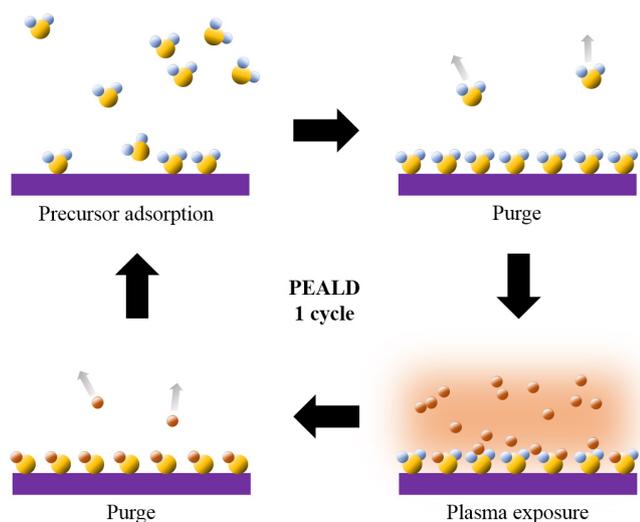


Figure 1. (Color online) Schematic of a PEALD cycle.

Table I. Summary of recent studies on PEALD SiN_x.

Si precursor	Reactant	Plasma source / Frequency	Process temperature	GPC (Å/cycle)	Ref.
Silane (SiH ₄)	N ₂	CCP 200–400 kHz	250–400 °C	0.25–2	15
Trisilylamine (TSA, Si ₃ H ₉ N)	NH ₃	ICP 13.56 MHz	250–350 °C	0.65	27
Hexachlorodisilane (HCDS, Si ₂ Cl ₆)	NH ₃	CCP 13.56 MHz	350–450 °C	1.2 (400 °C)	28
Bis(<i>tertiary</i> -butylamino)silane (BTBAS, SiH ₂ (NH ^t Bu) ₂)	N ₂	ICP 13.56 MHz	400–500 °C	0.15 (500 °C)	29
Trisdimethylaminosilane (3DMAS, C ₆ H ₁₉ N ₃ Si)	N ₂	ICP		0.12	
Dichlorosilane (DCS, SiH ₂ Cl ₂)	NH ₃	13.56 MHz	350 °C	0.24	30
Trisilylamine (TSA, Si ₃ H ₉ N)	N ₂			-	
Bis(dimethylaminomethylsilyl)trimethylsilyl amine (DTDN2-H2, C ₉ H ₂₉ N ₃ Si ₃)	N ₂	27.12 MHz	250–400 °C	0.36	31
Neopentasilane (NPS, (SiH ₃) ₄ Si)	N ₂	CCP 13.56 MHz	250–300 °C	1.4	32
Trisilylamine (TSA, (SiH ₃) ₃ N)	N ₂			1.2	
Di(<i>sec</i> -butylamino)silane (DSBAS, SiH ₃ N(^s Bu) ₂)	N ₂	ICP 13.56 MHz	100–500 °C	~0.19 (100 °C)	33
Diisopropylaminosilane (DIPAS, C ₆ H ₁₇ NSi)	N ₂	CCP 13.56 MHz	150–250 °C	0.3 (100 °C)	34
Pentachlorodisilane (PCDS, HSi ₂ Cl ₅)	N ₂	Hollow cathode 13.56 MHz	270–360 °C	0.2	35
	N ₂ / NH ₃			~1 (270 °C)	
Hexachlorodisilane (HCDS, Si ₂ Cl ₆)	N ₂ / NH ₃	Hollow cathode	270–360 °C	~0.68	5
	Ar / NH ₃			~0.57	
Two step Hexachlorodisilane (HCDS, Si ₂ Cl ₆) + Methylamine (CH ₃ NH ₂)	N ₂	CCP 13.56 MHz	400 °C	~0.9	36
1,3-di-isopropylamino-2,4-dimethylcyclosilazane (CSN-2, C ₈ H ₂₂ N ₂ Si ₂)	N ₂	27.12 MHz	200–500 °C	0.43	37
	NH ₃ / N ₂ + N ₂			~0.35	
Bis(<i>tertiary</i> -butyl-amino)silane (BTBAS, SiH ₂ (NH ^t Bu) ₂)	N ₂	ICP 13.56 MHz	85 °C 155 °C 275 °C	0.8 0.3 0.2	38
Bis(<i>tertiary</i> -butyl-amino)silane (BTBAS, SiH ₂ (NH ^t Bu) ₂)	N ₂	ICP 13.56 MHz	80 °C 120 °C 160 °C 200 °C	0.44 0.33 0.26 0.24	39
Dichlorosilane (DCS, SiH ₂ Cl ₂)	NH ₃	13.56 MHz	400–630 °C	1.39 (550 °C)	40

dose time, purge time, process temperature, and others. In this section, the effects of some process parameters influencing SiN_x deposition are briefly discussed.

1) Precursors

Many kinds of precursors such as trisilylamine (TSA), diisopropylaminosilane (DIPAS), bis(*tertiary*-butyl-amino)silane (BTBAS), trisdimethylaminosilane (3DMAS), di(*sec*-butylamino)silane (DSBAS), hexachlorodisilane (HCDS), pentachlorodisilane (PCDS), dichlorosilane (DCS), and tetramethylsilane (TMS) have been reported as Si sources of SiN_x PEALD [5,27–30,32–36,38–40]. Table I summarizes studies on SiN_x PEALD in recent decades; it also provides details on Si precursors, reactant gas, plasma source, deposition temperature, and growth rate.

2) Plasma conditions

Plasma characteristics such as density of radicals, energy, and density of electrons and ions have a considerable influence on SiN_x PEALD processes. Therefore, controlling plasma conditions such as rf

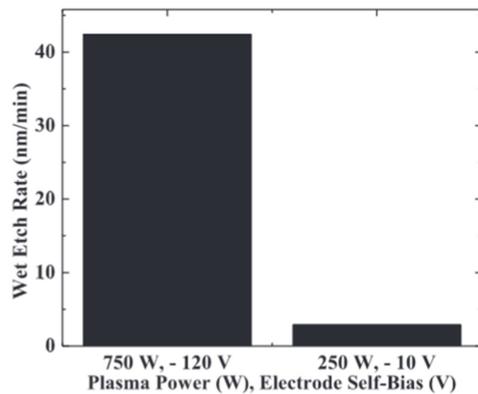


Figure 2. Wet etch rate of SiN_x deposited using NPS and N₂ plasma with different plasma conditions. Reproduced with permission from [32], Copyright, American Institute of Physics.

Table II. Hydrogen concentration and film density of SiN_x for different plasma power. Reproduced with permission from [32], Copyright, American Institute of Physics.

Power (W)	H (at. %)	Density (g/cc)
750	23.5	1.86
250	11	2.21

power and frequency is important.

Weeks *et al.* [32] reported PEALD SiN_x using a precursor of neopentasilane (NPS) and N₂ plasma by using a CCP source at the temperature of 275 °C. They determined the effects of plasma power on the wet etch rate, hydrogen concentration, and film density. A high plasma power resulted in more energetic ions that could damage the depositing film. Therefore, as shown in Fig. 2, the wet etch rate of the SiN_x films in a 100:1 HF solution (HF: deionized water = 1:100) decreased when the plasma power decreased from 750 to 250 W, which was consistent with the results of low hydrogen contents and high film density, as summarized in Table II. With a decrease in the power from 750 to 250 W, the H concentration decreased while the film density increased. The wet etch rate is closely related to the H concentration in the film and film density [30].

Park *et al.* [31] also investigated the effect of RF power on film property. Charge trap density, wet etch rate, N/Si ratio, carbon concentration, and oxygen concentration were measured for SiN_x film deposited by PEALD using DTDN2-H2 and N₂ plasma with various plasma powers ranging from 75 to 400 W. As shown in Fig. 3, the carbon and oxygen concentrations of the film increased with increasing RF power; however, the N/Si ratio decreased. This can be attributed to the dissociation of precursor ligands that desorbed from the surface because of the high RF power of the plasma. Owing to the deposition of the carbon impurity, the wet etch rate of the film was also increased with increasing RF power.

Another important parameter influencing plasma properties such as electron density and electron temperature is the frequency of plasma generation. Thus, the plasma generation frequency also needs to be controlled to improve SiN_x thin film quality. King *et al.* compared the Fourier-transform infrared spectroscopy (FTIR) results for SiN_x deposited using SiN₄ and N₂ plasma with different source frequencies [15]. As shown in Fig. 4, PEALD SiN_x film using a frequency of 13.56 MHz showed a high intensity of the Si-N stretching mode. In

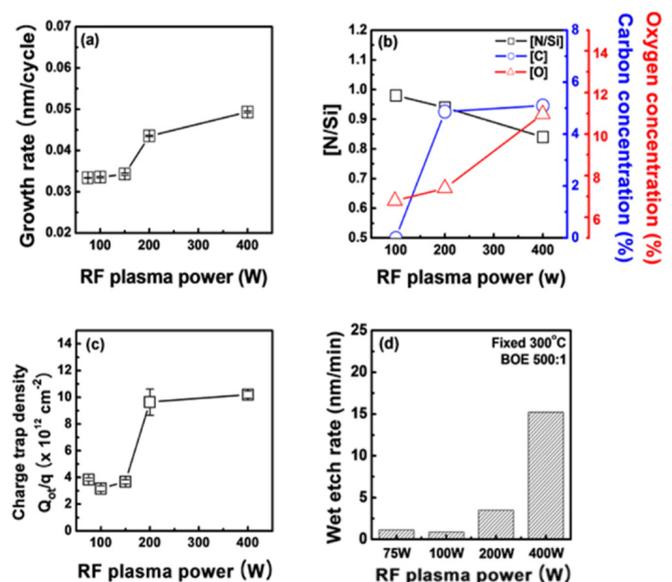


Figure 3. (Color online) (a) Growth rate per cycle (GPC), (b) carbon and oxygen concentrations and N/Si ratio, (c) charge trap density, and (d) wet etch rate with increasing RF power in the PEALD process. PEALD SiN_x films were deposited with different rf plasma power. Reproduced with permission from [31], Copyright 26, American Chemical Society.

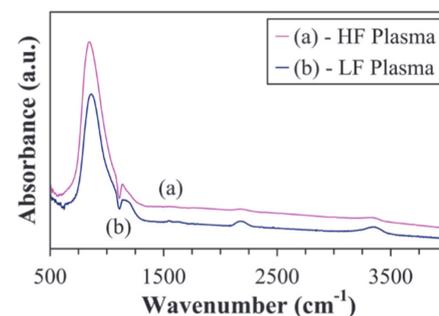


Figure 4. (Color online) FTIR spectra for PEALD SiN_x films deposited using (a) HF (13.56 MHz) N₂ plasmas and (b) LF (200–400 kHz) N₂ plasmas. Reproduced with permission from [15], Copyright, American Institute of Physics.

contrast, the peak intensities of the Si-H and N-H stretching modes were lower than those obtained from films deposited using a frequency of 200–400 kHz. A higher plasma frequency can achieve the effective decomposition of N₂ gas, thus allowing a higher density of N and N⁺ ions. Reactive species such as N and N⁺ ions can react with the Si-H surface bonds and form a bond with Si by replacing the H atom. Therefore, PEALD SiN_x films deposited using higher frequency showed less Si-H and N-H bonding compared to those using lower frequency.

3) Process temperature

PEALD methods can lower the deposition temperature compared to other deposition techniques. However, a considerably lower process temperature is still required for SiN_x PEALD for various applications of SiN_x films. For example, in the case of polymer or flexible substrates, very low process temperatures are required. The effect of the substrate temperature on the surface reaction mechanism

in SiN_x PEALD systems has been studied. Results showed that the PEALD process could lower the process temperature compared to other deposition techniques (LPCVD and ALD, etc.). However, the quality of SiN_x thin films is still temperature dependent.

Park *et al.* investigated the effect of process temperature on step coverage and wet etch rate [37]. They deposited SiN_x films on trench patterned wafers (aspect ratio (AR) of 5.5) using 1,3-di-isopropylamino-2,4-dimethylcyclosilazane (CSN-2) precursor and N₂ plasma (27.12 MHz) at temperatures between 250 and 500 °C. As shown in Fig. 5, SiN_x thin films grown at 500 °C showed a high step coverage of 98 % at the center of the trench. With increasing temperature, the step coverage of the middle and bottom side walls showed substantial enhancement in conformality. In addition, the wet etch rate of a silicon nitride film in a diluted HF solution (300:1 diluted HF solution) decreased with an increasing process temperature as shown in Fig. 6. Differences in the wet etch rates of the bottom and lower sidewalls also decreased for films deposited at 500 °C. These results indicate that a higher process temperature offers more reactions between N radicals of the plasma and the precursor ligands on the surface. At a high process temperature, N radicals can reach the lower sidewall of the trench and effectively remove ligands from the surface, which results in excellent step coverage and wet etch rate.

Similarly, Jang *et al.* studied the temperature dependency of SiN_x thin film properties such as surface roughness and refractive index [27]. Their results revealed that SiN_x thin films deposited at lower temperatures showed lower defect density caused by high hydrogen content.

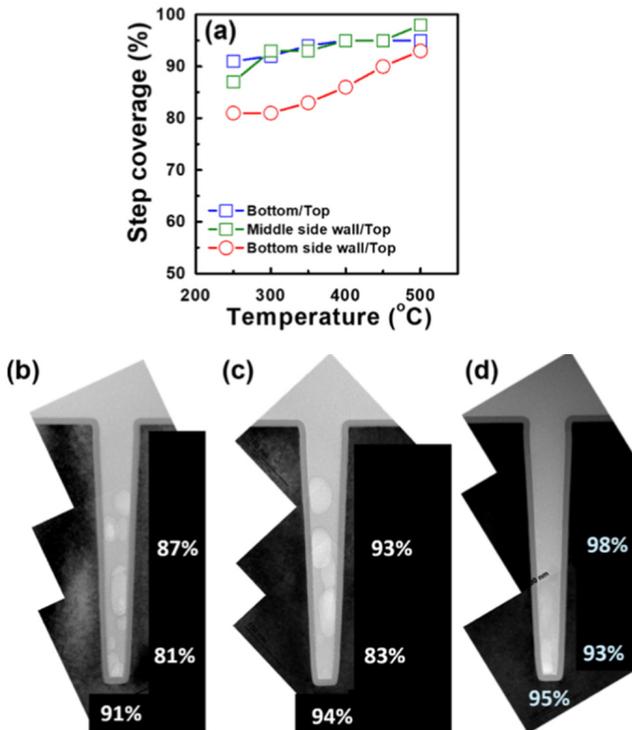


Figure 5. (Color online) (a) Step coverage of PEALD SiN_x thin film deposited by PEALD at various process temperatures. Cross-sectional TEM images of SiN_x thin films deposited by PEALD consisting of CSN-2 exposure and N₂ plasma at (b) 250, (c) 350, and (d) 500 °C. Reproduced with permission from [37], Copyright 2018, American Chemical Society.

High defect density is the common reason for hysteresis in a capacitance-voltage (C-V) curve. Therefore, as shown in Fig. 7, the SiN_x thin film deposited at 250 °C showed a considerably lower hysteresis curve. Although lower hysteresis was observed at lower PEALD temperature because of the high hydrogen content, crystallographic defects were found to be higher at lower deposition temperatures. In addition, hydrogen could be removed during processes such as annealing. Therefore, the deposition of SiN_x at the lower PEALD temperature tends to cause more defect formation in the film.

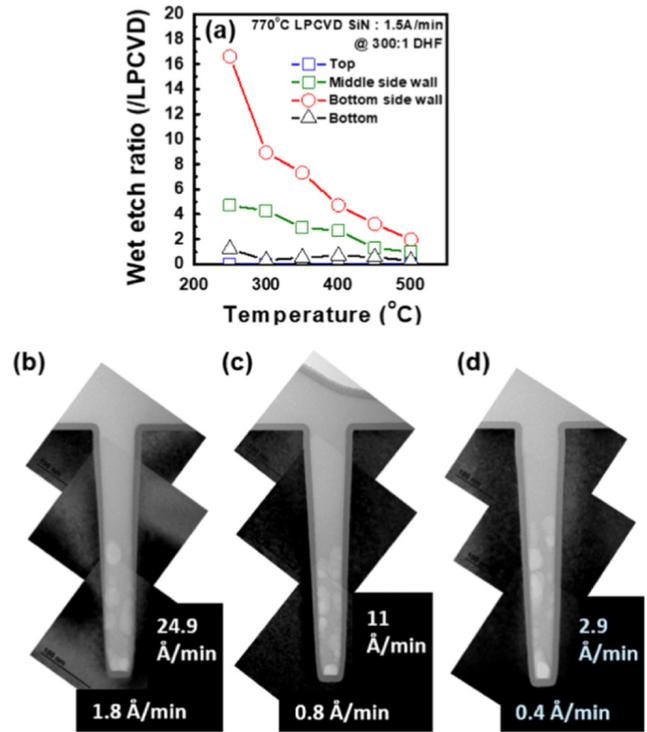


Figure 6. (Color online) (a) Wet etch rate of PEALD SiN_x depending on process temperature. Cross-sectional TEM images of SiN_x films deposited by PEALD consisting of CSN-2 exposure and N₂ plasma at (b) 250, (c) 350, and (d) 500 °C after wet etch in a 300:1 diluted HF solution. Reproduced with permission from [37], Copyright 2018, American Chemical Society.

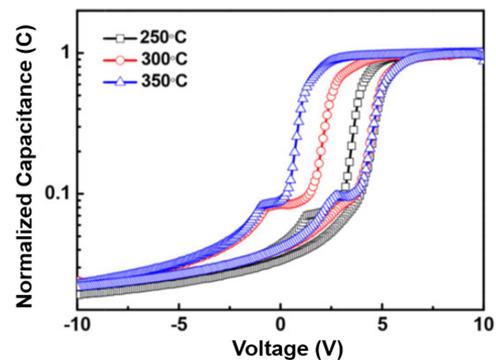


Figure 7. (Color online) C-V curves of SiN_x films deposited by PEALD as a function of process temperature. Reproduced with permission from [27], Copyright 2014, John Wiley and Sons.

III. Properties of SiN_x

In this section, we discuss common properties of SiN_x thin films. These properties are important for applications of the film.

1) Step coverage

When the feature size of a semiconductor device decreases, high conformality on high aspect ratio structures is a critical requirement. Studies on SiN_x PEALD focusing on the conformality of the film have been reported. For example, Faraz *et al.* deposited a SiN_x layer on trench-patterned wafers with an aspect ratio of 4.5 using a PEALD method [33]. Films were deposited by alternating the exposures of di(*sec*-butylamino)silane (DSBAS) precursor and N₂ plasma (13.56 MHz) at 500 °C. The step coverage of SiN_x film was examined. As shown in Fig. 8, a bottom coverage of 69 % and sidewall coverage of 50 % were observed using PEALD. The step coverage was not as good as that in ALD processes. However, several reports on SiN_x thin films deposited by the PEALD method showed a good step coverage of above 90 %.

Ovanesyan *et al.* [36] reported SiN_x thin films with ~95 % conformality on patterned structures (aspect ratio of ~5). Figure 9 is a TEM image for a ~25-nm-thick SiN_x thin film deposited from Si₂Cl₆,

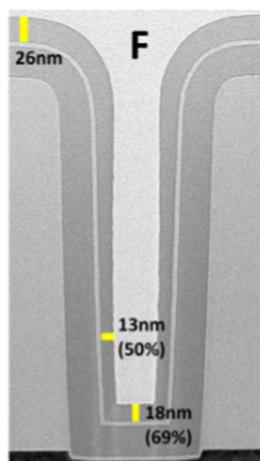


Figure 8. (Color online) Cross-sectional TEM image of SiN_x thin film deposited on a high aspect ratio structure (AR = 4.5:1) by PEALD using DSBAS and N₂ plasma. Reproduced with permission from [33], Copyright 2017, American Chemical Society.

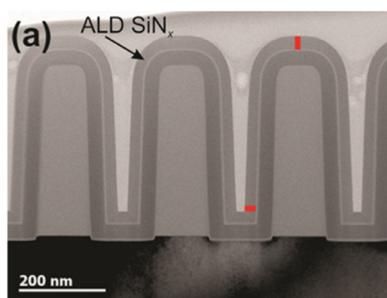


Figure 9. (Color online) Cross-sectional TEM image of post wet-etch SiN_x deposited by a three-step PEALD process using Si₂Cl₆, CH₃NH₂, and N₂ plasma at 400 °C. Reproduced with permission from [36], Copyright 2018, American Chemical Society.

CH₃NH₂, and N₂ plasma at 400 °C. In this case, Ovanesyan *et al.* [36] suggested a novel three-step PEALD process. By adding a CH₃NH₂ exposure step in the PEALD cycle, conformality and growth rate per cycle (GPC) were increased compared to those with the PEALD method using aminosilanes and N₂ plasma. Park *et al.* [37] also reported that SiN_x thin films deposited by PEALD at 500 °C showed a high conformality of ~95 % on patterned wafers of AR 5.5. Thus, PEALD methods can be used to deposit SiN_x thin films with high conformality on trench structures. However, to achieve higher conformality on a high aspect ratio structure (AR > 6) with low process temperature, various attempts such as using a novel Si precursor or additional steps in PEALD process are required.

2) Wet etch rate

The wet etch rate is highly correlated with the integrity and density of the film. Kim *et al.* [5] demonstrated the relationship between film density and wet etch rate and suggested a SiN_x etching mechanism. They investigated the effects of plasma gas composition and process temperature on wet etch rates of SiN_x deposited by PEALD. The SiN_x thin films deposited using hexachlorodisilane (HCDS) precursor and Ar/NH₃ plasma at 300 °C showed a wet etch rate of 1.2 nm/min in a 500 : 1 HF solution. By comparing the wet etch rates of SiN_x deposited at various conditions, Kim *et al.* [5] found that the densification of the SiN_x thin film is caused by increased Si-N bonds, which eliminate hydrogen and result in low wet etch rates.

In another study, Knoops *et al.* [29] deposited SiN_x thin films on planar substrates using bis(tertiary-butyl-amino)silane (BTBAS) and N₂ plasma at 400 °C, and they investigated the wet etch rate of the films before and after a dip in a 7:1 HF solution (H₂O : HF = 7:1). A low wet etch rate (~1 nm/min) was obtained because of the low hydrogen concentration in the films. This result is comparable to the wet etch rate of the SiN_x thin film deposited by chemical vapor deposition (CVD) at high temperature.

Besides studies on the wet etch rate of SiN_x thin films on planar surfaces, the wet etch rate has also been studied for high aspect ratio structures. However, most studies showed different wet etch rates for the sidewall, bottom, and top surface of 3D trench patterns [36]. The poor wet etch rate at the bottom sidewall is problematic in silicon nitride PEALD processes.

IV. Concluding remarks

This brief review examined not only the deposition of SiN_x thin films using the PEALD process, but also summarized the characteristics of SiN_x thin films. Compared to other deposition methods, the PEALD process provides atomic scale thickness control and excellent conformality with a lower process temperature. Various SiN_x PEALD processes have been studied, and some results have shown excellent film properties such as good step coverage and low wet etch rate. However, for device applications, several issues need to be solved. To address these issues, the effect of each PEALD process parameter (temperature, plasma power, exposure time, and gas composition) on the film growth mechanism needs to be understood in detail. Further, new plasma sources and precursors that can deposit highly conformal and dense thin films at low process temperatures for a variety of state-of-the-art devices, which require SiN_x thin films, must be developed. Further studies on the deposition mechanism are required to enhance the quality of the deposited SiN_x thin films.

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