Critical dimension improvement of plasma enhanced chemical vapor deposition silicon nitride thin films in GaAs devices

I. Hallakoun a,b,*, I. Toledo a, J. Kaplun a, G. Bunin a, M. Leibovitch a, Yoram Shapira b

a Gal-El (MMIC), P.O. Box 330, Ashdod 77102, Israel
b Department of Physical Electronics, Faculty of Engineering, Tel-Aviv University, Ramat-Aviv 69978, Israel

Abstract

Silicon nitride thin films are widely used in GaAs device fabrication. Plasma enhanced chemical vapor deposition is the most commonly used technique by which silicon nitride is deposited. Changing the process conditions may significantly change the layer physical properties and chemical composition. The device definition includes both deposition of the dielectric layer and its patterning, by etching the exposed SiN via a photoresist mask with sub-microns resolution. The current study examines the influence of the deposition and etching process conditions on the layer characteristics and the devices critical dimension (CD) control. Empirical formulae that correlate process parameters (temperature, gas flow ratio, pressure and RF power) with layer properties have been found and with very high precision yield the deposition rate, wet and dry etching rates and refractive index of the layer. Better understanding of the deposition and etching processes led to significant improvement in controlling the device CD.

© 2003 Elsevier B.V. All rights reserved.

Keywords: Deposition; Pressure; Resolution

1. Introduction

Silicon nitride thin films (SiN x ) are widely used in GaAs monolithic microwave integrated circuit (MMIC) fabrication. The layers serve as dielectric films in MIM (metal–insulator–metal) capacitors [1], semiconductor surface passivation [2], humidity and metal-ion diffusion barrier [3] and mechanical protection. The most commonly used technique, by which silicon nitride is deposited, is plasma enhanced chemical vapor deposition (PECVD) [4]. It uses RF plasma, instead of pyrolysis, to break reactant molecular bonds. This technique is preferred over conventional CVD because it allows the deposition process to take place at relatively low temperatures, below 300 °C. This is important since GaAs processing does not tolerate higher temperatures. However, using hydride source gases may result in hydrogen incorporation into the layer, which is then designated by SiN x :H. Therefore, all reaction byproducts must be in the gaseous state to be evacuated from the chamber. The PECVD process parameters, such as substrate temperature, reactant flow, total chamber pressure and RF generator power may vary, significantly affecting the resulting SiN x layer physical properties and chemical composition.

The device definition includes both deposition and patterning of the dielectric layer. The latter is carried out by etching the exposed SiN x via a photoresist mask. The etching process has to be very accurate to define lines and intervals with sub-micron resolution. The smaller the devices get, the finer is the critical dimension (CD) of the circuit. Wet etching of the SiN x layers is usually performed in a hydrofluoric acid (HF) [5] or phosphoric acid (H3PO4) solutions [6]. The wet etching process is isotropic in nature, i.e., the lateral etching rate is of significance and cannot be ignored. The etching solution penetrates underneath the photoresist mask, causing widening of the etched structure and poor pattern definition. A dry etching process in a reactive ion
etching (RIE) system is usually a better choice for pattern definition with sub-micron resolution [7]. However, this process is composed of both chemical reaction and physical bombardment of the SiN$_x$ layer. Thus, it may result in different pattern definitions on different types of SiN$_x$ layers.

Several researchers have investigated the relations between PECVD process conditions and some of the layer physical properties, such as deposition rate, wet etching rate, refractive index, mechanical stress, and moisture penetration resistance [8–10]. It has been suggested that a higher refractive index (above 1.95) and a slower wet etching rate can be associated with a denser film and less hydrogen content in the layer, hence superior electronic quality [11,12]. Other publications have compared different etching chemistries of identical SiN$_x$ films [13]. The fluorinated gas chemistries were preferred over chlorine- or bromine-containing gases, due to the high selectivity of the former to SiN$_x$ without damaging the GaAs substrate. However, none of the studies has examined the etching characteristics, especially CD control and line definition, as a function of the deposition conditions.

The current study examines the influence of the combination of SiN$_x$ deposition and etching conditions on the layer characteristics. Capacitors with various types of SiN$_x$ were fabricated and measured to determine the most suitable insulating layer. Final layer parameters: capacitance, Si/N ratio, and refractive index, as well as deposition rate, wet etching rate (in buffered HF solution), and dry etching rate (in CF$_4$, RIE system) were measured as a function of deposition parameters, e.g., substrate temperature, reactant (silane/ammonia) ratio, total chamber pressure and RF generator power. Pattern definition parameters, such as line width, CD and definition were measured as a function of both deposition and etching parameters. Our goal has been to improve the CD control of the MMIC device patterns, without damaging the electrical performance, by finding a suitable combination of layer deposition and etching conditions.

### Experimental procedure

All SiN$_x$ films were deposited in a PlasmaLab model 80 PECVD system (Oxford Plasma Technology, UK) using a gaseous mixture of silane (SiH$_4$) and ammonia (NH$_3$) as reactants, both diluted to 5% in nitrogen (N$_2$) for safety. For the dry etching process, a mixture of CF$_4$ with 3.5% O$_2$ in a RIE system (Nexctal model NE110, France) was used. The wet etching was done using a 6:1 buffered HF solution. All substrates were 3” semi-insulating GaAs [1 0 0] wafers or slices.

Several screening experiments of depositing SiN$_x$ layers in different conditions were performed. The quality of the SiN was estimated only by the wet etching rate. The slower the etch rate, the better the layer. The refractive index and deposition rate were also measured. Based on those experiments, a desirable process window was established. The process window was chosen taking into consideration the limitations set by other steps in the device fabrication process (such as not exposing the pre-deposited metal contacts to high temperature).

The Design of Experiments (DOE) was done using a statistical method of Fractional Factorial Design. Nine experimental set-ups were chosen for full runs of capacitor fabrication, out of the 81 possibilities of a 3-level 4-factor design. The process parameters of all 9 experiments are listed in Table 1. Each set-up differs from the other in more than one factor and the results are interpreted with regard to this fact.

Based on the results of this experimental matrix a set of deposition parameters for an improved layer was chosen. It was not similar to any of the 9 experimental set-ups. This set of parameters was composed of the optimal point of each of the parameters separately. The resulting layer was designated as SiN type2 and was deposited in a consecutive experiment. It was compared to a SiN type1, which had been the standard layer used in the device fabrication prior to this study. We measured the capacitor performance, uniformity and repeatability of the thin film deposition process, pattern edge definition and CD control during the dry etching process.

### Table 1

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Temperature (°C)</th>
<th>Pressure (mTorr)</th>
<th>NH$_3$/SiH$_4$ ratio</th>
<th>Power (W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>245</td>
<td>1000</td>
<td>0.54</td>
<td>30</td>
</tr>
<tr>
<td>2</td>
<td>245</td>
<td>1200</td>
<td>0.96</td>
<td>50</td>
</tr>
<tr>
<td>3</td>
<td>245</td>
<td>1400</td>
<td>0.75</td>
<td>40</td>
</tr>
<tr>
<td>4</td>
<td>260</td>
<td>1000</td>
<td>0.96</td>
<td>40</td>
</tr>
<tr>
<td>5</td>
<td>260</td>
<td>1200</td>
<td>0.75</td>
<td>30</td>
</tr>
<tr>
<td>6</td>
<td>260</td>
<td>1400</td>
<td>0.54</td>
<td>50</td>
</tr>
<tr>
<td>7</td>
<td>275</td>
<td>1000</td>
<td>0.75</td>
<td>50</td>
</tr>
<tr>
<td>8</td>
<td>275</td>
<td>1200</td>
<td>0.54</td>
<td>40</td>
</tr>
<tr>
<td>9</td>
<td>275</td>
<td>1400</td>
<td>0.96</td>
<td>30</td>
</tr>
</tbody>
</table>
The comparison of dry etching characteristics was carried out using the same (standard) etching process for both type1 and type2 SiN layers. This process was developed for the type1 SiN. Thus, another step of optimizing the etching process for type2 SiN was performed.

The layer characteristics vs. deposition conditions: deposition rate as a function of: a—Rf generator power, b—chamber pressure, c—silane to ammonia ratio, wet etching rate as a function of: d—silane to ammonia ratio, e—temperature, N-refraction index as a function of: f—silane to ammonia ratio.

Fig. 1. Layer characteristics vs. deposition conditions: deposition rate as a function of: a—Rf generator power, b—chamber pressure, c—silane to ammonia ratio, wet etching rate as a function of: d—silane to ammonia ratio, e—temperature, N-refraction index as a function of: f—silane to ammonia ratio.
3. Results and discussion

3.1. Deposition parameters and layer physical properties

Empirical formula, which correlate process parameters (temperature, gas flow ratio, pressure and RF power) with layer properties have been found and with very high precision yield the deposition rate, wet and dry etching rate and refractive index of the layer. The empirical formulae calculated, based on all data collected from the experiments, in first order approximation:

Deposition rate
\[ = 10.2 \times \text{power} + 360 \times \text{pressure} - 146 \times \text{gas ratio} - 295 \]

Wet etching rate
\[ = 198 + 43 \times \text{pressure} + 80.5 \times \text{gas ratio} - 1.1 \times \text{temp} \]

Dry etching rate
\[ = 3.8 \times \text{temp} - 271 \times \text{gas ratio} - 600 \]

Refractive index
\[ = 2.28 - 0.21 \times \text{gas ratio} \]

The units used in all equations are: power, W; temperature, °C and pressure in Torr.

Indeed, the deposition parameter set-ups that were chosen based upon those results yielded the predicted characteristics with very high accuracy.

Fig. 1a–f shows some of the layer characteristics as a function of the major deposition parameters affecting them. It must be emphasized that all other parameters were not kept constant (as explained in the DOE) and this is the main cause of the result scattering.

Table 2 summarizes the most significant parameters affecting the measured physical properties of the layer,

<table>
<thead>
<tr>
<th>Variable</th>
<th>1st</th>
<th>2nd</th>
<th>3rd</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wet etch rate</td>
<td>Temperature</td>
<td>+NH₃/SiH₄</td>
<td>+Pressure</td>
</tr>
<tr>
<td>Dry etch rate</td>
<td>+Temperature</td>
<td>−NH₃/SiH₄</td>
<td>−</td>
</tr>
<tr>
<td>Ref index</td>
<td>−NH₃/SiH₄ ratio</td>
<td>−</td>
<td>−</td>
</tr>
<tr>
<td>Deposition rate</td>
<td>+Power</td>
<td>+Pressure</td>
<td>−NH₃/SiH₄ ratio</td>
</tr>
</tbody>
</table>
such as wet and dry etching rate, refractive index and deposition rate. A negative sign indicates decrease while a positive sign indicates increase in the measured response with increasing the process parameter. The results are in good accordance with those presented by Chou et al., [8], except for the dry etching rate, which was not reported by them. The dry and the wet etching rates are almost inversely dependent on the deposition conditions, which indicates a different mechanism for both processes. A layer that is highly resistive to chemical attack (which makes it a good protective layer for the device) can be very easily patterned and etched by plasma reactive ions, in a relatively short process, leaving no residues on the surface. This led to a

Fig. 2. Edge definition of 3-μm line SiN etched structure a. SiN type1, b. SiN type2.

Fig. 3. A 3-μm line SiN etched structure width distribution: (a) SiN type1 ‘old’ etching process, (b) SiN type2 ‘old’ etching process, (c) SiN type2 ‘new’ etching process.
correlation between the deposition conditions and the pattern definition characteristics of the silicon nitride layer.

The capacitor measurements from all 9 wafers of the DOE yielded a value of $300 \pm 20\%$ pF, instead of $\pm 10\%$ of the standard process, indicating changes in thickness uniformity and/or dielectric constant of the layer, from one experimental set-up to another.

3.2. Pattern definition and critical dimension control

As a result of the experimental matrix in the previous section, a new set of deposition parameters was established, to manufacture layers with better chemical resistance, higher refractive index and reasonable dry etching rate while maintaining relatively moderate temperature (below 270 °C). This layer, designated as SiN type2 was compared to a SiN type1, which had been the standard layer used in the device fabrication prior to this study. Fig. 2a and b show that the edge definition of the etched structures achieved with the new layer is much better than with the standard layer. Fourier transform infrared spectroscopy measurements of both samples gave higher transmission in the wavelengths 3350 and 2090 cm$^{-1}$ of SiN type2. The association of those wavelengths with the absorbance of the Si–H and N–H bonds, respectively, means less hydrogen bonds in the layer. Ellipsometric measurements of the two layers (SiN1, SiN2) yielded a refractive index of 2.05 and 2.3, respectively. Combined with relative atomic concentration measured by XPS, this indicates that SiN2 is a more silicon-rich layer. Wet etching of silicon nitride in a HF solution involves a substitution of Si–NH$_2$ bonds by Si–F [5]. The equivalent dry etching process is more chemical in nature with raising the hydrogen content of the layer, in the case of etching with CF$_4$ (no hydrogen source in the plasma). As mentioned before, the chemical etching process is more isotropic and less controlled by the energy and the direction of the ion bombardment in the RIE process.

Fig. 3a–c demonstrates the improvement in the CD control of the pattern definition, switching from SiN type1 to type2 (Fig. 3a and b), and from the etching process developed for type1 to a new dry etching process optimized for type2 (Fig. 3b and c). From an uncontrollable process with abnormal and very wide statistical distribution, we first narrowed the distribution and made it normal by changing the deposition process. Then, by modifying the etching process, we managed to shift the distribution into the desired window dictated by the manufacturing specifications, which were 3, 4 and 5±0.5 μm in the represented cases (indicated by the vertical lines in the graphs).

4. Conclusions

A comprehensive study of PECVD silicon nitride layer characteristics and the way they relate to the deposition process provided better understanding of etching processes, which makes it possible to tailor the dielectric silicon nitride layer to the MMIC device manufacturing needs at any step of the process.

Moreover, selecting suitable process parameters that yielded lower hydrogen content, higher refractive index, and higher thickness uniformity led to significant improvement in CD control of the dry etching process, which, in turn, contributed to finer definitions/design rules of circuit components.

Acknowledgements

Yoram Shapira is grateful to Dinah and Henry Krongold for their generous support.

References