

REACTIVE PLASMA ETCHING OF SILICON CARBIDE, SILICON
CARBONITRIDE, AND TITANIUM NITRIDE IN A
TETRAFLUOROETHANE / OXYGEN PLASMA

THESIS

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by

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ABSTRACT

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SUPERVISING PROFESSOR: HEATHER GALLOWAY

In this study we investigate the etching of the diffusion barrier between the silicon substrate and the low- κ dielectric material. We will use silicon carbide (SiC), silicon carbonitride (SiCN), and titanium nitride (TiN) as our diffusion layer. Using the RF plasma and the magnetron gun with 1,1,1,2 tetrafluoroethane and oxygen as the active gases, we will obtain a reliable and reproducible method for determining the maximum etch rate for the various materials. An etch is required in order for a proper ground contact with the substrate. Si and Al selectivity will also be discussed. The etch process along with the aluminum mask are prepared using standard photolithography techniques, and reactive ion etching (RIE) parameters (pressure and power) will be discussed. RIE is chemistry-specific process that enables the selective etching of sample elements. Etch profiles were taken using a profilometer.

CHAPTER 1

INTRODUCTION

In the move to Cu/low-k, the metal lines of the wafers will have a silicon carbide (SiC), silicon carbonitride (SiCN), or titanium nitride (TiN) under layer between the silicon substrate and the low-k. This layer acts as a diffusion barrier for the copper. Ground lines were created in order to reduce the resistance of the ground return through the substrate. To contact the substrate, a small trench or via had to be etched through the low-k and the SiC, SiCN, or TiN. These materials are rather chemically resistant materials and are typically etched with fluorinated plasmas.

The ability to pattern thin films of SiC, SiCN, and TiN is a critical step in the fabrication of new high-temperature, high-speed, and high-power electronic devices. In particular SiC is known to be suitable for high-power devices due to its high thermal conductivity, high breakdown electric field strength, wide bandgap chemical stability and high current densities. SiCN and TiN have characteristics that are similar to SiC. The relative inertness of silicon carbide has caused many problems with the processing of the material. Techniques that have been established, such as wet etching with strong acids, are not possible for the etching of silicon carbide [1]. The strong bond nature of these semiconductor materials requires plasma-based etching in order to take advantage of the relative anisotropy that is required at the sub-micron scale.

Reactive ion etching with fluorinated plasmas has been developed to the point where it is widely employed for both research and commercial product fabrication. The most common and in many cases, the most effective fluorinated gases for plasma etching have very high global warming potentials. Some fluorinated hydrocarbons (HFC's) on the other hand have very high fluorine content as well as being an environmentally benign replacement for previously used environmentally damaging gases.

Prior to any etching, standard photolithography is used to create waveguides on the surface of the thin film. This process includes a series of steps using photoresist, the semiconductor laser writer, an acid "wet etch" for the removal of aluminum, and a sputtering system used to deposit an aluminum film of specific thickness. The end result of this process is a varying trench through the thin film of SiC, SiCN, or TiN down to the silicon substrate that is then filled with aluminum. This allows testing of its electrical properties by sending current through the waveguides to a sensor on the other end. The majority of electrical devices require ohmic contact for maximum performance of electrodes. The quality of the contacts between the ground lines and the wafer was checked by electrical characterization.

Before any of the electrical testing was done, an etch rate test was done in order to obtain a trench that would be deep enough to go through the diffusion layer to the silicon substrate. This trench was needed so that there would be access to the substrate for a ground contact between the substrate and the ground lines. In fabricating microelectronics device, plasma etching can be used to pattern the substrate surface. During plasma etching, both energetic ions and reactive radicals are created.

Throughout this study, the primary aim was to study the effectiveness of dry etching of silicon carbide, silicon carbonitride, and titanium nitride within a tetrafluoroethane/oxygen gas mixture using reactive ion etching (RIE). Silicon carbide has been previously etched in other fluorinated plasmas, with additions of argon or hydrogen. The additions have been used to improve the surface roughness in RIE etchers [3].



Figure 1. Cross section of a low-k wafer as is

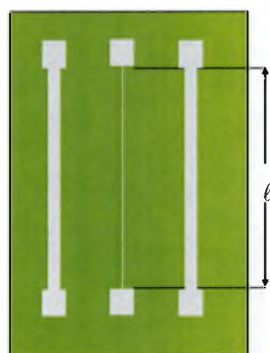


Figure 2. Top view of a microstrip transmission line. The outer two lines are the ground planes, and the middle is the signal line. The contact pads are the squares located at the ends of each line.

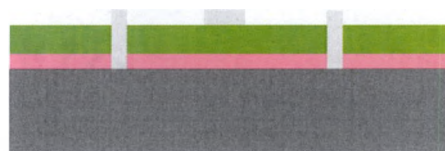


Figure 3. Cross section of a microstrip on a low-k wafer. This is showing the ground lines contacting the Si substrate.

CHAPTER 2

PROPERTIES OF DIFFUSION LAYERS

PROPERTIES OF SILICON CARBIDE

Silicon carbide is a semiconductor with attractive material properties for microelectronic applications. SiC is also an important semiconductor for high temperature, high power, and high frequency electronics due to its large band gap, high breakdown field, and high thermal conductivity [4,5]. SiC has a unique stability that allows it to be used for applications in aerospace technologies and for microelectromechanical systems (MEMS) [6,7].

The material properties of silicon carbide make the material useful for numerous applications. The wide band gap of SiC enables the device to operate at high temperatures without problems arising due to the intrinsic conduction effects. SiC is also able to emit or detect short wavelength photons due to the wide bandgap, making it useful for emitter or detector technologies in the ultraviolet region. The high thermal conductivity of silicon carbide shows that the material is a good conductor of heat. Therefore, heat and energy flow easily throughout the material, for operation at high power and at high temperatures with excess heat being efficiently dissipated from the device. Moreover, silicon carbide can withstand a high breakdown electric field, several times higher than that of silicon, allowing the material to be applied in a high voltage and a high power device without the

danger of avalanche breakdown, and for devices to be closely packed together on SiC integrated circuits.

Property	SiC	SiCN	TiN	Si
Bandgap	3.26 eV	2.0-3.0 eV	3.35 - 3.45 eV	1.12eV
Breakdown electric field (v/cm)	2.2×10^6	na	na	2.5×10^5
Thermal conductivity (w/cmK)	3.0-3.8	na	na	1.24

Table 1. Material properties of SiC, SiCN, and TiN with comparison to Si

PROPERTIES OF SILICON CARBONITRIDE

Table 2 lists some physical properties of the SiCN, together with those of silicon and SiC for comparison. The major properties of the SiCN are in the same range as those of SiC and superior over those of Si. The SiCN also shows creep resistance as good as SiC, while its oxidation resistance is even better than SiC. Obviously, the thermal shock resistance appears very promising for SiCN. Thus SiCN ceramics are promising for high temperature applications [10].

	SiCN	Si	SiC	TiN
Density (g/cm³)	2.20	2.33	3.17	5.43
E Modulus (GPa)	158	163	405	465
Poisson's Ratio	0.18	0.22	0.14	0.25
CTE (x 10⁻⁶/K)	0.5	2.5	3.8	9.4
Hardness (GPa)	15	11.2	30	18
Strength (MPa)	250	175	418	972
Toughness (MPa.m^{1/2})	3.5	0.9	4-6	5

Table 2. Comparison of Physical Properties between SiCN, Si, SiC, and TiN [10,11]

PROPERTIES OF TITANIUM NITIDE

Titanium nitride (TiN) is commonly used as a diffusion barrier in integrated circuitry, serving to prevent the diffusion or migration of conductive materials into insulating materials and active regions of transistors. It also serves as an adhesion promoter

that eliminates delamination and voids between the conductive materials and the surrounding region of insulating, dielectric material [19].

Although copper interconnects have been introduced to high speed logic devices, aluminum interconnects are still used for the majority of large scale integrated circuits (LSIs). TiN is used because the layer prevent interconnects from electromigration failures and also because the bottom TiN layer prevents the diffusion of the Al in Si which might cause short-circuit failures due to the formation of Al alloy spikes through diffusion wells to the Si substrate [20].

Titanium nitride has also been most widely used as a diffusion barrier in Ultra-Large Scale Integration (ULSI) devices because of its very low resistivity, good chemical and thermal stability, and impermeability to silicon diffusion as well as the excellent adhesion to silicon and SiO₂ films. TiN barrier layers have been deposited predominantly by sputtering and chemical vapor deposition methods.

CHAPTER 3

PLASMAS

INTRODUCTION

Plasmas are considered the fourth state of matter and they are used in this work for sputtering and etching. Plasmas are composed of collections of free moving electrons and ions. In the magnetron, applying a high voltage across a low-pressure gas, usually argon, creates plasma. Plasmas contain both ions and neutrals so it is considered a charged gas, with high energy electrons causing excitations and ionizations within the plasma. Although plasmas are in the gas phase, the dynamics of the plasma may be described by fluid equations and therefore no information from the plasma motions is lost. An explanation of the physics and chemistry within plasmas can be found in several textbooks [15]. Discussions of the etch mechanisms, and the advances in the field from the past are described in several review articles [12-14].

This study is necessarily limited to the main ideas behind the explanations and a brief outline of the equations governing the plasma, the Lorentz force law, the Boltzmann equation, the concept of the distribution function and the Debye length. The plasma is really a collection of particles for which the motion of any one particle can be explained by Maxwell's equations and the Lorentz force law. The Maxwell equations for electromagnetism and the plasma Boltzmann equation are the basic equations for studies of electromagnetic systems.

PHYSICS OF PLASMAS

A single particle's motion can be described using the Maxwell distribution function and the Lorentz force, but the following solution does not consider the Coulomb field produced by the particle itself and the effect of that field back onto the plasma. To explain this behavior it is necessary to consider the origin of both the long range and the short range interactions which are taking place.

The long range interactions are the Coulomb interactions, caused by the magnetic and electric fields acting upon the system. These fields are both external and internal, coming not only from the fields produced by the reactor, but also from the internal fields produced by the charged particles within the ionized gas. Complications in the plasma analysis arise from the coupling of the interaction. The short range interactions are due to the collisions between the atoms, molecules and electrons. The collisions taking place within the ionized gas occur on a shorter length scale than those of the Coulomb interactions. The Debye length given by equation 1 determines the length scale of these interactions.

$$\lambda_{De} = (\epsilon_0 T_e / e n_0)^{1/2} \quad \text{Equation 1}$$

The long range Coulomb interactions are governed by the Coulomb interaction fields. The fields exert forces on the plasma given by the Lorentz force law shown in equation 2.

$$\mathbf{F} = m\mathbf{a} = q (\mathbf{E} + \mathbf{v} \times \mathbf{B}) \quad \text{Equation 2}$$

Fluid equations are used as an advantage to describe the motion of the plasma. The complex motions caused by the oscillations of the particles within the plasma are retained, including the wave nature of these motions. A distribution function is needed where the velocity and position of the particles, at a given time, should be considered to study the motion caused by this force. E.g. the particles are considered in six dimensional phase space $(\mathbf{r}, \mathbf{v})$ by means of a distribution function, equation 3

$$f(\mathbf{r}, \mathbf{v}, t) d^3r d^3v \quad \text{Equation 3}$$

The simplest distribution function is a Maxwellian distribution function. The Maxwellian velocity distribution function is shown in equation 4.

$$f = \left(\frac{m}{2\pi k_B T} \right)^{\frac{3}{2}} \exp\left(-\frac{v^2}{v_T^2} \right) \quad \text{Equation 4}$$

However, if the plasma did follow a true Maxwellian distribution function it would be uniform in all space. In that case, high energy electrons and ions, which provide the characteristic behavior of the plasma, would be lost. It is therefore important to use an applicable distribution function, although the determination of this function can be complicated.

The analysis so far has also considered all of the particles as distinguishable. From quantum mechanics, it is known, that particles cannot be considered as distinguishable in a real system, thus adding further complications to the full analysis. Nevertheless, it is possible to approximate the plasma as a non-interacting group of particles using the Lorentz force law and a localized Maxwell distribution function while still retaining the

Coulomb interaction fields. A fuller understanding of plasma dynamics can be reached using kinetic theory. The distribution function given by equation 3 may be substituted into the collision less Boltzmann equation or the Vlasov equation as shown below.

$$\frac{\delta f}{\delta t} + \mathbf{v} \cdot \nabla_r f + \frac{\mathbf{F}}{m} \cdot \nabla_v f = \frac{\delta f}{\delta t} \Big|_c \quad \text{Equation 5}$$

Where $\mathbf{F}$ is the Lorentz force. Due to the wave nature of the fields applied, oscillations are taking place in the plasma. On the local scale the oscillations can be considered by Newtonian mechanics with the vibrations of electrons and atoms being similar to those of vibrations on a string. This leads to the electron plasma frequency in equation 6 and the ion plasma frequency in equation 7.

$$\omega_{pe} = \left(\frac{e^2 n_0}{\epsilon_0 m} \right)^{\frac{1}{2}} \quad \text{Equation 6}$$

$$\omega_{pi} = \left(\frac{e^2 n_0}{\epsilon_0 M} \right)^{\frac{1}{2}} \quad \text{Equation 7}$$

The natural frequency of the plasma is then

$$\omega_p = \left(\omega_{pe}^2 + \omega_{pi}^2 \right)^{\frac{1}{2}} \quad \text{Equation 8}$$

This can be related to the Debye length and the thermal velocity of the particles within the plasma by

$$\lambda_{De} = \frac{v_{th}}{\omega_{pe}} \quad \text{Equation 9}$$

By beginning with the Coloumb force interactions, it is also possible to derive the fundamental length scale of the particle and the oscillations observed. However, the mathematics becomes more complicated. This section has briefly outlined the principle physical theories pertaining to the plasma. A detailed explanation of the full physics is not always essential to understand the etch processes involved in the magnetron to get a plasma.

MAGNETRON

DC magnetron sputtering is a powerful and flexible technique that can be used to coat virtually any work piece with a wide range of materials - any solid metal or alloy and a variety of compounds. DC magnetron sputtering is a low pressure plasma process that involves the removal of atomized material from a solid due to energetic bombardment of its surface layers by ions or neutral particles. A permanent magnet structure is located behind the target serving as a deposition source. The resulting magnetic field forms a closed-loop annular path acting as an electron trap that reshapes the trajectories of the secondary electrons ejected from target into a toroidal path, greatly increasing the probability of ionization of the sputtering gas within the confinement zone. Inert gases with a large atomic mass, specifically argon, are usually employed as the sputtering gas because they tend not to react with the target material or combine with any process gases and because they produce higher deposition rates due to their high mass.

Positively charged argon ions from the plasma are accelerated toward the negatively biased target (cathode), resulting in material being sputtered from the target surface. Oxygen and nitrogen can be added to form oxides or nitrides. This is one method of sputtering compounds. This material will then begin to deposit elsewhere in the chamber. A substrate (anode) placed above the magnetron gun will get coated with the target material.

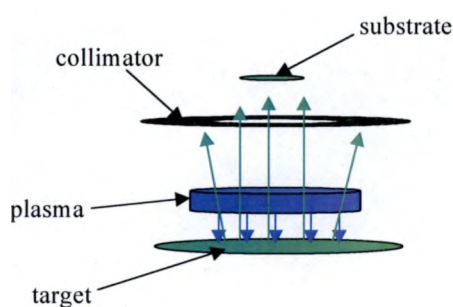


Figure 4. Schematic of magnetron gun



Figure 5. View of magnetron chamber

In this study, the chamber was pumped to a base pressure of at least 4×10^{-6} Torr using a Pfeiffer vacuum TC600 Turbo pump in stand by mode (550 Hz). An Argon flow rate of 7.75 sccm (standard cubic centimeters) controlled with a MKS 247C controller and a 0-10 sccm mass flow controller resulting in a pressure of 6 mTorr with the throttle valve fully closed and the turbo pump on stand by. The MDX-1K DC power supply was used with a power of 200 Watts, and the Sparc-le 20 was set to Arc Out Mode yielding a deposition rate of ~ 100 Angstroms a second.

The Sparc-le 20 (Small Package Arc Repression Circuit-Low Energy) 20kHz is the core to pulsed DC sputtering. It receives the DC power from the MDX power supply and in Arc Out mode functions as a resonant LC network. During an arc, it will discharge and

then commutate off the arc through its reversing action. The ability to quickly clear arcs improves film quality and lessens defects.

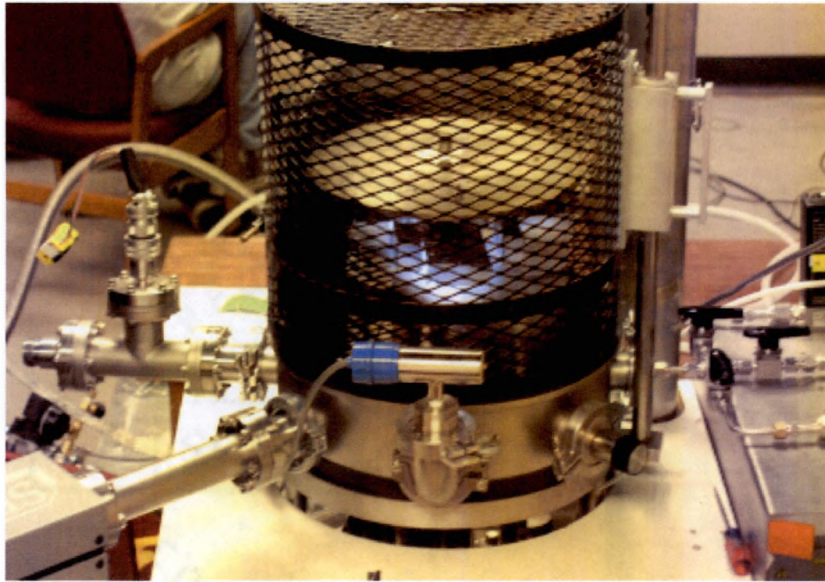


Figure 6. View of magnetron while etching

REACTIVE ION ETCHING

In Reactive Ion Etching (RIE), the substrate is placed inside the magnetron in which several gases are introduced. Gas pressure is typically maintained in a range between a few millitorr and a few hundred millitorr by adjusting gas flow rates and/or adjusting an exhaust orifice. The RF power is used to create an oscillating electric field that ionizes the gas molecules by stripping them of electrons, creating plasma. The ions are accelerated towards, and react at the surface of the material being etched, forming another gaseous material. This is known as the chemical part of RIE. There is also a physical part that is similar in nature to the sputtering deposition process. If the ions have high enough energy, they can knock atoms out of the material to be etched without a chemical reaction. It is a very complex task to develop dry etching processes that balance chemical and physical

etching, since there are many parameters to adjust. By changing the balance it is possible to influence the anisotropy of the etching, since the chemical part is isotropic and the physical part is highly anisotropic. Etch conditions in an RIE system are very much dependent on the many process parameters, such as pressure, gas flows, and RF power.

The material removal in reactive ion plasma etching is a combination of several mechanisms [1]. The mechanisms are: sputtering, chemical plasma etching, ion-enhanced chemical etching, and inhibitor controlled chemical etching. Sputtering is the physical removal of material by energetic ion bombardment. Chemical plasma etching is where the neutral radicals produced in the plasma remove material by chemically reacting with the substrate material and producing volatile species. Ion-enhanced chemical etching takes place when energetic ions damage the surface of the substrate enhancing the absorption rate of the etching species. Inhibitor controlled chemical etching is when the inhibitor layers are removed by ion bombardment allowing the reaction to proceed. Giving an overall etch rate of R:

$$R = R_{\text{Sputter}} + R_{\text{Neut}} + R_{\text{IEN}} + R_{\text{ICN}} \quad \text{Equation 10}$$

The effects of the arrival rate of ions and neutrals on the overall etch rate can be explained by expanding the sputter rate equation [12, 15] as follows:

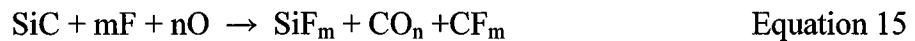
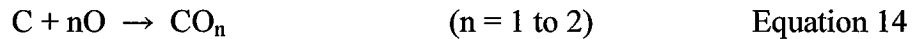
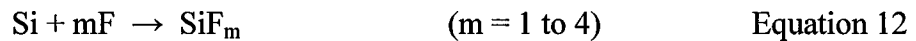
$$R = F_1 \phi_s + F_N (1 - \alpha\alpha - \beta) \phi_N + F_N \alpha \phi_N^* + F_N \beta \phi^* N^* \quad \text{Equation 11}$$

Where F_1 is the ion flux (ions/cm² s), F_N is the flux of neutral particles, ϕ_s is the sputtering efficiency (cm³/ion), ϕ_N is the chemical etch rate efficiency of neutral species (cm³/neutral), ϕ_N^* and $\phi^* N^*$ are the chemical etch rate efficiencies of neutral species on

the fraction ϕ of the surface which has been sensitized, ion bombarded, and on the surface fraction ϕ covered by an etch inhibitor. This model explains the association between the neutral radicals and ion plasma species involved in the etching process. The ion flux increases the sensitized surface fraction, and negatively affects the inhibitor covered fraction, enhancing the chemical neutral etch rate.

PLASMA ETCH THEORY

Silicon carbide and silicon carbonitride have been successfully etched using fluorinated plasmas. Many researchers have also investigated both fluorinated and chlorinated plasmas. The information from these investigations has allowed for the etch mechanisms of the materials to be uncovered. It is postulated that the etching of SiC is successful during plasma etching due to the ions striking the substrate surface with sufficient energy to break the Si-C bond. For SiC etching in mixtures of fluorinated gases and oxygen, the most probable chemical reactions for the removal of Si and C atoms are given in the following equations [1]:



where the carbon comes from the SiC substrate itself.

Initially, parts of the Si-C double layer were removed by physical sputtering, and then cracking of the Si-C bonds took place within the plasma sheath by electron and ion bombardment. The final stage required the free Si and C atoms to be saturated by F and O radicals and to form volatile products. More recently, in [2], where the gas phases and etch phase products were studied, it was concluded that after the incident ions have weakened or broken the Si-C bond, the fluorine atoms from the etching gas react preferentially with the Si atoms. This then results in a carbon rich layer being formed. The removal of this carbon layer is thought to be a rate limiting step, but is accelerated by the presence of oxygen within the etch gas which allows for new volatile products to be formed.

An anisotropic etching profile was obtained for SiC in SF_6/O_2 mixtures, which is not the case of the normally undercut etching profile for Si [16, 17]. This is due to the SiC itself providing carbon. That carbon promotes polymer formation which prevents the sidewall from being etched. There was not a fluorinated gas investigated by [1] which produced an undercut profile during the SiC etching. Therefore we can assume that HFC 134a will also produce an anisotropic etch. In addition to the indirect roles of oxygen in the gas phase reaction, oxygen also takes part by directly removing C atoms in SiC through the reaction given in equation 14. Carbon can be etched in either pure fluorine containing plasma (equation 13) or pure O_2 (equation 14) plasma. Several references have reported that a thin carbon rich layer is formed on the etched surface, indicating that C is not removed sufficiently fast from the etched surface through the reaction of either carbon-fluorine, C-F, or carbon-oxygen, C-O, reactions [1]. At low O_2 percentage, it has been suggested that carbon is preferentially removed through the formation of CF_m (equation 12) rather than C-O_n (Equation 13), whereas at high O_2 percentage the removal of carbon is

dominated by the C-O reaction. However, generally the SiC etch rate decreases as the O₂ percentage increases. Therefore the C-O reaction may not be as efficient as the C-F reaction [1]. Along with the purely chemical plasma etching process, the effect of the energetic ion flux needs to be considered. The ion bombardment breaks the surface Si-C bond (4.52 eV), which enhances the chemical reaction efficiency, and removal of non-volatile surface species. This enables the chemical reaction to proceed, including providing sufficient energy to break the strong C-C bonding (6.27 eV) that could exist in the C-rich layer [1]. This combination of effects have led to a two system model for the effect of DC bias on the etch rate of polycrystalline SiC [21]. The first system is at low dc bias conditions, the low energy and effectiveness of the ion flux is the dominant mechanism. The second system is at sufficiently high values of the dc bias, the ion energy is high enough to no longer limit the process and the etch rate is determined by the removal efficiency of the chemical reaction [1].

There was not any etch rate data available for silicon carbide, silicon carbonitride, and titanium nitride with a HFC 134a/oxygen mixture. Therefore this process required an etch rate study in order to determine the right mixture of HFC 134a and oxygen, and their respective etch rates. A constant flow of HFC 134a was investigated as compared to various oxygen flow rates.

CHAPTER 4

ETCH MECHANISMS

INTRODUCTION

The primary aim of the investigation is to study the effectiveness of dry etching of silicon carbide, silicon carbonitride, and titanium nitride with a HFC 134a/ O₂ gas mixture in a laboratory magnetron. The work began with an examination of the different oxygen flow rates to determine the optimum flow to etch the deepest trench.

HFC-134a

A method was devised using a magnetron gun attached to an RF network as a plasma source and a mixture of 1,1,1,2-Tetrafluoroethane or HFC 134a and O₂ as the process gas. Tetrafluoroethane is of interest due to its high fluorine content, and it is readily available. In fact, a can of Genetron 134a that was used for this study is made by

Interdynamics[®] and was purchased at a local automotive parts store. It is also a nontoxic, ozone friendly gas with a short atmospheric lifetime

and a low global warming potential. This gas is available at higher purities but was not commercially available in small quantities making the purchase of a higher purity prohibitively expensive.



Figure 7. HFC 134a used for studies

HFC 134a specifications:

–1,1,1,2-tetrafluoroethane 99.5%

–Formula: $C_2H_2F_4$

–Molecular Weight: 102.03

–CAS Registry Number: 811-97-2

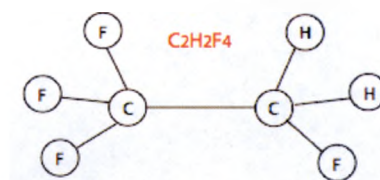


Figure 8. HFC 134a molecule

Fluorinated compounds are extensively used in plasma processing applications in the microelectronics industry. Many of the fluorinated compounds used in plasma processing have high global-warming potentials (GWPs) and the reduction of global-warming gas emissions from plasma processing is an issue of growing significance. One of several approaches to this problem being considered is to identify substitute plasma chemistries based upon gases having lower GWPs than those currently in use [8].

HFC-134a has emerged as a leading low GWP replacement for chlorofluorocarbon refrigerants. The only reported investigation of etching with HFC-134a plasmas is that of Kirmse, et al. [9]. Incidentally, they selected to study HFC-134a electron cyclotron discharges not for environmental reasons, but rather because of the high carbon-to-fluorine ratio of this molecule. Nevertheless, HFC-134a has a considerably shorter atmospheric lifetime, and lower GWP than many conventional plasma processing gases (Table 3).

Greenhouse Gas	Atmospheric Lifetime (yrs)	GWPs
Carbon Dioxide (CO ₂)	5-200*	1
HFC-134a	9.6	1,300
NF ₃	740	10,800
SF ₆	3,200	22,000
CF ₄	50,000	5,700

*Carbon Dioxide varies spatially in the atmosphere

Table 3. HFC 134a compared to other gases used for etching [8]

EXPERIMENTAL SETUP

For this study, the samples consisted of SiC, SiCN, or TiN on top of a Si substrate. It was determined that the SiC samples consisted of ~350 nm of SiC, SiCN consisted of about ~200 nm of SiCN, and TiN consisted of ~40 nm of TiN on the Si substrate. The samples were cut into 2 cm² pieces. The samples were all cleaned with acetone and Isopropanol alcohol (IPA). The thicknesses of these films were measured optically with a Filmetrics system or were calculated using the four-point probe. These measurements correlated to profilometer measurements taken after etching. Then, 1000 nm of Aluminum was sputtered on to the substrates. After this, the laser writer was used to create a trench on the surface. The etch samples were then placed on top of a Si target in the magnetron gun, and arranged so that the highest plasma density was in the area of the mask features. In past research, the scaling control setting was set at a different value. Using that value, and the new gas correction factor, it was determined that a fixed value of HFC 134a at a flow rate of 11 sccm was high. With the corrected value a new value of 5.5 sccm was used. The HFC 134a flow rate was fixed at 5.5 sccm and the O₂ flow was studied at 4, 6, 7.33, 9, and 11 sccm, resulting in a process pressure around 20 mTorr. All etches were done at 50 Watts RF (0.6 Watts/cm²). The substrates were allowed to self-bias during etching and the

computer recorded the bias voltage. The computer also controlled the RF network that powered the magnetron guns.

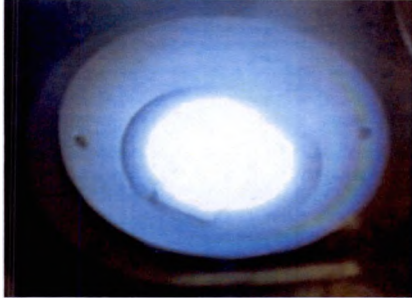


Figure 9. Samples being etched in the magnetron gun by a plasma

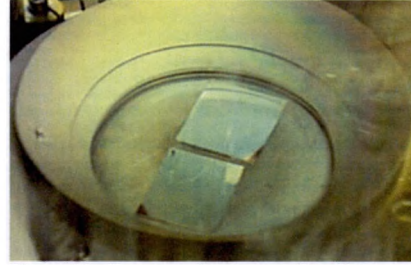


Figure 10. Two samples on top of an Si target. Samples are on the magnetron gun before etching.

The RF network that powered the magnetron consisted of an Advanced Energy RFX600A 600 Watt RF power supply, and an Advanced Energy ATX-600 impedance-matching network. The RF power is used to create an oscillating electric field that ionizes the gas molecules by stripping them of electrons, creating plasma. The power supply generates a typical RF power signal at 13.56 MHz, and is designed to power a 50 Ω non-reactive load. Because the basic characteristics of plasma can be electrically represented as a diode, a resistor, and a capacitor in parallel, it has reactive impedance that is related to the chamber, the ion composition and other chamber conditions. The diode effects arise from the fact that the much lighter electrons can move much faster than the ions. In order to provide the non-reactive 50 Ω load, a matching network is connected in series between the power supply and the magnetron gun.

The function of an impedance-matching network is to transform the resistive and capacitive characteristics of the plasma to 50 Ω . The impedance-matching network is constructed of a combination of capacitors, resistors, and inductors. There are two active components in the matching network, a forward power and a reflected power capacitor. These capacitors are variable open-air capacitors that are controlled either manually or by

the ATX-600 controller to tune the system to the required $50\ \Omega$ load, thus matching the load impedance to the AC generator's impedance.

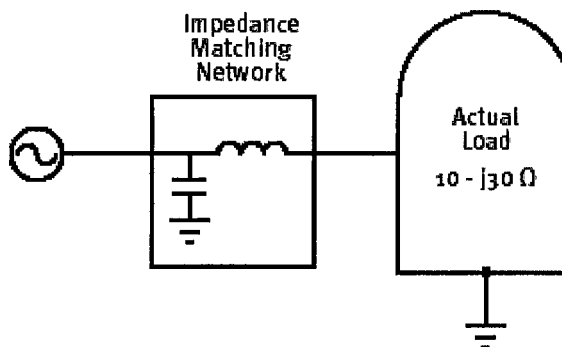


Figure 11 Impedance matching network schematic This is attached to the magnetron chamber

A system was developed for computer automating the power supply with the use of a software package called Labview, and a PCI-6023E data acquisition (DAQ) card both by National Instruments. Labview is a Microsoft Windows based graphical programming package that gives the programmer the ability to easily design logic circuits and manipulate both analog and digital, in and out signals, with the use of a DAQ card. This provides the ability to interface with the pin-out on the RFX600A power supply. The Labview is also used to set the device parameters, and record the forward and bias voltages at 0.1 s intervals

ETCH RATE RESULTS

The etch rates were determined by measuring the feature depth with a Tencor Alpha step 500 stylus profilometer. Further information about the etch profile and roughness was collected with an Atomic Force Microscope. As depicted in the graphs in the appendices, the highest etch rate of $92\ \text{\AA}/\text{s}$ was achieved at a mixture of 5.5 sccm HFC 134a and 6.0 sccm O_2 for SiC. For SiCN, an etch rate of $83\ \text{\AA}/\text{s}$ was achieved at a mixture

of 5.5 HFC 134a and 4.0 sccm O_2 . TiN had an etch rate of 33 Å/s was achieved at a mixture of 5.5 HFC 134a and 4.0 sccm O_2 .

Etch Rates of Various Fluorinated Gasses (Å/Min)				
	HFC 134a / O_2	CF_4 / O_2	CHF_3 / O_2	$CBrF_3$ / O_2
SiC	5520	525	675	570
Si	2760	1050	465	225
Selectivity (SiC/Si)	2.0	0.5	1.5	2.5

Table 4. Etch Rates of Various Fluorinated Gasses (CF_4 , CHF_3 , and $CBrF_3$ are from reference 3. The original values were given at 0.4 W/m², they were linearly interpolated to 0.6 W/m² for comparison to HFC 134a

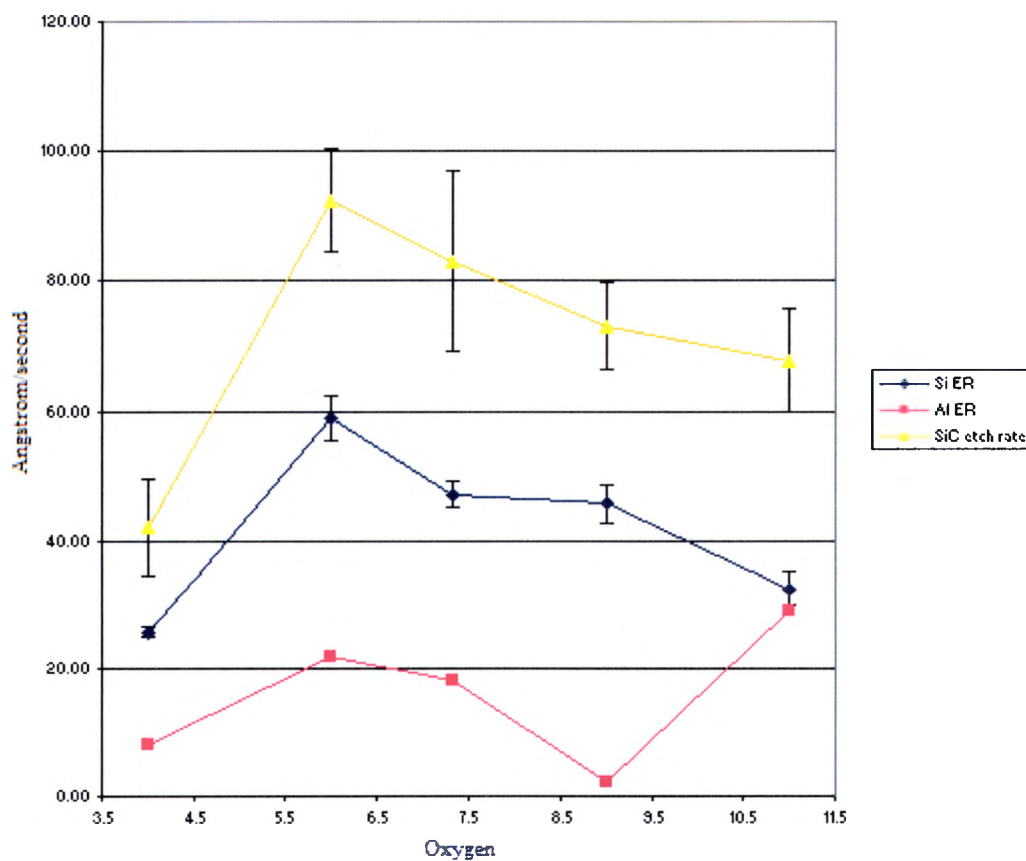


Figure 12. SiC etch rate vs. oxygen flow for Si, SiC, and Al

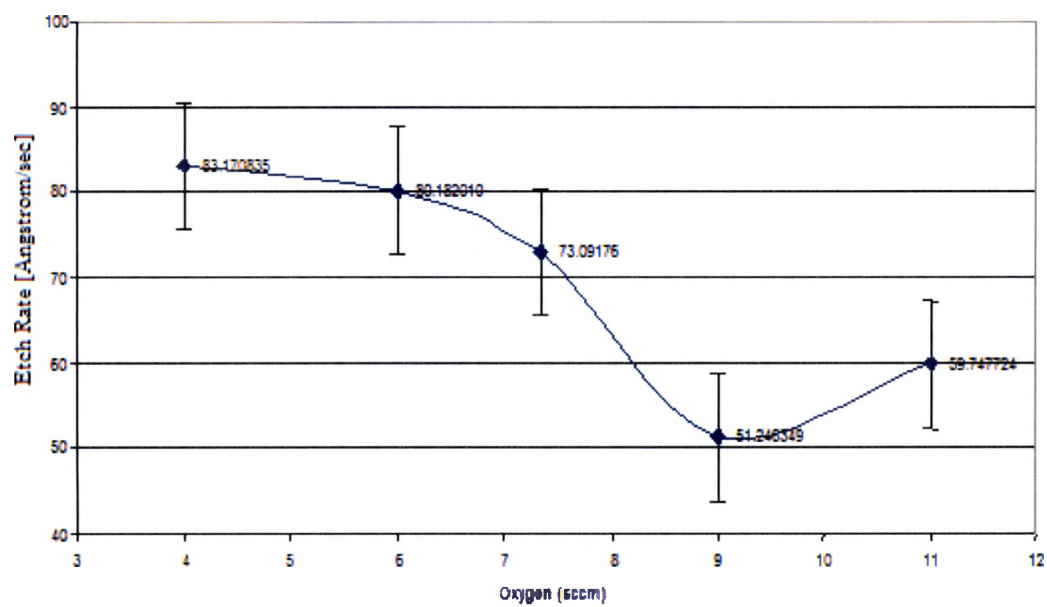


Figure 13. SiCN etch rate vs. oxygen

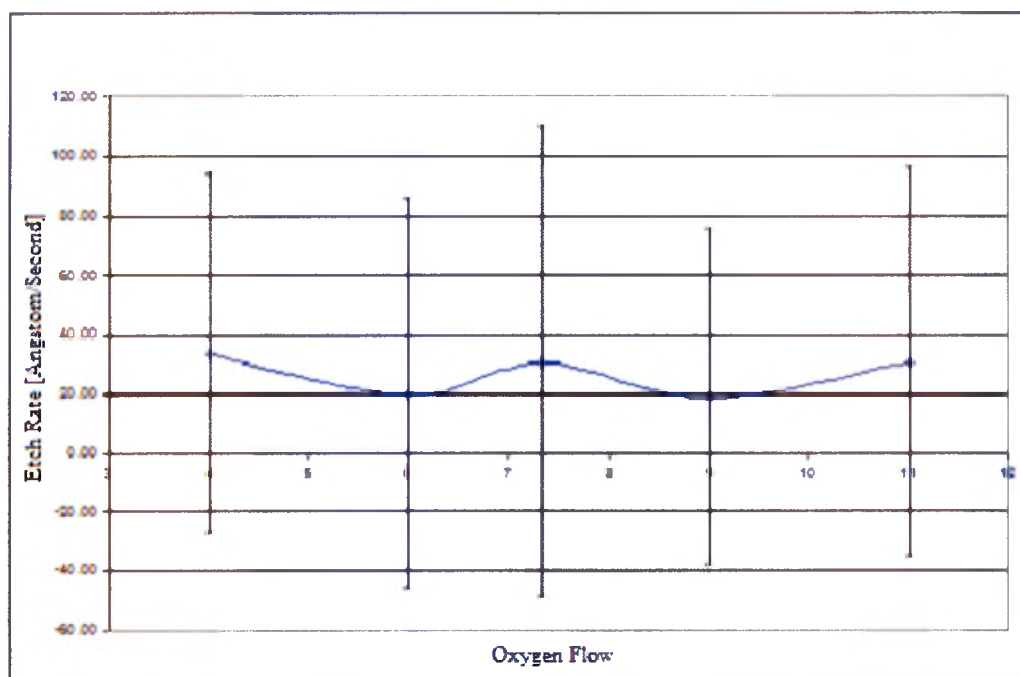


Figure 14. TiN etch rate vs. oxygen

CHAPTER 5

FABRICATION

INTRODUCTION

The fabrication process for the creation of the trenches used standard laboratory methods for building microelectronic devices. The aluminum films were used for masking the substrate during plasma processing. Semiconductor laser beam lithography was used for creating the photo masks used to create the various aluminum features. The plasma processing was done in a RF magnetron gun using HFC 134a and oxygen gases. Each of these processes will be detailed later. The masked wafer is then etched in the magnetron with a plasma consisting of HFC 134a (1,1,1,2-tetrafluoroethane) and O₂ at 20mTorr to remove the exposed low-k and SiC under layer. In this process the primary mechanism for material removal is F-C and F-Si reactions between fluorine ions and the Si and C atoms, followed by ion sputtering removal. This etch process should be anisotropic in nature due to the SiC providing carbon, enhancing polymer formation on the sidewalls of the feature. This polymer inhibits sidewall etching and therefore stops undercutting.

OUTLINE OF FABRICATION PROCESS

1. Preclean samples in acetone and then isopropanol and blow dry with nitrogen.
2. Sputter 3000 Å of aluminum for mask in magnetron.

3. Spin on photoresist.
4. Photolithography with appropriate file.
5. Develop and inspect for proper exposure with 10x microscope.
6. Aluminum wet etch until feature visually turns different color.
7. Strip mask with acetone then wash in isopropanol and blow dry.
8. Plasma etch in magnetron.
9. Strip aluminum mask with aluminum etch, clean, dry and inspect.
10. Measure with Profilometer.

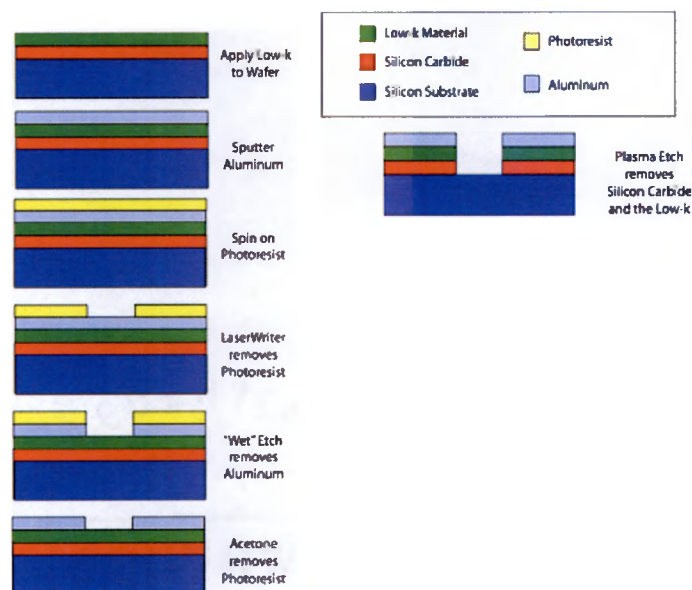


Figure 15. Diagram of process steps

PHOTOLITHOGRAPHY

Photolithography is a combination of several steps; spinning, baking, exposing, and developing. Each of the steps will be explained in some detail.

BAKE

Three different bakes are used in the process; a pre-bake, a soft bake and a post bake. Humidity is a factor for adhesion of the photoresist. The humidity needs to be between 20 to 60 percent for good adhesion, however the humidity is higher in Texas. To counter this a pre-bake is used; the substrate is placed on a hot plate for 60 seconds at 110 °C immediately before being placed in the spinner. The soft bake is used after the photoresist is spun on. To soft bake, the substrate is placed in a hotplate at 90 °C for 60 seconds. Soft baking dries the photoresist by evaporating the solvents. To further help with adhesion and to improve the wet etch profile of the Al mask a post bake is used. The substrate is placed on the hot plate for 120 seconds at 120 °C up to a few hours before wet etching.



Figure 16. Torrey Pines Scientific Hot Plate

SPIN



Figure 17. Laurell Technologies Corp. Spinner

Spinning is a process used to distribute the photo resist on the surface of a substrate to form a thin film with good uniformity over a range of thicknesses. A Laurell Technologies Corp. WS-400A-6NPP-LITE spinner was used to distribute a film of Shipley AZ 5214-E photoresist. The spinner can be programmed for multiple steps of different acceleration, time and revolutions per minute (rpm). There can be many programs saved, but program R was programmed for our use. Program R is a three-step program with the first step going to 500 rpms for five seconds, then a velocity of 3000 rpm is reached for twenty seconds, lastly it slows to 500 rpm/s for five seconds. This will result in a film of photoresist 1.63 μm thick. The basic procedure for this process step is to drop a few ml of resist on the wafer with a plastic pipette, spin for 30 seconds at 3000 RPM. Next the sample was soft baked for 1 min at 90 $^{\circ}\text{C}$.

LASER WRITER

All photolithography was done with a Lasiris Tec Laser. The laser has a thermoelectric cooling device (TEC) that maintains the diode at 19° C. The laser has wavelengths from 400-700 nm. From the laser, it goes through a system of optics and apertures to produce a small square beam to expose the resist. The APO SL 50X lens was used with an aperture of 40 X 40 was used to achieve a 5 μ m beam. A computer controls a stepper motor driven stage with an accuracy of 1/40th of a micron in the X and Y direction. An electromechanical shutter is controlled to turn the laser on and off in conjunction with moving the stage in order to “write” the programmed features. A script defines the stage acceleration velocity user units (a unit is 1/40th of a micron, so a user unit of 40x will set a distance of 1 equal to a micron) and 0,0 position. It then runs an auto focus procedure that uses a CCD camera connected to a PROSCAN auto focus controller. The procedure moves the stage to four points on the outer corners of the feature and uses the image contrast on your alignment beam to set the Z height of the lens. The focus controller then linearly interpolates Z during the writing procedure to keep the beam in focus. Once the focus procedure is complete, the script controls the stage and shutter to expose the desired features.

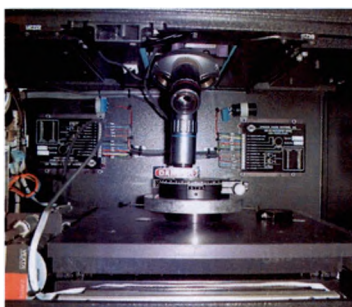


Figure 18. Inside view of laser writer system

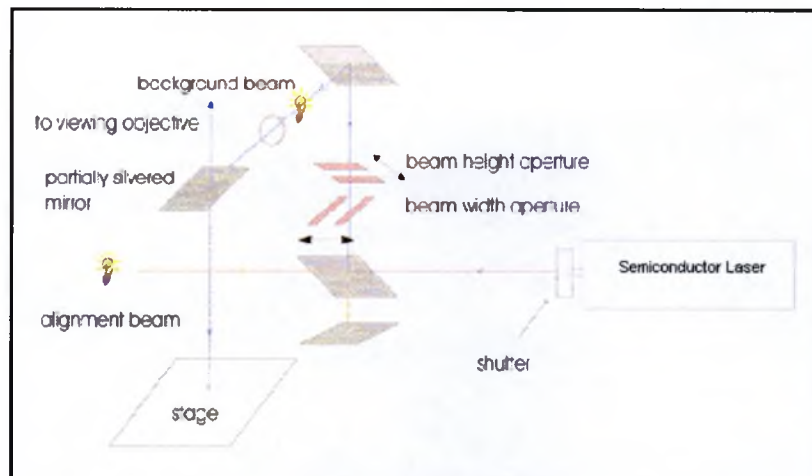


Figure 19 Schematic of Laser writer/Courtesy of Anita Acevedo

DEVELOP

After the samples are exposed, they are developed to remove the exposed photoresist and reveal the desired features. The developing procedure uses Shipley AZ 400K developer diluted 4:1 with DI water. The exposed samples were hung in a 50 ml graduated cylinder with 5ml developer and 20 ml DI water using locking forceps and agitated gently in the solution for 60 s. The sample is then inspected under a 5x microscope and if needed, can be developed for an additional 15 to 30 seconds.

WET ETCH

After the photoresist masks are made on top of the sputtered aluminum, the next step is to etch through the exposed aluminum to create the aluminum mask. This was done using Aluminum etchant Type A ($16 \text{ H}_3\text{PO}_4 : 1 \text{ HNO}_3 : 1 \text{ HAc} : 2 \text{ H}_2\text{O}$)³¹. This wet etch is isotropic in nature so it is important not to over etch the sample as under cutting of the mask will occur. Approximately 15 ml of etchant was placed in a 20 ml beaker and set on a

water filled Petri dish that is on the hotplate at 50 °C. Samples were set in the etchant and slowly agitated until the exposed areas turned dark gray. Aluminum etchant type A has an etch rate of up to 6600 Angstroms/min but this can reduce significantly as etchant gets dirty so detecting the endpoint visually is necessary. Once etching is finished the sample is immediately rinsed with DI water and blown dry with nitrogen gas. The Aluminum Etchant Type A is then properly disposed in the marked bottle. This process will be used again, after the dry etching step, to remove the aluminum mask.

STRIP

When the photoresist layer is no longer needed, a process called stripping removes the photoresist of the surface of the sample. The basic procedure to strip the mask is rinsing it in acetone. The acetone removes the photoresist at a rate of $> 39 \text{ k } \text{\AA}/\text{min}$. The sample is then rinsed in isopropanol and blown dry with nitrogen gas.

PLASMA ETCH

After the photoresist is removed, an aluminum mask remains on the surface of the wafer. The masked wafer is then etched in the magnetron with a plasma consisting of HFC 134a (1,1,1,2-tetrafluoroethane) and O_2 at 20 mTorr to remove the exposed low-k and SiC, SiCN, and TiN under layer. In this process, the primary mechanism for material removal is F-C and F-Si reactions between fluorine ions and the material atoms, followed by ion sputtering removal. This etch process should be anisotropic in nature due to the SiC providing carbon, enhancing polymer formation on the sidewalls of the feature. This polymer inhibits sidewall etching and therefore stops undercutting.

PROFILOMETER

The Tencor Alpha-Step 500 Profiler is a computerized, high-sensitivity surface profiler that measures roughness, waviness, and step height in a variety of applications. The profilometer auto-levels and auto-scales step heights up to 13 μm in the high-resolution mode (1 $\text{\AA}$ vertical resolution), or up to 300 μm in the low resolution mode (100 $\text{\AA}$ vertical resolution), with scan lengths up to 1 cm. The profilometer uses a stylus with a radius of 12.5 μm to profile the surface of the sample.



Figure 20. Tencor Alpha-Step 500 Profiler

CHAPTER 6

CONCLUSION

The plasma properties were studied over a variety of etching conditions. It was confirmed that the etching of these materials is ion induced, both by the relation between energetic ion flux, the etch rate. It is also confirmed by the resulting profile viewed with the profilometer, which clearly indicated trenching after etching at initial conditions. It can be stated that successful etching of these materials takes place, when the flux of energetic ions reaching the substrate surface is high, and there is a balance within the plasma chemistry. The etch mechanism of these materials requires sufficient fluorine and oxygen radicals for the etching of SiC, SiCN, and TiN to take place, without these collisions within the plasma causing a significant decrease in the ionization of the plasma gas.

The diffusion layer etch study involved a new method of reactive ion etching in a HFC 134a and oxygen plasma. It has been observed that fast anisotropic dry etching of silicon carbide, silicon carbonitride, and titanium nitride is successful in the magnetron using HFC-134a. Etch Rates as high as 92 Å/s for SiC, 33 Å/s for TiN and 77 Å/s for SiCN were observed while maintaining a uniform trench with a smooth bottom and with the power set at 50 watts.

HFC 134a is a viable alternative to more common industrially used etch gases. Etch rates are much higher compared to other fluorinated compounds used in previous studies (CF₄, CF₃, etc)[1]. Etch rates were highest at a relatively high oxygen content as

compared to single carbon gases which have higher etch rates at low oxygen content [1].

The extra carbon can be explained by the HFC 134a being removed by the C-O reaction allowing the SiC to be etched with the more efficient fluorine reactions. Si and Al selectivities were also seen to be in line with previously studied gases. The SiC had a 2:1 selectivity with Si and a 4:1 selectivity with Al.

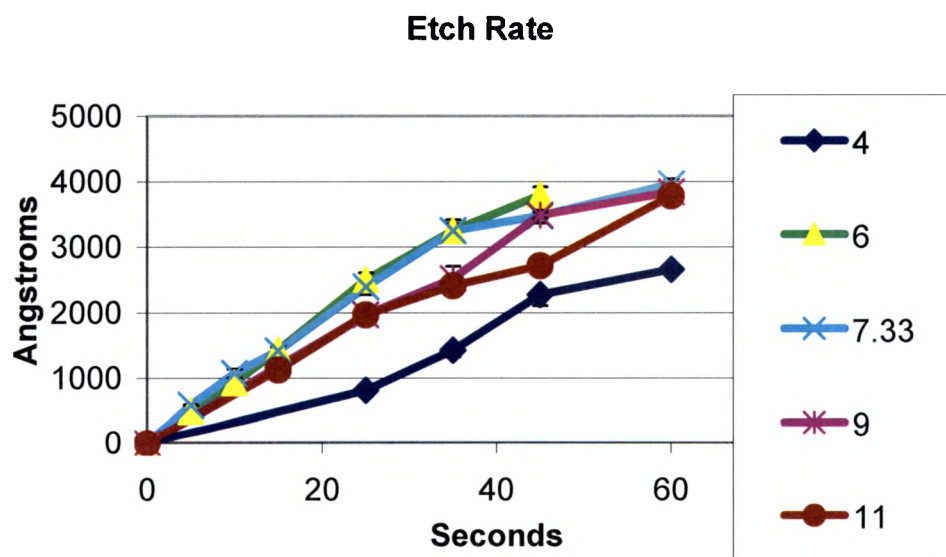
Further investigations involving the etching of these materials in HFC-134a/oxygen gas mixtures would be to etch at different pressure rates and see if that has an effect. Check more into what happens at the surface of TiN. It is known that from [2] that after the TiN sample has followed the oxygen plasma treatment of the CF_x contaminated TiN sample. It also seems apparent from our data graphs. To determine the etch characteristic selectivities, a further investigation into adding other gases, such as Argon at different flow rates should be conducted.

APPENDICES

(A) Raw Data SiC

Etch Depth (Angstroms)					
Oxygen Flow Rate (sccm)					
Time (s)	4	6	7	9	11
5		474	586		
		494	594		
		501	593		
		496	576		
avg		491	587		
10		881	982		
		966	1083		
		941	1052		
		914	1157		
avg		926	1069		
15		1398	1315		1212
		1414	1411		1209
		1499	1431		1069
		1398	1514		1031
avg		1427	1418		1130
25	899	2518	2260	1954	1919
	761	2584	2539	1967	1930
	847	2548	2436	1833	2019
	746	2347	2335	2101	1988
avg	813	2499	2393	1964	1964
35	1524	3359	3120	2335	2376
	1516	3120	3450	2400	2490
	1331	3256	3321	2550	2400
	1338	3278	3107	2778	2364
avg	1427	3253	3250	2516	2408
45	2216	3689	3313	3550	2515
	2090	3663	3501	3462	2774
	2290	3940	3553	3465	2736
	2500	3868	3530		2861
avg	2274	3790	3474	3492	2722
60	2663		3962	3735	3596
	2540		4000	3883	3735
	2693		4037	3923	3883
	2729		3850		3923
avg	2656		3962	3847	3784

Average SiC Etch Rate (/Second)					
Oxygen Flow (sccm)					
Seconds	4	6	7.33	9	11
25	33	100	96	79	79
35	41	93	93	72	69
45	51	84	77	78	60
60	44		66	64	63
avg	42	92	83	73	68

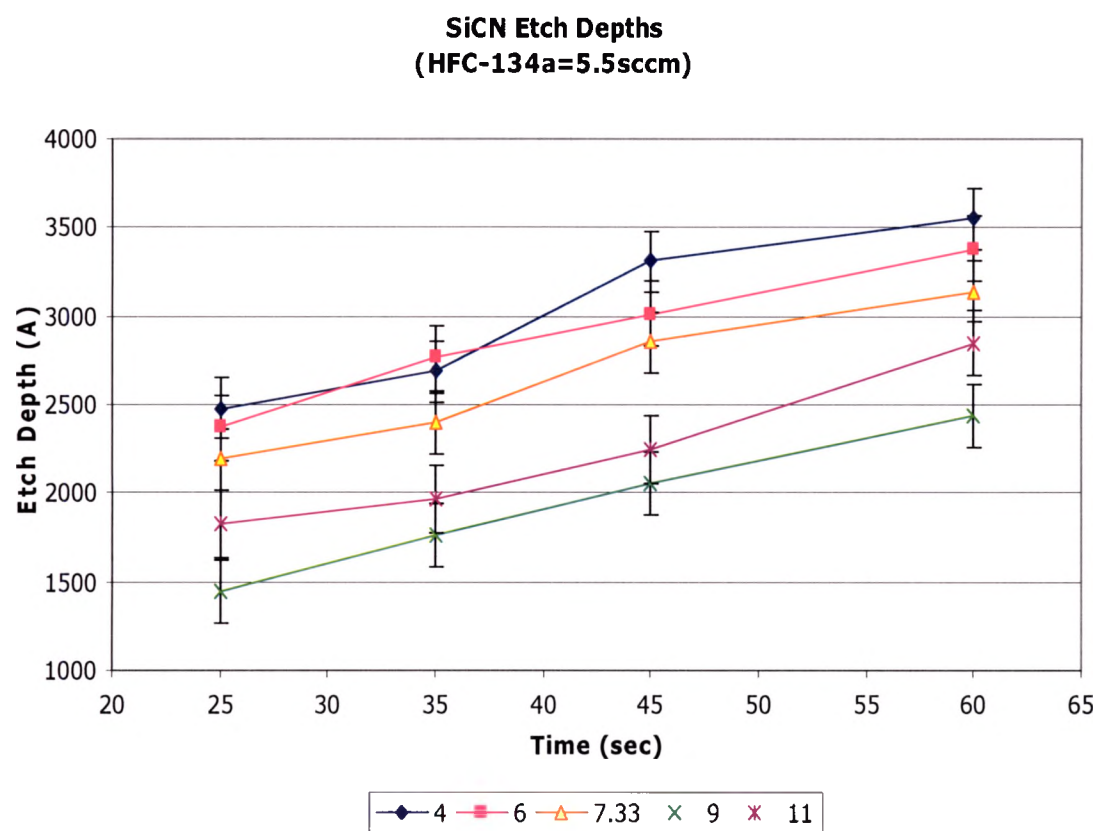


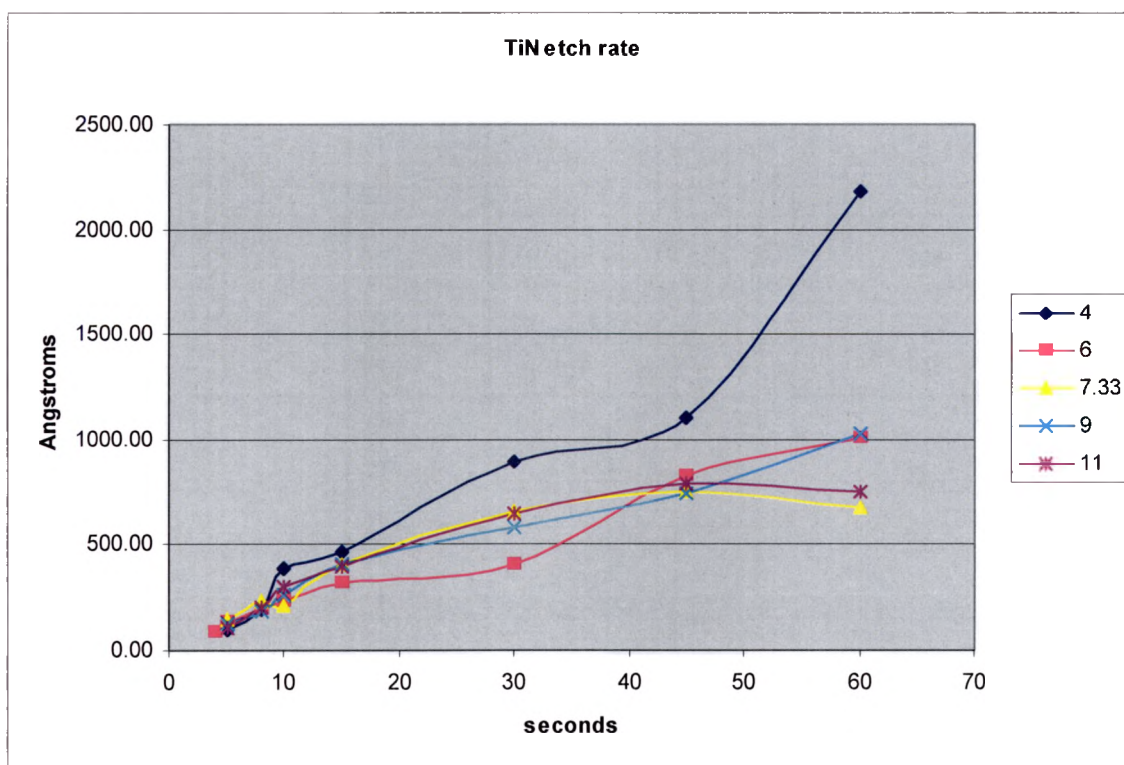
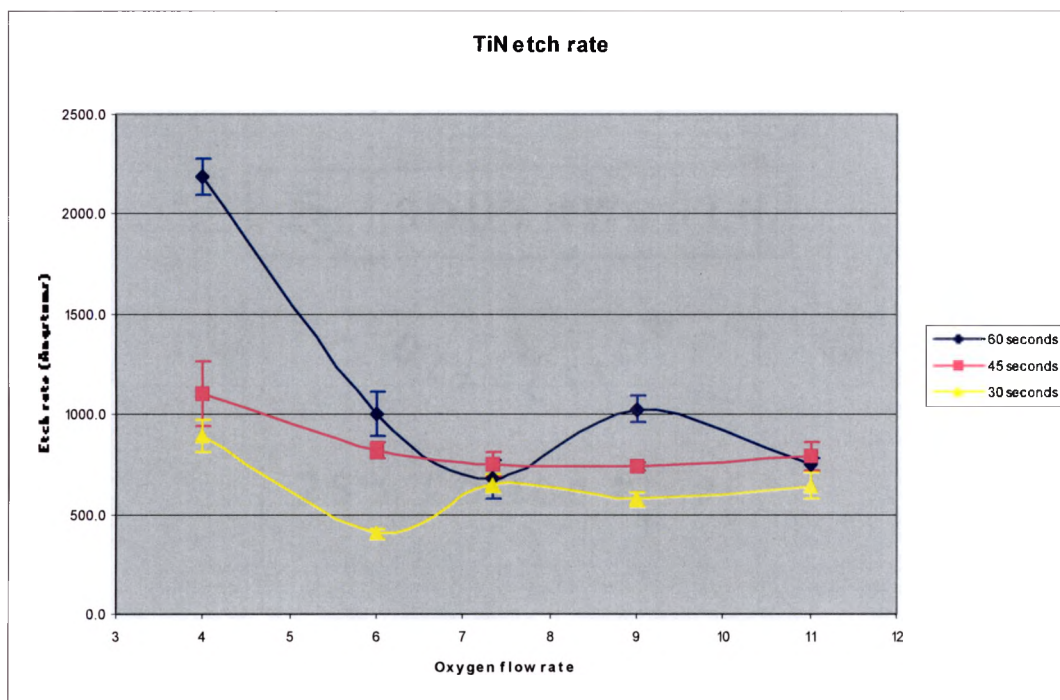
(B) Raw Data SiCN

Time (sec)		Oxygen Flow Rate (sccm)				
25		4	6	7.33	9	11
	1	2508	2278	2188	1437	1930
	2	2551	2257	2103	1417	2012
	3	2485	2600	2314	1684	1690
	4	2453	2242	2139	1340	1909
	5	2405	2458	2207	1362	1604
Mean		2480	2367	2190	1448	1829
SDOM		25	70	36	62	77
35						
	1	2700	2914	2212	1902	1806
	2	2794	2828	2455	1695	1982
	3	2452	2910	2606	1679	1925
	4	2623	2741	2226	1787	2044
	5	2856	2414	2461	1730	2091
Mean		2685	2761	2392	1759	1970
SDOM		71	92	76	40	50
45						
	1	3275	3067	2800	2047	2423
	2	3330	2964	2800	1964	2289
	3	3273	2907	2945	2000	2283
	4	3325	3057	2801	2265	2200
	5	3353	3073	2902	1978	2012
Mean		3311	3014	2850	2051	2241
SDOM		16	33	31	55	68
60						
	1	3669	3465	3190	2521	2888
	2	3554	3358	3271	2478	2703
	3	3370	3296	3050	2590	2968
	4	3754	3527	3125	2311	2715
	5	3425	3260	3057	2278	2971
Mean		3554	3381	3139	2436	2849
SDOM		72	50	42	61	59

Average Etch Rate

Time	4	6	7.33	9	11
25	99 22	94 68	87 61	57 92	73 16
35	76 71	78 90	68 34	50 25	56 27
45	73 58	66 97	63 32	45 57	49 81
60	59 24	56 35	52 31	40 59	47 48
Mean	77.19	74.22	67.90	48.58	56.68
SDOM	8 27	8 23	7 37	3 68	5 80



(C) Raw Data TiN

Time	4	6	7.33	9	11
60	2184 58	1005 06	673 63	1025 67	749 89
45	1100 08	820 50	744 33	738 08	787 42
30	891 00	401 75	649 75	574 58	640.19
15	457 89	315 00	400 00	401.00	395.00
10	385 08	232 25	214 58	255 08	294 25
8	190 08	193 25	232 25	180 92	201 42
5	93 26	134 00	139 75	124 50	107 88
4		86 11			
STDEV	726 11	336 01	250 61	327 99	272 64
Slope	32 28	19 75	30 83	18 81	31 18

(D) Develop Procedure

1. Mix 5ml AZ 400K developer and 20ml DI water in a 50ml graduated cylinder.
2. Using the blue locking forceps hold the sample in the solution and gently rotate it for 60s.
3. Rinse with DI water and blow dry with nitrogen.
4. Inspect with 10x microscope and develop for another 15 to 30s if needed.

(E) Spin Procedure

1. If humidity is high (greater than 60%), pre-bake($\sim 60^{\circ}\text{C}$) for about a minute.
2. Turn on vacuum pump under table.
3. Select spinner program R, and press start.
4. Place sample in center of chuck and push vacuum button.
5. Dispense a puddle of photoresist with plastic pipet on the center of sample and close lid.
6. Once program is finished open lid and press vacuum, release the sample.
7. Remove sample and soft bake on the hot plate at 90°C for 60s.

(F) Laser Writer Procedure

1. Switch on the computer, switch on the system power, turn on power strip, turn on the Proscan, and turn on the monitor.
2. Turn the switch on the back of the semiconductor laser on. The green LED will light up.
3. Choose the 50X objective.
4. Set the shutters both to 40.
5. Click on the laserwriter icon on the computer.
6. Press F5 and enter "laseroff.txt" at the command prompt to close the shutter.
7. Place the sample on the XY-stage under the aperture so the red alignment beam, which appears as a small red dot, is near the center of the sample.
8. Focus laser so that a square is seen on the screen using the Proscan knob.
9. Close all doors.
10. Press F5 and run the appropriate file.
11. After you are complete, close the program, and turn off all the equipment.
12. To move the stage: make sure the num lock is off and push F5 "crap" moves the stage fast or F5 "reset" moves it slow. Pushing the arrows on the number key pad make it travel farther/faster compared to the arrow keys. If you push up the stage moves back, left stage moves left, right stage moves to the right, and down stage moves towards the doors.
13. The laser will go out of focus, so the alignment will always have to be watched.

(G) Magnetron Procedure

1. To Pump the Magnetron out:
 - a. Remove any debris on the bell jar flange and gasket.
 - b. Bring the bell jar in contact with the flange on the well and hold it ensuring that the gasket is centered on the flange.
 - c. Press the button with the circle with a line in the middle of it in the lower left corner of the DCU (located in the center on the magnetron control panel).
 - d. Wait until you have reached the desired base pressure.
2. Turn valve on for appropriate gas.
3. Open the valve on the right of the chamber correlating to the Mass Flow Controller (MFC) connected to the sputtering gas you are using.
4. Turn on the Model 247C 4-channel read out on the rack.
 - a. Turn the display channel dial to the channel number for your MFC.
 - b. Set the right toggle switch for the MFC channel to flow.
 - c. Hold the left toggle switch up to SET PT. and adjust the display to the desired flow with the screw just next to the switch.
 - d. Turn the channel for your MFC on with the toggle switch on the 247, a green LED above the switch should illuminate.
5. Close the throttle valve with the rocker switch on the right on the magnetron panel, red light should illuminate. This closes the butterfly valve.

6. Move the shutter with the rotators on the side of the base plate to cover the magnetron gun being used.
7. Turn on the appropriate power supply for the gun you are using with the switch on the back and then the switch on the front.
8. Set the display in the power supply to set point power.
9. Set the regulation to power.
10. Turn the level knob to the desired power.
11. Press the start or output on button on the power supply (the gun should light).
12. Wait a few minutes to clean the target.
13. Rotate the substrate above the gun with the rotator.
14. Open the shutter using a stopwatch to time the deposition; then close the shutter when time is up.
15. When finished sputtering press stop or output off on the power supply.
16. Open the throttle valve with the rocker switch.
17. Turn gas off.
18. Open the chamber
 - a. Press the button with the circle with a line in the middle of it in the lower left corner of the DCU (located in the center on the magnetron control panel).
 - b. Open the valve on top nitrogen bottle, and the black valve after the regulator.

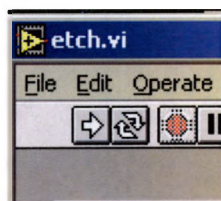
- c. When you hear the vent solenoid open and nitrogen begin to flow turn off the MFC channel on the 247C and close the MFC valve to the right of the chamber.
 - d. Turn off the 247C.
 - e. When the pressure is at ~760 Torr the chamber will open.
19. After samples are removed pump out the chamber.

(H) Lift Off Procedure

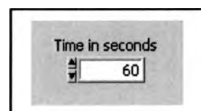
1. Sonicate the sample in acetone by holding it with the blue locking forceps with the aluminum side facing down.
2. When all the unwanted aluminum is loose lift the sample out of the bath while spraying loose the aluminum on the surface.
3. Rinse with isopropanol and blow dry.

(I) RF Power Supply Procedure

1. Turn on the power switch on the back of the RFX600A power supply.
2. Turn on the ATX-600 with the power button on the front.
3. Double click the etch.vi icon on the desktop of the pc.
4. Click on the arrow button in the upper left corner of the window.



5. Set the sputter time in seconds (default is 60s).



6. Press the RF enable button.
7. Set the power level (default is 50 watts).
8. To save bias data.
 - a. Press the save bias data button.
 - b. Enter a file name for the bias data.
9. Flow 5.5 sccm 134a and desired oxygen with the mass flow controller.
10. Close the throttle valve (pressure should reach ~ 20 mTorr).
11. Switch the RFX 600 power supply to standby.
12. Press the start button on the Labview.

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