Studies on deposition parameters of silicon-nitride films prepared by a silane-nitrogen plasma-enhanced-chemical-vapour-deposition process
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Silicon-nitride films were deposited by a plasma-enhanced-chemical-vapour-deposition (PECVD) technique using silane-nitrogen as the reactant-gas sources. The influence of the process parameters (such as the flow ratio of the reactant gases, the pressure, the substrate temperature, the radio frequency (r.f.) power, the time of deposition and the electrode spacing) on the deposition and etch rates were investigated and the experimental results are presented in detail.

1. Introduction
Silicon-nitride films have many useful applications in the fabrication of semiconductor microelectronic devices [1]. Silicon nitride is highly suitable as a passivation layer because of its excellent barrier property to moisture and sodium. Silicon nitride is useful as a masking layer for selective oxidation of silicon, because oxygen has difficulty in penetrating it. For selective oxidation, silicon-nitride films could be deposited in a high-temperature (700–800 °C), low-pressure-chemical-vapour-deposition (LPCVD) process, but when used as a final passivation and protective layer the deposition process must be compatible with low-melting-point metals such as aluminium. Thus, silicon nitride must be deposited by a low-temperature process (300–400 °C) [2]. For such applications, plasma-enhanced chemical vapour deposition (PECVD) is the deposition method of choice as nitride films can be deposited at temperatures below 400 °C. The silicon-nitride films that result from PECVD tend to be non-stoichiometric and they have a substantial hydrogen concentration. The hydrogen is bonded to the silicon as Si–H and it is bonded to the nitrogen as N–H. A high hydrogen content (in the range 20–30 at % has been reported as being responsible for degrading metal-oxide semiconductor field-effect transistor (MOSFET) device lifetimes, particularly those with shorter channel lengths [3]. The source of hydrogen in the nitride films is the reactant gases used in the plasma-assisted deposition process, namely SiH₄ and NH₃. To minimize the hydrogen content, low (or no) hydrogen-containing source gases can be employed such as N₂, NF₃, SiF₂ and SiF₄ [4, 5, 6]. Chlorinated gases should be excluded because of the potential corrosion risk to the underlying aluminium. Fluorinated gases have some problems with fluorine incorporation in the nitride films; this changes the film properties [7, 8].

Currently, the most practical solution for the deposition of low-hydrogen-content-silicon-nitride films is to use N₂ instead of NH₃ as the nitrogen source. Through the use of a combination N₂ and SiH₄, a plasma nitride film with a hydrogen content as low as 7% has been reported [9]. However, N₂ has an inherently much higher bonding energy than NH₃, which makes N₂ more difficult to dissociate into free-nitrogen active species, thus the nitride-deposition rate is significantly reduced [10]. But if low-hydrogen-content nitride films can be obtained, which may give better device performance, the deposition rate may not be an important factor and PECVD of silicon nitride using N₂ and SiH₄ is still an attractive process.

The important deposition parameters for any PECVD are substrate temperature, pressure, r.f. power, the flow ratios of the reactant gases and the electrode spacing (in the case of a parallel-plate system). All these parameters have significant influence on the deposition and etch rates and other physical and optical properties of the film. Denser films of silicon nitride may be required for some microelectronic applications; in contrast to applications, such as sacrificial layers, which prefer fast etching rates of the films. In the present work, a deposition parameter matrix was constructed for a SiH₄–N₂ PECVD process and the effect of the variation of the above parameters on the deposition and etch rates were studied extensively.

2. Experimental procedure
A parallel-plate PECVD configuration was used for the deposition of silicon-nitride films using a modified-
reactive-ion etching (RIE) system (Plasma Technology Inc., model RIE-80). The RIE system, used for the plasma-etching process, consisted of a water-cooled bottom electrode acting as the cathode and a top electrode as the anode. The electrodes were made out of anodized aluminum. By changing the electrode configuration, the RIE system was converted to a PECVD system where the bottom electrode (diameter 24 cm), with the substrate heater \(T < 400 \, ^\circ\text{C}\), acted as the anode, and the top electrode (diameter 24 cm), with water cooling, acted as the cathode. The system consisted of four main blocks: the main body, the pumping system, the gas cabinets and a gas pod, as shown schematically in Fig. 1. The main body was a chamber with top and bottom electrodes, the process logic, a safety interlock, and an r.f. generator with its tuning circuit. The system had two pumps; a molecular drag pump (Alcatel MDP 5030 CP) used as a process pump with a controller, and a mechanical pump used as a roughing pump with foreline, roughing, throttle and vent valves. The oil-free molecular-drag pump achieved a background pressure of \(1 \times 10^{-3} \, \text{Pa}\) and it reduced the contamination in the film. Two gas cabinets, one for silane gas and the other for nitrogen gas, were used with a high-pressure nitrogen capability for safety purposes. The gas pod had two process-gas controllers and a gas-mixing manifold. Both the nitrogen and the silane lines had their own mass-flow controllers (0–100 s.c.cm.) and they were connected to the gas-mixing manifold. These mass-flow controllers were calibrated for the respective gases by the manufacturer. The mixed gases were distributed near the substrates via a shower head in the top electrode. A 3%-silane-in-helium mixture was used as the silicon-reactant source. In order to use the mass-flow meter for this diluted silane source, the required conversion factor was calculated on the basis of the density and the specific-heat values. This factor was found to be 1.0352 for a 3%-silane-in-helium mixture which, corresponds to a 2.3506 cm\(^3\) gas mixture for 1 cm\(^3\) of 100% silane. The purity of the silane and the nitrogen was of electronic grade (99.999%).

The substrates used for the study were n-type silicon (100) wafers polished on one side. The silicon wafers were cleaned before loading into the chamber [11]. When a vacuum of the order of \(1 \times 10^{-3} \, \text{Pa}\) was achieved, the throttling was adjusted to obtain the required pressure for deposition. The system was then purged with nitrogen at this pressure to clean the system of any \(\text{H}_2\text{O}\) residuals. Before introducing the silane, the plasma was established with nitrogen, and this plasma was continued for 3 min. The required deposition parameters were then adjusted and all the conditions were stabilized before the deposition was started. During the deposition the deviations from the set readings were less than 10%. After completing the deposition and before opening the system, the process-gas lines were cleaned with a nitrogen purge to remove any residual trapped silane.

The deposition parameters studied were: the flow ratio of silane to nitrogen, the chamber pressure, the r.f. power, the substrate temperature, the deposition time and the electrode spacing. The range of these parameter variations are listed in Table I with their abbreviations. For each set of experiments one parameter was varied while all the other parameters were fixed. Similar experiments were conducted for all the other parameters and their effect on the deposition and etch rates was studied. After the deposition was completed, a step was etched in the film and an optical-interferometer technique was used to determine the thickness of the deposited film. The etch rates were determined by etching the silicon-nitride film with a buffered oxide etch (13NH\(_4\)F:2HF equal to 9:1) diluted in water.

### 3. Results

#### 3.1. Effect of the silane-to-nitrogen flow ratio

The range of the silane-to-nitrogen flow ratio tested was 0.0005–0.030. Fig. 2 shows the variation of the deposition and the etch rate with respect to an increasing gas-flow ratio of \(\text{SiH}_4/\text{N}_2\). The r.f. power and the substrate temperature were kept constant. It can be seen that the deposition rate slowly increased when the gas-flow ratio increased; this is consistent with observations made by Gereth and Scherber [12]. It has been also found that an increase in the chamber pressure causes an increase in the deposition rate. The same trend was reported by Lee et al., Sinha et al. and Piccirillo and Gobbi [13, 14, 15] for nitride films deposited by a silane/ammonia process. This is due to the fact that as the amount of silane increases the films get richer in silicon, and silicon-rich films have higher growth rates.

![Figure 1 A schematic diagram of the PECVD system: (o-o) valve, (O) regulator, and (O) filter.](image-url)