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Research paper

Electron beam generated plasmas: Characteristics and etching of silicon nitride☆

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ABSTRACT

The Naval Research Laboratory (NRL) has developed a processing system based on an electron beam-generated plasma, where unlike conventional discharges produced by electric fields (DC, RF, microwave, etc.), ionization is driven by a high-energy (~keV) electron beam. The resulting plasmas are characterized by large electron densities (10^{10}–10^{11} cm^{-3}) and low electron temperatures (0.3–1.0 eV). Accordingly, a large flux of ions can be delivered to substrate surfaces with kinetic energies of only a few eV, a feature that can be attractive to processing applications that require low damage and atomic layer precision. This work describes the salient features of these plasmas produced in mixtures of argon and sulfur hexafluoride (SF_6) and their use in silicon nitride etching, with particular attention paid to developing processing parameters relevant to atomic layer processing.

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I. Introduction

As basic research continues to highlight the advantages of single nanometer scale materials systems and devices, there is a growing demand to develop industrially-scalable, atomic layer processing approaches that can bring them to market. Whether it be deposition, etching, chemical modification, or cleaning, the challenge is clear: the ability to rapidly process large areas with single monolayer precision. Plasmas have long been an important industrial tool given their ability to meet the demands of high throughput and precision. Plasma etching, in particular, has allowed the semiconductor industry to adhere to the forecast of Moore [1]. The unique attributes of non-equilibrium plasmas – the simultaneous delivery of reactive species and energetic ions to a material in a cold gas background [2] – provide a means to rapidly, but selectively and anisotropically, etch materials with high resolution over wafer-scale dimensions. However, there are issues associated with transitioning plasma etch to the atomic scale that must be overcome.

Ideal atomic layer etching (ALEt) schemes involve self-limiting processes that result in the removal of one and only one monolayer of target material, without damaging or altering the underlying monolayer and/or non-target materials. [3] In other words, the selective removal of one atomic layer of material at a time, which when repeated, can be used to remove a predetermined amount of target material only. While thermal ALEt approaches can achieve a desirable selectivity [4–6], the process is isotropic. Plasma-based approaches to ALEt are attractive given their ability to deliver a flux of energetic ions and thus add directionality to the etch profiles [7]. Such processes, however, require not only control over plasma chemistry to achieve the desired etch selectivity, but also tight control over the kinetic energy of ions incident to the processing surface in order to avoid damage while maintaining anisotropic etch profiles. [8–10].

The ion energy range is not well-defined and will undoubtedly be material specific. In general though, one should expect that the ion energy must be lower than in conventional etch processes to optimize the process and avoid material damage. Consider, for example, plasma-enhanced ALEt of silicon consisting of a repeated cycles of plasma chlorination followed by argon plasma exposure. [8,11] In this scheme, a Si substrate is exposed to a chlorine containing plasma to form a monolayer of $SiCl_x$ which is then etched away during the argon plasma step via energetic Ar^+ ions. The kinetic energy considerations during this two-step process are the threshold energy to remove Si with and without a Cl monolayer. With chlorine present Shin et al. [12] and Chang et al. [13] determine the threshold energy to be ≈16 eV for Ar^+ ions, while Chang and Sawin [14] and Vitale et al. [15] find the threshold drops to ≈10 eV for Cl^+ and/or Cl_2^+ ions. Agarwal and Kushner [8] also note that roughness scales with ion energy due to etching during the passivation step. For bare Si, Oehrlein et al. [16] suggest a physical sputter

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threshold of about 20 eV for Ar^+ ions. On the other hand, Graves and Humbird [2] find that Ar^+ ion kinetic energy should be lower than about 12 eV to preserve the crystallinity of the Si. While this is a simplified description of the processes, the point is that low ion energies are needed to optimize the passivation step (i.e. minimize etching) and not damage the Si (i.e. change the crystallinity) during the etch step. In this regard, high-density plasmas with a low electron temperature (T_e) are an attractive approach to ALEt because the ion kinetic energy at surfaces scales with T_e.

Electron beam generated plasmas are formed by injecting a high-energy electron beam into a gas background to drive plasma production. The resulting plasmas are characterized by high plasma densities (10^{10}–10^{11} cm^{-3}) and low electron temperatures ($T_e < 1$ eV), [17] and thus, can deliver a large fluence of reactive ion and neutral species where the kinetic energy of the ions is as low as a few eV. As such, they are well-suited for low damage applications and atomic layer processing. It is interesting to note that electron beam produced plasmas were used in some of the earliest investigations of plasma-based atomic layer etch processes. [18,19] Recently, they have been used to chemically functionalize graphene – a single layer of carbon – without erosion over a large range of operating conditions [20–22]. The successful treatment of graphene is due, in large part, to the low ion energies. Previously, we have vetted their use in low-damage etching applications [23] where we showed we could remove a film of Si_xN_y over graphene with little damage to the graphene even when the optimal etch time was exceeded. This work employed electron beam generated plasmas produced in Ar/SF_6 and forms the basis of the present manuscript. Specifically, we describe the results of the Si_xN_y etch studies used to develop the etch process to successfully removal Si_xN_y from graphene.

This paper is organized as follows: First, we describe the electron beam generated plasma processing system developed at the Naval Research Laboratory (NRL) and highlight the salient features when applied to SiN etching using a combination of diagnostics and simulations. Then, we present the results from SiN etch studies that were used in Ref [23] and relate those results to the plasma characterizations. While there is no explicit attempt to achieve ALEt conditions, it provides etch rates for Si_xN_y when subjected to reactive gas plasmas with ion kinetic energies ranging from a few eV up to about 50 eV, a range amenable to ALEt.

II. Processing system and experimental approach

A. Plasma processing system

The large area plasma processing system (LAPPS) is NRL-developed technology that employs magnetically-collimated, sheet-like electron beams to generate similarly sized plasma sheets for use in materials processing [24,25]. A typical embodiment of the system is shown in Fig. 1. The base processing system is relatively simple, consisting of an electron beam source, entrance aperture, termination anode, sample holder, and magnetic field coils. While these components are considered critical, there is significant flexibility in their design and operation. Typically, 1–3 keV electron beams with current densities of 1–5 mA/cm^2 are used. Co-axial magnetic fields of 100–300 G are used to collimate the electron beam and thus improve uniformity along its length [26]. These parameters are sufficient to produce uniform plasma sheets compatible with typical wafer-scale systems (diameter ≤ 300 mm) operating at low pressures (< 100 mTorr).

For the etch results described here, the plasma processing system and its operation in SF_6 gas backgrounds has been previously described in detail. [27,28] Briefly, the system vacuum was maintained by a 250 L/s turbo pump, with a base pressure ~10^{-6} Torr. The operating pressure was achieved by introducing Ar (purity >99.9999%) and SF_6 (purity >99.999%) through mass flow controllers and throttling the pumping speed using a manual gate valve. The electron beam was produced by applying a − 2 kV pulse to a linear hollow cathode for a selected pulse width (2–4 ms) and duty factor (10–20%). The emergent beam passed through a slot in a grounded anode, traversed the gas, and was then terminated at a second grounded anode located further downstream. The electron beam volume between the two anodes defines the ionization source volume, with the dimensions set by the slot size (1×25 cm^2) and the anode-to-anode length (40 cm). A magnetic field of 150 G was produced by a set of external coils. The samples were placed on a 10.2 cm diameter stage located 2.5 cm from the nominal edge of the electron beam. The stage was either ground or powered with RF (13.56 MHz) to achieve a self-bias of up to − 40 V. The stage was held at room temperature.

The samples used in this work were 30 nm thick Si_xN_y films grown on Si by plasma enhanced chemical vapor deposition at a temperature of 400 °C. While the growth substrate in this work is Si, the growth method was specifically developed for deposition on graphene to provide a negligible impact on graphene properties [29]. The operating parameters were explored and varied according to Table 1. Etch rates were determined by measuring the film thickness before and after processing using a VB-400 VASE variable angle spectroscopic ellipsometer (J. A. Woollam Co., Inc). Three different angles of incidence (55°, 60°, and 65°) and wavelengths ranging from 190 to 2250 nm were used to measure the real and imaginary parts of the complex reflectance ratio. The film thickness was determined using the following procedure. For SiO_2/Si, a two step Cauchy fit was applied. For the amorphous Si a point by point fit for amorphous polysilicon from Woollam library was used. To evaluate the thickness of the Si_xN_y layer, the following protocol was followed. First, the thickness of the native SiO_2 layer on clean Si wafer was measured and set to 2.4 nm, which is reasonable for native oxide layer. Then, a multilayer Cauchy model was built, in which the SiO_2 layer thickness was fixed and the thickness of Si_xN_y was varied until the fits match the experimental data. The standard deviation in all cases was <5%.

We report two etch rates for each parameter study in this work. The first (etch rate) is determined by the time the beam is on and the second (time-averaged etch rate) is determined by the total process time. That

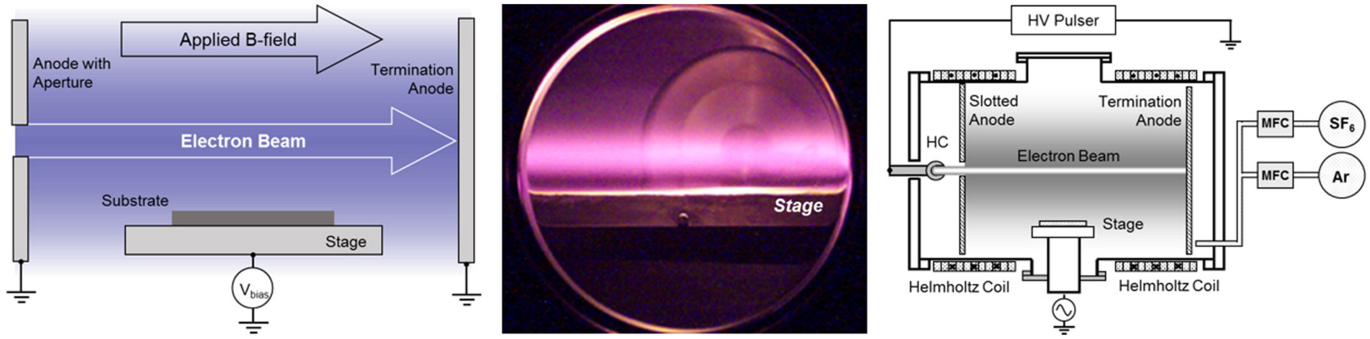

Fig. 1. The US Naval Research Laboratory's Large Area Plasma Processing System (LAPPS), which employs an electron beam generated plasma. (Left) An illustration of the system, (Middle) an image of the plasma through a 6″ port, and (Right) a schematic diagram of the processing reactor used in this work.

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