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To cite this article: Per Morgen et al 2007 J. Phys.: Conf. Ser. 86 012019

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Plasma assisted growth of ultrathin nitrides on Si surfaces under ultrahigh vacuum conditions

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Abstract.
Plasma assisted nitridation of Si is a very useful and easily controlled process for the direct in-situ deposition of pure silicon-nitride films on Si under ultrahigh vacuum conditions. The plasma used in the present work is formed by microwave dissociation of nitrogen gas. Under these circumstances an additional activation barrier to the formation of nitride exists which can be overcome by isothermal processing at temperatures from 300°C and up. Room temperature growth of ultrathin silicon-nitride films on top of different substrates is also found to be possible through the use of dispersed atomic Si layers which react with the microwave excited nitrogen plasma at room temperature. Studies are undertaken with the Si(111) and Si(100) surfaces and the results for these two surfaces are compared. The conditions for the growth of amorphous versus microcrystalline films are established with a critical temperature around 500-600°C.

1. Introduction
This work was carried out in different laboratories, using identical sample handling and plasma exposure conditions. The microwave cavity excitation region is separated from the ultrahigh vacuum chamber by a long capillary, which directs the flow of gas to the sample surface. Similar plasma sources are installed in all three ultrahigh vacuum facilities available for this project. For one of these sources a mass spectrometer mounted in line-of-sight of the capillary showed about 50 per cent atomic nitrogen in the beam, compared with the same inlet pressure of unexcited nitrogen.

Most of the results reported and discussed here are obtained at the Aarhus electron storage ring ASTRID, where we have access to a chamber with a SCIENTA 200 analyzer for surface studies with photoemission at high resolution and intensity. In our laboratories in Odense we have a SPECS surface analysis system, with facilities for XPS (X-ray induced photoemission) with Al- and Mg Kα radiation, Auger electron spectroscopy with a focused electron beam, and LEED (low energy electron diffraction) as complementary surface analysis techniques to

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the synchrotron radiation facility. Other techniques, available through the partner in Aalborg, include optical second harmonic generation spectroscopy and spectroscopic ellipsometry.

Synchrotron radiation permits high resolution photoelectron spectroscopy, sensitive to changes in chemical environments, such as it happens when Si atoms get bonded to different numbers of other atoms during nitridation and oxidation. With the present monochromator, the region with the Si 2p signal, and the Si valence band (and Si-nitride and -oxide valence bands) may be recorded, along with the N 1s signal.

Particularly for studies of the composition of the valence bands the variation of the photon energy is very important, as the photoelectric cross sections for the different atomic orbitals vary differently in the region of 30 to 130 eV, which is the region used for these studies.

For the Si 2p core levels (of around 100 eV binding energy), the typical photon energy is chosen to be 130 eV, which gives a high throughput and kinetic energies of the photoelectrons of the order of 30 eV, which assures the highest possible surface sensitivity (lowest mean free path for electron transport to the surface and into the vacuum).

The relation between relevant energies for photoemission is: $E_K = E_P - E_B$, where $E_K$ is the kinetic energy of the emitted electrons ("photoelectrons"), $E_P$ the photon energy, and $E_B$ the binding energy of the atomic electrons before the photon impact.

The presence of other elements like C and O at or near the surface may be monitored with XPS, which is also available at the ASTRID beam line from a normal two anode X-ray source like the one we use in Odense.

The Si samples are cut from polished, $n$-type 5 Ohm-cm $P$-doped (111)- and (100) wafers. They are $3\times1$ cm$^2$ rectangularly shaped and mounted with Ta-clamps on a rotatable holder. After cutting they are cleaned in ethanol in an ultrasonic bath, and further cleaning is done by passing a current through them inside the vacuum chamber after bakeout which heats them up to above 1000$^\circ$C. The cleanliness of the surface is assured with photoemission, scanning the beam spot across the sample, and with LEED, also scanned over the surface of the entire sample.

Typically the Si(100) surface is the most studied surface in the technical literature, as this surface is historically the preferred surface for device applications, due to its minimal defect concentrations at the oxide/Si interface after normal industrial oxidation procedures. The Si(111) surface has fewer practical applications, but has been the focus of many years of basic studies to understand surface reconstructions at the atomic level.

The clean surfaces are reconstructed, the Si(111) in the 7×7 structure [1] and the Si(100) in the 2×1 structure (also sometimes named the 4×2 structure accounting for alternating dimer tilts). These reconstructions are by now well understood and the atomic positions and surface unit cells are characterized. The different surface atomic coordinations are measurable as chemical shifts of the different Si atom positions in the surface unit cells. The specific reactivities of the dangling bonds and back bonds on the Si(111)- and the dimers on the Si(100) surfaces are also known through work by many groups, including ourselves [2].

Various modifications of the procedures described herein will be useful for self limiting reaction cycles on surfaces and for the local, patterned deposition of nitride films. Thus direct in-situ growth of nitride films with atomic layer control and with lateral resolution of structures on the nanometer scale becomes possible.

As an example a sandwich structure has been fabricated in this project, consisting of a 7-8 A oxide layer on top of silicon with a 4-5 A thick amorphous nitride layer deposited over the oxide at room temperature (RT), with no intermixing thus preserving the benign properties of the oxide/Si interface and the diffusion limiting properties of the nitride layer for B from the polysilicon gate electrode used in a typical CMOS structure.

These structures with ultrathin silicon nitride films could be candidates for future CMOS devices or other microelectronic components, and their properties will be briefly discussed and evaluated with such applications in mind. However, generally nitride materials including metal
Figure 1. Si 2p spectra at 130 eV photon energy of the Si(111)\textit{7} \times \textit{7} surface. The effect of the first exposure of N (5 min at 2 \times 10^{-7} Torr total nitrogen exposure) is shown, at 300 °C.

Figure 2. Si 2p spectra at 131 eV photon energy of the Si(100)-\textit{2} \times \textit{1} surface. The effect of the first exposure of N (10 min at 5 \times 10^{-7} Torr total exposure) is shown, at 500 °C.

Nitrides are very valuable ceramics and have many uses in the form of thin surface coatings.
Their fabrication has often been difficult in the past, but we propose that nitride coatings on unobstructed surfaces may be formed rather easily in processes similar to those explored here for the direct *in-situ* nitridation of Si surfaces.

2. **Experimental details and results**

2.1. **Core level studies**

The initial steps of nitridation with surfaces kept at relatively low temperatures are shown in Figs. 1 and 2. The very first sign of N-atom chemisorption on a Si(111) surface ever recorded is shown in Fig. 1, where the effect of N-incorporation in the surface is seen to result in loss of intensity at the right foot of the main (bulk) Si 2p peak, indicating the removal of some of the reconstruction of the surface. On the clean surface this foot is related to the presence of so-called rest-atoms [1] of the 7×7 reconstruction.

On the Si(100) surface (Fig. 2) a somewhat higher exposure affects the Si 2p signal. Thus the peak due to upwards pointing dimers [3], at the right foot of the Si peak of the clean surface, is removed, and the intensity ratio between the two spin-orbit components of the main peak are changed. At the same time, the intensity in a region of higher binding energy (lower kinetic energy) next to the main peak seems to increase with a continuous distribution of intensity.

In Fig. 3 further exposure of Si(111) to the nitrogen plasma is followed in the Si 2p spectrum. The efficiency of nitridation is seen to change rapidly with temperature in going from 400°C to 500°C. Now, at 500°C a clear structure is emerging at the high binding energy side of the main peak. This new structure is due to chemically shifted Si 2p levels in the Si atoms in contact with nitrogen atoms, and in different coordination geometries [4]. The total nitride layer thickness may be estimated from the relative intensities of the shifted peaks and the main peak, from calculated attenuation lengths of electrons through Si3N4 with these kinetic energies. Alternatively ellipsometry measurements may be used, if available *in-situ*.

Similarly to Si(111), the Si(100) surface is rather efficiently nitrided at 500°C, as seen in Fig. 4.

We next grew thicker nitride films by going to higher temperatures of the Si substrates (Figs. 5, 6, and 7). By comparing these results systematically, it appears that the nitridation of Si is self limiting, and that the final thickness of nitride obtained is dependant on the temperature. The thickest films are around 30 Å thick.

2.2. **Discussion of details extracted from the Si 2p spectra**

To extract more information from these measurements each spectrum has been analyzed and deconvoluted with curve-fitting procedures. Using the program FitXPS [5] the spectra obtained after every step have been decomposed in pairs of spin-orbit split Gaussian-Lorentzian shaped components. In this way the Si 2p spectra were seen to consist of the bulk and the four nitride components similarly to the spectra obtained during oxidation. We label these four components of Si with their effective coordination numbers, n (n=+1,+2,+3, and +4).

One example of the fitting is shown in Fig. 8. The fitted spectrum ("best fit") completely agrees with the experimental data within the noise. This fit is one of a number of fits done systematically with the data from all experiments at closely similar conditions of count rates (noise) and resolution. The only physical parameters varying are the exposure conditions. The fits are started from fixed widths and energies for a given peak (n) in all spectra and only relative intensities of that peak are allowed to vary significantly. However, for the final agreement also some small relative changes of the energy differences between the bulk Si (n=0) and the shifted peaks are allowed based on the understanding of the effect of a varying field in the nitride which may shift and broaden the +3 and +4 peaks originating at the top of thicker nitride layers.

For fitting the bulk- and nitride-coordinated Si 2p peaks the following values have been used: Lorentz-full-width-at-half-maximum (LFWHM) = 0.18 eV (Si (0)) and 0.3 eV (Si(+n), n=1, 2,
Figure 3. Si 2p spectra of Si(111) during gradually increasing nitridation (Top: 400°C and bottom: 500°C).

3 and 4), Gaussian-full-width-at-half-maximum = 0.20 eV (Si (0)) and 0.50 eV (Si(+n), n=1,
Figure 4. Si 2p spectra of Si(100) during gradually increasing nitridation at 500°C.

Figure 5. Si 2p spectra of Si(100) after exposure to 900 - 3150 L nitrogen (total) at 700°C.

2, 3 and 4), and the Si 2p spin-orbit splitting is equal to 0.61 eV.

These and previous fits are obtained essentially with only the relative intensities as "free" parameters, and are treated systematically through a large set of data under similar experimental conditions.

The systematic changes of the intensities of the resolved components are plotted in Figs. 9
Figure 6. Si 2p spectra of Si(111) during gradually increasing nitridation at 800°C.

Figure 7. Si 2p spectra of Si(100) after exposure to 600 - 3600 L nitrogen (total) at 900°C.

(for nitridation at 500°C) and 10 (nitridation at 900°C). These results are obtained with the Si(100) surface.

The systematics of the change of intensities of the components differ as a function of the temperature. The changes in intensities with time are all nearly linear with the dose of nitrogen
Figure 8. Core-level photoelectron spectra of Si(100)-2 \times 1 exposed to nitrogen during 105 min at 700°C.

at 900°C, while at 500°C changes occur faster for similar exposures. The nitride coverage is judged to be uniform in the latter case.

The Si +1 and +2 components are thus attenuated during growth at all temperatures, but slower than the bulk Si signals, which indicates that their origin is nearest to the interface. The growth rates of the Si +3 and +4 peaks are parallel at 900°C but somewhat decoupled at 500°C. One way of explaining this could be that there is no Si +3 coordination at the interface at 900°C. At 500°C there is some Si +3 coordination at the interface.

We believe that the generally slower growth and decay rates at the higher temperature has to do with different structural properties obtained at the two different temperatures. Thus for the Si(111) surface, through the discussion of the interface structures and other details [6] of the spectra, it was inferred that for temperatures up to around 500°C the structure of the nitride film is amorphous while it is microcrystalline for higher temperatures. Thus the linear characteristics of the intensity variations for all Si 2p peaks during growth at 900°C could be due to a similar kind of crystalline growth as seen for the Si(111) surface with columnar (open) structures [6].

Occasional observations of a shift to higher BE for the Si +4 component during growth may be explained by an electric field across the nitride from charges near the interface varying from case to case. This field,

$$ E = -\frac{\partial V}{\partial x} $$

produces the largest changes in binding energy farthest from the interface, i.e. in the region of the largest number of Si +4 coordinated Si atoms.

The films grown here have thicknesses from below 10 Å to about 30 Å as judged from the relative intensities of the features in the Si 2p spectra.
2.3. Valence band studies
The valence band of clean Si, and the changes of its structure upon formation of nitride on top, is studied at two photon energies, 40.8 eV and 131 eV.
**Figure 11.** Experimental and deconvoluted 131 eV valence band spectrum of clean Si(100) 2×1.

**Figure 12.** Photoemission from the valence band of a clean Si(100) 2×1 surface with 40.8 eV photons. Besides the three broad peaks mentioned in the text, three narrow peaks are identified (S1-S3).

The clean Si(100) 2×1 surface is known to have dimers at the surface. The peaks in the valence band of clean Si in the fitted spectra (see later) Fig. 11 and Fig. 12 are labeled S (surface states) and B (bulk states). One can actually see three sharply defined states S1, S2 and S3 (Fig. 12). S1 corresponds to a dangling-bond surface state with a sizeable orbital
Figure 13. Exponential fits (solid lines) of the normalized intensities of the three surface states of the Si (100) 2×1 during nitrogen adsorption at 900°C.

The surface states of the Si(100) 2×1 surface during nitrogen adsorption at $E_{\text{photon}} = 131$ eV

occupation along the surface normal, probably of $(s, p_z)$-like character. It is localized on the "upper-" Si-atoms of the tilted dimers. S2 with its higher intensity is ascribed to third layer Si-atoms, while the S3 feature disperses with energy (not shown) and is therefore believed to contain both "down-" dimer Si-atom dangling bonds and some bulk intensity [7]. The surface states are overlapping the bulk valence bands and are thus completely occupied in agreement with what is expected for tilted dimers. These surface features disappear with the initiation of nitridation as shown in Fig. 13.

The features in the valence band of bulk Si$_3$N$_4$ have previously been assigned [8, 9, 10] to N 2p lone pair orbitals bonding with Si 3d and 3p orbitals, nearest the VBM (valence band maximum, 4 eV below the Fermi level), N 2p lone pair orbitals bonding with Si 3p orbitals below these (8 eV below the Fermi level), and Si 3s states (13 eV below). We observe the upper features in the present spectra at both photon energies, but there is also clear evidence for additional intensity above the expected position of the VBM, like found earlier by Kim et al. [11].

Comparing Figs. 15 and 18, it is obvious that two main features (shoulder and peak) vary in the same way with growing thickness of nitride at the 500°C. Thus they must contain nearly equal proportions of Si- and N-atomic orbitals. This equal mixture is also found in the above mentioned calculations of the composition of the density of states of bulk Si$_3$N$_4$. Thus if the orbital compositions of the peaks were hypothetically very different their intensities should have varied at different rates at the two photon energies.

Figures 14 through 18 show what happens in the valence band spectra when adsorbing nitrogen on heated Si(111) and Si(100) surfaces [12]. For the Si(111) surface we used 50 to 240 min. exposures. There are only minimal changes in intensity and structure in this case, and the nitride growth is clearly seen to saturate on this surface. The first and last spectra are smoothed, while the other spectra are displayed as recorded. The count rate was relatively low
through these experiments. However, we do find definitive similarities with a 134 eV spectrum of a thin nitride, grown with NH$_3$ at high temperature on the Si(111) surface [11]. This is in the form of the two small peaks above the VBM. The measurements on the Si(100) surface were carried out in more detail and for various temperatures, as shown in Figs. 15 - 17, at 131
Figure 16. 131 eV valence band spectra of Si (100) 2×1 during gradually increasing nitridation at 700°C.

Figure 17. 131 eV valence band spectra of Si (100) 2×1 during gradually increasing nitridation at 900°C.

eV photon energy, and at 40.8 eV photon energy (Fig. 18). The structures emerging for the nitridation of the Si(100) surface are here relatively sharply defined peaks, contrary to the case for Si(111).
Figure 18. 40.8 eV valence band spectra of Si(100) 2×1 during gradually increasing nitridation at 500°C.

Figure 19. Deconvoluted 131 eV valence band spectrum of Si(100) 2×1 exposed to nitrogen during 70 min at 500°C.

2.4. Discussion of valence band studies
The common structure of the valence band across the measurements is seen as essentially two broadened structures. As discussed above the origin of these structures is peaks in the density...
Table 1. Peaks in VB: Notation, center energy, and composition

| Peak | $E_k$ (eV) | State |
|------|------------|-------|
| A    | 129.0      | Si 3p + N 2p (?)(interface) |
| B    | 127.1      | Si 3d + N 2p |
| C    | 123.5      | Si 3p + N 2p |
| D    | 118.6      | Si 3s |

of states of the bulk of the film, and of the clean Si sample. Major features of the spectra should change their weights depending on the growth mode of the nitride film. Thus Si surface states should disappear with initial exposures to N, and the bulk Si states gradually (exponentially) with the coverage of a uniformly growing film on top. All of the spectra of the nitrided samples are decomposed into four Gaussian peaks as shown in Figs. 11 and 19, labeled A, B, C, D in Fig. 19, with central kinetic energies of 129, 127.1, 123.5, and 118.6 eV, respectively.

Besides structures with origin in the band structure and density of states of bulk Si$_3$N$_4$, well below the Fermi level, all spectra contain intensity nearer the Fermi level (0-4 eV). The clean Si surface spectrum reveals surface states in the upper region near the Fermi level, which disappear with N-exposure. The other peaks in this region varying in intensity with growing nitride could therefore more likely be identified as interface electronic states, or as surface states of the nitride film [11]. From their systematic behavior with coverage of nitride, as discussed in more detail later, we find support for an assignment of at least part of the intensity as due to interface states for Si(100), and assume that the same is true for Si(111).

The clean 131 eV Si (100) spectrum (Fig. 11) shows the same surface related features, S1, S2, and S3 as the spectrum at 40.8 eV, but the widths of peaks S2 and S3 are larger than the width of S1, which could indicate additional bulk intensities in S2 and S3.

Table 1 gives information about the partitioning of the 131 eV spectra and the corresponding spectroscopic composition of features labeled A, B, C, D. Feature A is tentatively assigned as a combination of Si- and N orbitals, due to its similar behavior with photon energy as peak B.

The growth of nitride at 500°C is shown in Fig. 20 with the intensity of peaks A, B, C, and D increasing linearly with nitridation time. At 900°C (Fig. 21) peak A clearly decreases with increasing exposure time, while B, C and D increase with nitrogen exposure like at 500°C.

Thus the main difference between these two experiments is the variation of peak A. We suggest that this peak represents the interface states. The different behavior at the two temperatures is due to the amorphous nature of the nitride film growing at 500°C versus the crystalline film growing at 900°C. Thus the growth is slower at 500°C and the number of interface states is lower and grows slowly. At 900°C the interface states are formed very early during the reaction, as on Si(111), and the density is stable during further growth. The decay seen in this experiment is thus not due to a lowered concentration but caused by the covering film. The results at 700°C are similar to the 900°C case.

The region of the Si(100) spectra around peak C is showing much more changes in structure and intensity than the similar Si(111) region (Fig. 14). In this region we expect contributions to the intensity from N 2p states bonding with Si 3p states in the bulk of the nitride. Thus these different changes indicate a different bulk crystalline structure of the ultrathin nitride on the two surfaces.

It is clear, that if the states identified here as interface states are electrically active in devices in these concentrations, they would cause severe difficulties for the use of these nitride layers as alternative gate dielectrics, which has also been the prevailing opinion in the literature for a long time [13].
Figure 20. The variation in intensities of deconvoluted valence band peaks of Si (100) 2×1 at 500°C.

Figure 21. The variation in intensities of deconvoluted valence band peaks of Si (100) 2×1 at 900°C.

For the Si(111) surface the growing nitride only shows small changes in valence band structure for thin and thicker films. The structure always extends up to near the Fermi level, and shows one peak in this region but with less intensity than the peak discussed in [11].

2.5. Growth of stacked structures
In the other set of experiments (Figs. 22 and 23), a 7 Å bottom oxide layer was initially grown by a self limiting isothermal oxidation process at 500°C for 90 min, and a thin Si-nitride film was then deposited in a series of cycles of deposition of slightly below 1 ML (monolayer) of Si atoms from a heated Knudsen source on top of the oxide with the sample held at room temperature (RT), followed by exposing this system to atomic N, still at RT. In total 3 ML of Si and 200 min of N exposure were used, equivalent to about 4-5 Å of nitride formed.
Figure 22. $Si\text{ 2p spectra of SiO}_2$ on Si(100) while gradually depositing Si-nitride at room temperature.

Figure 23. Valence band spectra of SiO$_2$ on Si(100) while depositing Si-nitride at room temperature.

We judge that the changes in both the Si 2p and the valence band spectra indicate that this thin deposited amorphous nitride layer stays on top of the oxide, while preserving the Si / SiO$_2$ interface integrity.

A closer analysis of the Si 2p spectrum is possible by using the same numerical deconvolution procedure as above (FitXPS [5]). We are here able to separate the Si$^{+4}$ components from the
nitride and oxide layers (Fig. 24) differing from the case of other systems studied, such as mixed (oxynitride) systems [4] with four mixed Si-O-N peaks.

3. Summary and conclusions
In this work we have explored the reactivity of atomic nitrogen created by microwave excitation of nitrogen gas to grow ultrathin nitride layers on Si surfaces, and to follow the steps in much detail through the use of photoemission spectroscopy, and especially with synchrotron radiation. The activation of the nitridation mechanism takes place above 400°C but room temperature growth of Si-nitride may be undertaken at room temperature through an intermediate deposition of atomic layers of Si, which are then allowed to react with the atomic nitrogen created in the plasma source. This reaction does not touch other components, and is therefore self-controlled. This opens a nice perspective for the direct application of Si-nitride structures, through lithographic patterning, and for the control of the thickness.

To judge any potential of these nitride films on Si for device purposes, the fabrication of prototype components for electrical testing is crucial. Eventually the use of ultrahigh vacuum facilities as part of a chip fabrication procedure could become mandatory, which is not yet a popular idea with the big producers of chips. Also, the present finding of relatively large concentrations of defect states above the valence band maximum of Si-nitride, could lower the interest for trying these films for application in CMOS devices, as the conduction of holes, through such gate nitride dielectric defects as seen here, to the gate electrode might be detrimental.
Other important uses of ultrathin dielectrics on Si are foreseen, however, in the form of diffusion barriers which might prevent metal ions from other types of metal-oxide or -nitride dielectrics to penetrate into the Si channel of proposed non-Si-oxide based CMOS structures for the next generations of Si-based computer chips.

Finally, further, and more detailed, studies along the lines presented in this paper could possibly lead us to understand the dynamics of the nitridation reaction, including its self limiting character.

Acknowledgments
This work was supported by the Danish Research Council for Nature and Universe, and by the Carlsberg Foundation. The authors are grateful for the access to ASTRID, and for the support and excellent working conditions offered by the staff there. Ali Bahari was supported by the government of Iran, and by the University of Southern Denmark.

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