

DIELECTRIC GAPFILL OF HIGH ASPECT RATIO FEATURES UTILIZING A SACRIFICIAL ETCH CAP LAYER

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims benefit of U.S. Patent Application No. 15/703,917, filed
5 September 13, 2017, and titled “DIELECTRIC GAPFILL OF HIGH ASPECT RATIO
FEATURES UTILIZING A SACRIFICIAL ETCH CAP LAYER,” which is incorporated by
reference herein in its entirety and for all purposes.

BACKGROUND

[0002] Semiconductor fabrication processes involve patterning operations, including
10 depositing and etching of various materials on a semiconductor substrate. Substrates include
features of various types, including horizontal and vertical features, negative features with sloped
sidewalls, features with re-entrant features, and features made as negative features in substrates
having a multi-layered stack of two or more materials such that the composition of the material
on the surface of sidewalls of the feature varies depending on the depth of the feature. Various
15 techniques for filling such features exist, but as devices shrink and features become smaller,
feature fill without voids or seams becomes increasingly challenging.

SUMMARY

[0003] Methods and apparatuses for processing semiconductor substrates are provided herein.
One aspect involves a method of filling a feature on a substrate, the method including: providing
20 the substrate including the feature to a process chamber, the feature including a feature opening
and sidewall topography, the sidewall topography including stubs on the sidewall of the feature;
depositing a first amount of silicon oxide for a duration insufficient to fill the feature using a
silicon-containing precursor and an oxidant; exposing the first amount of the silicon oxide to an
etchant to etch at least some of the first amount of the silicon oxide; and after etching the first
25 amount of the silicon oxide, depositing a second amount of the silicon oxide over the etched first
amount of the silicon oxide.

[0004] In various embodiments, the stubs have a dimension perpendicular to a planar surface
of the sidewall of between 100 Å and about 300 Å.

[0005] In various embodiments, the duration insufficient to deposit the first amount of silicon
30 oxide into the feature forms an overburden of silicon oxide on a field surface of the substrate. In
some embodiments, exposing the first amount of the silicon oxide to the etchant includes etching
the at least some of the first amount of the silicon oxide at or near the feature opening relative to
the inside of the feature.

[0006] In various embodiments, the second amount of the silicon oxide is deposited by plasma-enhanced chemical vapor deposition. In some embodiments, the method may also include, after depositing the second amount of the silicon oxide, exposing the second amount of the silicon oxide to the etchant for a duration longer than the duration used for exposing the first amount of the silicon oxide to the etchant.

[0007] In some embodiments, the sidewalls of the feature include two or more materials layered in a stack.

[0008] In various embodiments, the depositing of the first amount of the silicon oxide and the exposing of the first amount of the silicon oxide to the etchant are performed without breaking vacuum. In various embodiments, the depositing of the first amount of the silicon oxide and the exposing of the first amount of the silicon oxide to the etchant are performed in the same chamber.

[0009] In some embodiments, the exposing of the first amount of the silicon oxide to the etchant and the depositing of the second amount of the silicon oxide are performed without breaking vacuum. In some embodiments, the exposing of the first amount of the silicon oxide to the etchant and the depositing of the second amount of the silicon oxide are performed in the same chamber.

[0010] In various embodiments, the depositing of the first amount of the silicon oxide, the exposing of the first amount of the silicon oxide to the etchant, and the depositing of the second amount of the silicon oxide are performed without breaking vacuum are performed without breaking vacuum. In various embodiments, the depositing of the first amount of the silicon oxide, the exposing of the first amount of the silicon oxide to the etchant, and the depositing of the second amount of the silicon oxide are performed without breaking vacuum are performed in the same chamber.

[0011] In various embodiments, the first amount of the silicon oxide is deposited by one or more cycles of atomic layer deposition, each cycle of one or more cycles of the atomic layer deposition including alternating pulses of the oxidant and the silicon-containing precursor. In various embodiments, the duration insufficient to fill the feature is the duration for performing about 40 cycles or less of the atomic layer deposition. In some embodiments, a plasma is ignited during the pulses of the oxidant. In various embodiments, each cycle of one or more cycles of the atomic layer deposition further includes purging the process chamber between the alternating pulses of the oxidant and the silicon-containing precursor. In some embodiments, the process chamber is purged after depositing the first amount of silicon oxide and before exposing the first amount of the silicon oxide to the etchant. In some embodiments, the process chamber is purged

after exposing the first amount of the silicon oxide to the etchant and before depositing the second amount of the silicon oxide.

[0012] In various embodiments, the etchant is any one of nitrogen trifluoride (NF₃), fluoroform (CHF₃), octafluorocyclobutane (C₄F₈), tetrafluoromethane (CF₄), and combinations thereof.

5 **[0013]** In various embodiments, the feature has a depth of at least 5 microns. In various embodiments, the feature has an aspect ratio of at least 15:1.

[0014] A method of filling a feature on a substrate, the method including: providing the substrate including the feature to a process chamber, the feature including a feature opening and sidewall topography, the sidewall topography including stubs on the sidewall of the feature;
10 depositing a first amount of silicon oxide for a duration insufficient to fill the feature using a silicon-containing precursor and an oxidant; after depositing the first amount of silicon oxide and prior to exposing the first amount of silicon oxide to an etchant, depositing a sacrificial helmet forming an overburden on a field surface of the substrate; exposing the substrate to the etchant to etch at least some of the first amount of the silicon oxide; and after etching the first amount of
15 the silicon oxide, depositing a second amount of the silicon oxide over the etched first amount of the silicon oxide to at least partially fill the feature.

[0015] In various embodiments, the stubs have a dimension perpendicular to a planar surface of the sidewall of between 100 Å and about 300 Å.

[0016] In various embodiments, the sacrificial helmet is deposited by plasma enhanced
20 chemical vapor deposition.

[0017] In various embodiments, the sacrificial helmet includes silicon nitride.

[0018] In various embodiments, the sacrificial helmet includes silicon oxide.

[0019] In various embodiments, the depositing of the first amount of the silicon oxide and the depositing of the sacrificial helmet are performed without breaking vacuum. In some
25 embodiments, the depositing of the first amount of the silicon oxide and the exposing of the substrate to the etchant are performed without breaking vacuum. In some embodiments, the depositing of the first amount of the silicon oxide, the depositing of the sacrificial helmet, and the exposing of the substrate to the etchant are performed without breaking vacuum. In some
30 embodiments, the exposing of the substrate to the etchant and the depositing of the second amount of the silicon oxide are performed without breaking vacuum. In some embodiments, the depositing of the sacrificial helmet, the exposing of the substrate to the etchant and the depositing of the second amount of the silicon oxide are performed without breaking vacuum. In some embodiments, the depositing of the first amount of the silicon oxide, the depositing of the sacrificial helmet, the exposing of the substrate to the etchant and the depositing of the second

amount of the silicon oxide are performed without breaking vacuum.

[0020] In various embodiments, the depositing of the first amount of the silicon oxide and the depositing of the sacrificial helmet are performed in the same chamber. In some embodiments, the depositing of the first amount of the silicon oxide and the exposing of the substrate to the etchant are performed in the same chamber. In some embodiments, the depositing of the first amount of the silicon oxide, the depositing of the sacrificial helmet, and the exposing of the substrate to the etchant are performed in the same chamber. In some embodiments, the depositing of the sacrificial helmet, the exposing of the substrate to the etchant and the depositing of the second amount of the silicon oxide are performed in the same chamber. In some embodiments, the depositing of the first amount of the silicon oxide, the depositing of the sacrificial helmet, the exposing of the substrate to the etchant and the depositing of the second amount of the silicon oxide are performed in the same chamber.

[0021] In various embodiments, the first amount of the silicon oxide is deposited by one or more cycles of atomic layer deposition, each cycle of one or more cycles of the atomic layer deposition including alternating pulses of the oxidant and the silicon-containing precursor. In various embodiments, the duration insufficient to fill the feature is the duration for performing about 40 cycles or less of the atomic layer deposition. In some embodiments, a plasma is ignited during the pulses of the oxidant. In various embodiments, each cycle of one or more cycles of the atomic layer deposition further includes purging the process chamber between the alternating pulses of the oxidant and the silicon-containing precursor. In some embodiments, the process chamber is purged after depositing the first amount of silicon oxide and before exposing the first amount of the silicon oxide to the etchant. In some embodiments, the process chamber is purged after exposing the first amount of the silicon oxide to the etchant and before depositing the sacrificial helmet.

[0022] In various embodiments, the etchant is any one of nitrogen trifluoride (NF_3), fluoroform (CHF_3), octafluorocyclobutane (C_4F_8), tetrafluoromethane (CF_4), and combinations thereof.

[0023] In various embodiments, the feature has a depth of at least 5 microns. In various embodiments, the feature has an aspect ratio of at least 15:1.

[0024] Another aspect involves a method of filling a feature on a substrate, the method including: providing the substrate including the feature to a process chamber, the feature including a feature opening and sidewalls having one or more reentrant surfaces; depositing a first amount of a material for a duration insufficient to fill the feature; exposing the first amount of the material to an etchant to etch at least some of the first amount of the material within the feature; and after etching the first amount of the material, depositing a second amount of the

material over the etched first amount of the material, whereby the material is any of silicon carbide, silicon nitride, silicon, tungsten, ruthenium, copper, cobalt, and molybdenum.

[0025] In various embodiments, the duration sufficient to deposit the first amount of material into the feature forms an overburden of silicon oxide on a field surface of the substrate.

5 **[0026]** In various embodiments, exposing the first amount of the material to the etchant includes etching the at least some of the first amount of the material at or near the feature opening relative to the inside of the feature.

[0027] In various embodiments, the second amount of the material is deposited by plasma-enhanced chemical vapor deposition.

10 **[0028]** In some embodiments, the method also includes, after depositing the second amount of the material, exposing the second amount of the material to the etchant for a duration longer than the duration used for exposing the first amount of the material to the etchant.

[0029] In various embodiments, the sidewalls of the feature include two or more materials layered in a stack.

15 **[0030]** In various embodiments, depositing of the first amount of the material and the exposing of the first amount of the material to the etchant are performed without breaking vacuum.

[0031] In various embodiments, the exposing of the first amount of the material to the etchant and the depositing of the second amount of the material are performed without breaking vacuum.

20 **[0032]** Another aspect involves a method of filling a feature on a substrate, the method including: providing the substrate including the feature to a process chamber, the feature including a feature opening and sidewall topography, the sidewall topography including stubs on the sidewall of the feature; depositing a first amount of a first material for a duration insufficient to fill the feature; after depositing the first amount of a first material and prior to exposing the first amount of the first material to an etchant, depositing a sacrificial helmet forming an
25 overburden on a field surface of the substrate, the sacrificial helmet including a second material; exposing the substrate to the etchant to etch at least some of the first amount of the first material; and after etching the first amount of the first material, depositing a second amount of the oxide over the etched first amount of the first material to at least partially fill the feature.

30 **[0033]** In various embodiments, the stubs have a dimension perpendicular to a planar surface of the sidewall of between 100 Å and about 300 Å.

[0034] In various embodiments, the first material is different from the second material. In various embodiments, the first material is compositionally the same as the second material. In various embodiments, the second material is deposited by plasma enhanced chemical vapor deposition.

[0035] In various embodiments, the first material is deposited by atomic layer deposition. In some embodiments, the second material is deposited by introducing a silicon-containing precursor and a nitrogen-containing reactant simultaneously while igniting a plasma to form silicon nitride. In various embodiments, the nitrogen-containing reactant is introduced with oxygen. In some embodiments, the etchant is a halogen-containing etchant. For example, in some embodiments, the etchant is nitrogen trifluoride. In various embodiments, the sacrificial helmet has etch selectivity relative to the first material when using the etchant. In some embodiments, the etch selectivity of sacrificial helmet to the first material is between about 1:2 and about 1:5, where the first material etches about 2 to about 5 times faster than the sacrificial helmet.

[0036] Another aspect involves an apparatus for processing a semiconductor substrate, the apparatus including: (a) at least one process chamber, the at least one process chamber including a pedestal for holding the semiconductor substrate; (b) at least one outlet for coupling to a vacuum; (c) one or more process gas inlets coupled to one or more process gas sources; and (d) a controller for controlling operations in the apparatus, including machine-readable instructions for: (i) introducing a silicon-containing precursor and an oxidant to deposit a first amount of silicon oxide on the semiconductor substrate for a duration insufficient to fill a feature on the semiconductor substrate, the feature having a feature opening and sidewall topography, the sidewall topography having stubs on the sidewall of the feature; (ii) introducing an etchant to the at least one process chamber for a duration to etch at least some of the first amount of the silicon oxide; and (iii) after introducing the etchant to the at least one process chamber, introducing the silicon-containing precursor and the oxidant to deposit a second amount of silicon oxide over the etched first amount of the silicon oxide.

[0037] In various embodiments, the controller further includes instructions for setting the duration of (iii) to be longer than the duration of (i). In various embodiments, the controller further includes machine-readable instructions for performing (i) and (ii) without breaking vacuum.

[0038] In various embodiments, the apparatus also includes a plasma generator for generating a plasma. In some embodiments, the controller further includes instructions for igniting the plasma when introducing the oxidant.

[0039] Another aspect involves an apparatus for processing a semiconductor substrate, the apparatus including: (a) at least one process chamber, the at least one process chamber including a pedestal for holding the semiconductor substrate; (b) at least one outlet for coupling to a vacuum; (c) one or more process gas inlets coupled to one or more process gas sources; and (d) a

controller for controlling operations in the apparatus, including machine-readable instructions for: (i) introducing a deposition precursor and reactant for depositing a first amount of a material for a duration insufficient to fill a feature on the semiconductor substrate; (ii) introducing an etchant to etch at least some of the first amount of the material in the feature; and (iii) after
5 introducing the etchant, introducing the deposition precursor and the reactant to deposit a second amount of the material over the etched first amount of the material, whereby the material is any one of silicon carbide, silicon nitride, silicon, tungsten, ruthenium, copper, cobalt, and molybdenum.

[0040] In various embodiments, the controller further includes instructions for setting the
10 duration of (iii) to be longer than the duration of (i). In various embodiments, the controller further includes machine-readable instructions for performing (i) and (ii) without breaking vacuum.

[0041] In various embodiments, the apparatus also includes a plasma generator for generating a plasma. In some embodiments, the controller further includes instructions for igniting the
15 plasma when introducing the oxidant.

[0042] Another aspect involves an apparatus for processing a semiconductor substrate, the apparatus including: (a) at least one process chamber, the at least one process chamber including a pedestal for holding the semiconductor substrate; (b) at least one outlet for coupling to a vacuum; (c) one or more process gas inlets coupled to one or more process gas sources; and (d) a
20 controller for controlling operations in the apparatus, including machine-readable instructions for: (i) introducing a silicon-containing precursor and an oxidant to deposit a first amount of silicon oxide on the semiconductor substrate for a duration insufficient to fill a feature on the semiconductor substrate, the feature having a feature opening and sidewall topography, the sidewall topography having stubs on the sidewall of the feature; (ii) introducing one or more
25 process gases for depositing a sacrificial helmet forming an overburden on a field surface of the semiconductor substrate; (iii) introducing an etchant to the at least one process chamber for a duration to etch at least some of the first amount of the silicon oxide; and (iv) after introducing the etchant to the at least one process chamber, introducing the silicon-containing precursor and the oxidant to deposit a second amount of silicon oxide over the etched first amount of the
30 silicon oxide.

[0043] In various embodiments, the controller includes instructions for delivering a second silicon-containing precursor and a nitrogen-containing reactant during (ii) to deposit the sacrificial helmet, the sacrificial helmet including silicon nitride.

[0044] Another aspect involves an apparatus for processing a semiconductor substrate, the

apparatus including: (a) at least one process chamber, the at least one process chamber including a pedestal for holding the semiconductor substrate; (b) at least one outlet for coupling to a vacuum; (c) one or more process gas inlets coupled to one or more process gas sources; and (d) a controller for controlling operations in the apparatus, including machine-readable instructions for: (i) introducing a first set of deposition precursors for depositing a first material to deposit a first amount of the first material on the semiconductor substrate for a duration insufficient to fill a feature on the semiconductor substrate; (ii) introducing one or more process gases for depositing a sacrificial helmet forming an overburden on a field surface of the semiconductor substrate, the sacrificial helmet including a second material; (iii) introducing an etchant to the at least one process chamber for a duration to etch at least some of the first amount of the first material; and (iv) after introducing the etchant to the at least one process chamber, introducing first set of deposition precursors to deposit a second amount of the first material over the etched first amount of the first material.

[0045] In various embodiments, the first material is different from the second material. In various embodiments, the first material is compositionally the same as the second material.

[0046] These and other aspects are described further below with reference to the drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

[0047] Figures 1A-1E are schematic illustrations of example substrates with features of various types.

[0048] Figures 2A-2E are schematic illustrations of example substrates with features of various types filled with material.

[0049] Figures 3A-3D are schematic illustrations of an example feature in a substrate with a re-entrant feature undergoing feature fill.

[0050] Figures 4A-4D are schematic illustrations of an example feature in a substrate with sidewall oscillations undergoing feature fill.

[0051] Figures 5A-5C are schematic illustrations of an example feature in a substrate with a multi-layer stack undergoing feature fill.

[0052] Figures 6 and 7 are process flow diagrams depicting operations performed in methods performed in accordance with certain disclosed embodiments.

[0053] Figures 8A-8D are schematic illustrations of an example feature in a substrate with a re-entrant feature undergoing feature fill in accordance with certain disclosed embodiments.

[0054] Figures 9A-9D are schematic illustrations of an example feature in a substrate with sidewall topography undergoing feature fill in accordance with certain disclosed embodiments.

[0055] Figures 9E-9H are schematic illustrations of an example feature in a substrate with sidewall topography undergoing feature fill in accordance with certain disclosed embodiments.

[0056] Figures 10A-10C are schematic illustrations of an example feature in a substrate with a multi-layer stack undergoing feature fill in accordance with certain disclosed embodiments.

5 [0057] Figure 11 is a timing schematic diagram depicting an example of operations performed in accordance with certain disclosed embodiments.

[0058] Figure 12 is a schematic diagram of an example process chamber for performing certain disclosed embodiments.

10 [0059] Figure 13 is a schematic diagram of an example process tool for performing certain disclosed embodiments.

DETAILED DESCRIPTION

[0060] In the following description, numerous specific details are set forth to provide a thorough understanding of the presented embodiments. The disclosed embodiments may be practiced without some or all of these specific details. In other instances, well-known process
15 operations have not been described in detail to not unnecessarily obscure the disclosed embodiments. While the disclosed embodiments will be described in conjunction with the specific embodiments, it will be understood that it is not intended to limit the disclosed embodiments.

[0061] Semiconductor fabrication processes often include dielectric gap fill using chemical
20 vapor deposition (CVD) and/or atomic layer deposition (ALD) methods to fill features. Described herein are methods of filling features with material, such as dielectric or metal material, including but not limited to silicon oxide, and related systems and apparatuses. The methods described herein can be used to fill vertical negative features. Features formed in a substrate can be characterized by one or more of narrow and/or re-entrant openings, constrictions
25 within the feature, and high aspect ratios. The substrate may be a silicon wafer, e.g., a 200-mm wafer, a 300-mm wafer, or a 450-mm wafer, including wafers having one or more layers of material such as dielectric, conducting, or semi-conducting material deposited thereon.

[0062] A feature may be formed in one or more of these layers. For example, a feature may be formed at least partially in a dielectric layer. A single substrate as described herein includes
30 multi-laminate stack having two or more materials, such a ONON (oxide-nitride-oxide-nitride) stack, a OPOP (silicon oxide on polysilicon) stack, or OMOM stack (silicon oxide on metal such as tungsten, cobalt, or molybdenum) and features may be formed in such multi-layer substrates where sidewalls of the features include two or more compositions. Multi-laminate stacks may

range from a bilayer (such as ON) to 500 combined layers (such as {ON}₁₅₀). Features may also have sloped sidewalls or sidewall topography. Sidewall topography include jagged sidewalls having one or more stubs, each stub having a dimension perpendicular to a planar surface of the sidewall of between 100Å and about 300Å. In various embodiments, sidewall topography is characterized by two or more stubs where the two or more stubs are present on the sidewalls of a feature depth wise. That is, one stub on the sidewall is at a depth different than the depth at which the second stub is present on the sidewall. Additionally, features may also include a re-entrant profile.

[0063] A feature hole may also have a dimension near the opening, e.g., an opening diameter or line width, of between about 1 nm to about 1 micron, for example between about 25 nm to about 300 nm, such as about 200 nm. A feature hole can be referred to as an unfilled feature or simply a feature. In some implementations, a feature hole may have an aspect ratio of at least about 2:1, at least about 4:1, at least about 6:1, or at least about 20:1, or greater.

[0064] Figures 1A-1E show example substrates with features of various types. Figure 1A shows feature 102a in a substrate having a multi-layer stack includes two compositions 104a and 114a in alternating horizontal layers, where feature 102a is defined by etching a hole into liner 106a. Note that while a liner 106a is depicted in various examples described herein, in some embodiments a liner is not present. Where a liner is present, the liner may be a nitride material, such as but not limited to silicon nitride, or in some embodiments, the liner may be a poly-silicon material.

[0065] Figure 1B shows a feature 102b in a substrate 104b having straight sidewalls defined by liner 106b. Feature 102b of Figure 1B has a high aspect ratio with straight sidewalls. Figure 1C shows an example feature 102c in substrate 104c with liner 106c thereby forming feature 102c with positively sloped sidewalls where the feature opening 120c at the top of the feature 102c is greater than the width at the bottom 112c. Figure 1D shows a feature 102d in a substrate 104d where the feature 102d is defined by etching a hole in liner 106d, and the feature 102d includes negatively sloped sidewalls, or a re-entrant profile, where the feature opening 120d at the top of the feature 102d is narrower than the width of the bottom 112d of the feature 102d. Figure 1E shows a feature 102e in a substrate 104e where the feature 102e is defined by etching a hole in liner 106e, and the feature 102e includes a sidewall topography having stubs. In some cases, a feature can have sidewall topography due to the type of etching performed to form the feature, or the one or more materials in which the feature is formed. Gapfill of features such as these examples shown in Figures 1A-1E is dependent on the feature type and profile. While existing techniques such as CVD and ALD may be used to fill such features, conventional techniques

result in formation of undesirable seams or voids within the feature.

[0066] Figure 2A shows an example feature 202a with liner 206a in a substrate including a multi-layer stack having material 204a and 214a in alternating layers. Dielectric material 208a is deposited into the feature 202a by conformal ALD, but a seam 210 forms in the middle of the feature 202a because dielectric material 208a growth from the sidewalls meets in the middle of the feature 202a, thereby forming the seam 210.

[0067] Figure 2B shows a similar phenomenon for feature 202b filled with dielectric material 208b by ALD over liner 206b defining the feature 202b having straight sidewalls in substrate 204b. Likewise, in Figure 2C, the positively sloped feature 202c including a narrower width at the bottom 212a in substrate 204c with sidewalls defined by liner 206c also results in formation of seam 210c when dielectric material 208c is deposited by conformal ALD.

Figure 2D also shows a feature 202d having negatively sloped features with a wider width at the bottom 212d defined by liner 206d in substrate 204d. In this example, a seam 210d is still formed when dielectric material 208d is deposited. In some cases, a void may also form at or

near the bottom of the feature 202d due to the conformal fill by ALD. Figure 2E shows an example of feature 202e having sidewall topography defined by liner 206e in substrate 204e. The sidewall topography is characterized by stubs 216e, and the bottom of the feature 212e is wider than the width at the stubs 216e. When material 208e is deposited by ALD, microvoids 210e form and the feature is unable to be filled completely. Microvoids may be defined as voids being less than about 50 nm in diameter, or in some embodiments between about 30 nm and about 50 nm in diameter, or less than 30 nm in diameter, such as less than about 15 nm wide.

[0068] Some dep-etch-dep (deposition, etch, deposition) techniques have been posited to fill various features with dielectric material. However, existing dep-etch-dep techniques also cause undesirable etching of other material on the substrate, thereby resulting in material loss. Examples are provided in Figure 3A-3D, 4A-4D, and 5A-5C as described below.

[0069] Figure 3A shows an example of a feature 302a in substrate 304a where the feature profile is defined by liner 306a. As shown, the bottom of the feature depicted at 312a has a narrower width than a portion of the width at or near the feature opening, for which a re-entrant profile is depicted. In conventional conformal ALD, material 308b fills the feature 302b layer by layer as shown in Figure 3B. In Figure 3B, material 308b is depicted as being deposited into feature 302b, the feature profile of which is defined in liner 306b of substrate 304b. However, ALD deposition is conformal and results in the formation of void 310b, while the bottom of the feature 312b is completely filled. Conventional dep-etch-dep involves etching after deposition

of ALD, such as shown in Figure 3C. In Figure 3C, the deposited dielectric material 308c is etched back to open the narrow feature opening of feature 302c in substrate 304c. However, even after the dielectric material 308c is etched to the surface of liner 306c, void 310c still remains while the opening 308c is not yet open. The bottom of the feature 312c remains filled.

5 In conventional dep-etch-dep, etch is performed until the feature is opened to allow further fill of the feature, and thus as shown in Figure 3D, etch back causes material loss 316d of the liner 306d to open the feature 302d and gain access to the void 310d formed from prior deposition by ALD of material 308d. The bottom of the feature 312d remains filled in the substrate 308d. However, such material loss 316d is undesirable.

10 **[0070]** Figures 4A-4D also show an example of undesirable etching caused by conventional dep-etch-dep processes for filling features with dielectric material. Figure 4A includes a feature 402a in substrate 404a having sidewall topography formed by etching in liner 406a, the sidewall topography having stubs where the feature width at the stubs is narrower than at the bottom 412a of the feature. In such an embodiment, some stubs are at the same depth. In
15 Figure 4B, ALD is performed to deposit dielectric material 408b conformally into the feature 402b over liner 406b. Note the space 410b between deposited material on the sidewalls of the feature 402b remains open and the material 408b is deposited over stubs 416b, including the bottom 412b. However, to avoid formation of voids between sidewall topography, in Figure 4C, the deposited dielectric material 408b is etched back to smoothen the deposited
20 material, as depicted by the smoothened surface 414c of material 408c. Note that the feature 402c still includes rough deposited material near the stubs 416c and the bottom 412c of the feature 402c of substrate 404c. In Figure 4D, the substrate 404 is further etched back to allow smoothening along the entire sidewall to reduce the formation of microvoids, as shown in the smoothening at the top of the feature 414d and the smoothening at the bottom of the
25 feature 416d. Note that the dielectric material 408d at or near the stubs 416d and the bottom 412d of the feature are smoothened such that subsequent deposition by ALD will not form microvoids. However, such etch back resulted in material loss 416d of the liner 406d, which could thereby cause problems downstream.

[0071] Figures 5A-5C also show an example of a substrate having a multi-layer stack of two
30 compositions 504a and 514a where the substrate includes features 502a with material 508a deposited over liner 506a conformally. In Figure 5B, the material 508b is etched back to open the feature opening of features 502b, but such etching results in etching of liner 506b, thereby resulting in a tapered profile. In subsequent deposition of material 508c in Figure 5C, the material deposits over the tapered profile, the result being a substrate with filled features but with

material loss of the stack.

[0072] These and other issues with existing techniques result in undesired feature profiles for gapfill of high aspect ratio features and features with sidewall topography.

5 [0073] Provided herein are methods and apparatuses for depositing dielectric material into features having high aspect ratios, sidewall topography, multi-layer stack compositions, and re-entrant profiles. Methods include modulated dep-etch-dep durations and conditions to reduce and eliminate etching of a feature profile and underlayers of the substrate, including depositing a sacrificial helmet using plasma-enhanced chemical vapor deposition on the field surface of the substrate, thereby resulting in an overburden of material at the feature opening (which may be
10 the same as or different from the material being deposited in the feature) and performing a longer etch back to open the feature while consuming only the sacrificial overburden of material without etching the underlying layers of the substrate and thus preventing etching of the feature profile.

[0074] While the description below focuses on dielectric feature fill, aspects of the disclosure
15 may also be implemented in filling features with other materials. For example, feature fill using one or more techniques described herein may be used to fill features with other materials including silicon-containing materials (e.g., silicon carbide, silicon nitride, silicon oxide, silicon) and metal-containing materials (e.g., tungsten, ruthenium, copper, cobalt, molybdenum, nitrides and carbides thereof).

20 [0075] Figure 6 is a process flow diagram depicting operations of a method performed in accordance with certain disclosed embodiments. Operations in Figure 6 may be performed at a substrate temperature between about 50°C and about 650°C.

[0076] In operation 602, a substrate is provided having features with sidewall topography. For example, the features may have a profile such as that depicted in Figure 1E. Although the
25 example described with respect to Figure 6 is related to features with sidewall topography, it will be understood that in some embodiments, disclosed embodiments may be implemented on any one or more of high aspect ratio features with vertical sidewalls, features with re-entrant profiles, features with positively sloped sidewalls, features with negatively sloped sidewalls, and features with multi-stack compositions on the sidewalls such as depicted in Figures 1B, 1C, 1D, and 1A
30 respectively. The substrate may be provided to a process chamber, which may be within a station of a single station or multi-station apparatus for processing one or more wafers.

[0077] In operation 604, a first amount of dielectric material is deposited in the features. In various embodiments, the first amount of dielectric material is insufficient to fill the feature. An insufficiently filled feature is defined as a feature having some dielectric material deposited

therein and one or more voids present in the feature. Insufficiently filled features include features where material is deposited along the sidewalls of the feature but the feature opening remains open. Deposition during operation 604 may be performed at a chamber pressure between about 0.1 Torr and about 15 Torr, such as about 6 Torr.

5 **[0078]** In various embodiments, the dielectric material is silicon oxide. Silicon oxide may be deposited by ALD, plasma-enhanced ALD (PEALD), CVD, or plasma-enhanced CVD (PECVD). ALD is a technique that deposits thin layers of material using sequential self-limiting reactions. ALD processes use surface-mediated deposition reactions to deposit films on a layer-by-layer basis in cycles. As an example, an ALD cycle may include the following
10 operations: (i) delivery/adsorption of a precursor, (ii) purging of the precursor from the chamber, (iii) delivery of a second reactant and optional plasma ignition, and (iv) purging of byproducts from the chamber. The reaction between the second reactant and the adsorbed precursor to form a film on the surface of a substrate affects the film composition and properties, such as nonuniformity, stress, wet etch rate, dry etch rate, electrical properties (e.g., breakdown voltage
15 and leakage current), etc.

[0079] In one example of an ALD process, a substrate surface that includes a population of surface active sites is exposed to a gas phase distribution of a first precursor, such as a silicon-containing precursor, in a dose provided to a chamber housing the substrate. Molecules of this first precursor are adsorbed onto the substrate surface, including chemisorbed species
20 and/or physisorbed molecules of the first precursor. It should be understood that when a compound is adsorbed onto the substrate surface as described herein, the adsorbed layer may include the compound as well as derivatives of the compound. For example, an adsorbed layer of a silicon-containing precursor may include the silicon-containing precursor as well as derivatives of the silicon-containing precursor. After a first precursor dose, the chamber is then
25 evacuated to remove most or all of first precursor remaining in gas phase so that mostly or only the adsorbed species remain. In some implementations, the chamber may not be fully evacuated. For example, the reactor may be evacuated such that the partial pressure of the first precursor in gas phase is sufficiently low to mitigate a reaction. A second reactant, such as an oxygen-containing gas, is introduced to the chamber so that some of these molecules react with
30 the first precursor adsorbed on the surface. In some processes, the second reactant reacts immediately with the adsorbed first precursor. In other embodiments, the second reactant reacts only after a source of activation such as plasma is applied temporally. The chamber may then be evacuated again to remove unbound second reactant molecules. As described above, in some embodiments the chamber may not be completely evacuated. Additional ALD cycles may be

used to build film thickness.

[0080] In some implementations, the ALD methods include plasma activation. As described herein, the ALD methods and apparatuses described herein may be conformal film deposition (CFD) methods, which are described generally in U.S. Patent Application No. 13/084,399 (now U.S. Patent No. 8,728,956), filed April 11, 2011, and titled "PLASMA ACTIVATED CONFORMAL FILM DEPOSITION," which is herein incorporated by reference in its entirety.

[0081] For depositing silicon oxide, one or more silicon-containing precursors may be used. Silicon-containing precursors suitable for use in accordance with disclosed embodiments include polysilanes ($\text{H}_3\text{Si}-(\text{SiH}_2)_n-\text{SiH}_3$), where $n \geq 0$. Examples of silanes are silane (SiH_4), disilane (Si_2H_6), and organosilanes such as methylsilane, ethylsilane, isopropylsilane, t-butylsilane, dimethylsilane, diethylsilane, di-t-butylsilane, allylsilane, sec-butylsilane, hexylsilane, isoamylsilane, t-butylidisilane, di-t-butylidisilane, and the like.

[0082] A halosilane includes at least one halogen group and may or may not include hydrogens and/or carbon groups. Examples of halosilanes are iodasilanes, bromosilanes, chlorosilanes, and fluorosilanes. Although halosilanes, particularly fluorosilanes, may form reactive halide species that can etch silicon materials when a plasma is struck, a halosilane may not be introduced to the chamber when a plasma is struck in some embodiments, so formation of a reactive halide species from a halosilane may be mitigated. Specific chlorosilanes are tetrachlorosilane, trichlorosilane, dichlorosilane, monochlorosilane, chloroallylsilane, chloromethylsilane, dichloromethylsilane, chlorodimethylsilane, chloroethylsilane, t-butylchlorosilane, di-t-butylchlorosilane, chloroisopropylsilane, chloro-sec-butylsilane, t-butyl dimethylchlorosilane, hexyldimethylchlorosilane, and the like.

[0083] An aminosilane includes at least one nitrogen atom bonded to a silicon atom, but may also contain hydrogens, oxygens, halogens, and carbons. Examples of aminosilanes are mono-, di-, tri- and tetra-aminosilane ($\text{H}_3\text{Si}(\text{NH}_2)$, $\text{H}_2\text{Si}(\text{NH}_2)_2$, $\text{HSi}(\text{NH}_2)_3$ and $\text{Si}(\text{NH}_2)_4$, respectively), as well as substituted mono-, di-, tri- and tetra-aminosilanes, for example, t-butylaminosilane, methylaminosilane, tert-butylsilanamine, bis(tert-butylamino)silane ($\text{SiH}_2(\text{NHC}(\text{CH}_3)_3)_2$ (BTBAS), tert-butyl silylcarbamate, $\text{SiH}(\text{CH}_3)-(\text{N}(\text{CH}_3)_2)_2$, $\text{SiHCl}(\text{N}(\text{CH}_3)_2)_2$, $(\text{Si}(\text{CH}_3)_2\text{NH})_3$ and the like. A further example of an aminosilane is trisilylamine ($\text{N}(\text{SiH}_3)_3$).

[0084] Additional reactants are also used during operation 604. For deposition of a silicon oxide helmet by PECVD, an oxidant is flowed to the process chamber with the silicon-containing precursor to react and deposit silicon oxide on the substrate. Example oxidants include oxygen gas, water, carbon dioxide, nitrous oxide, and combinations thereof. In various embodiments, the substrate is exposed to an oxidant and an inert gas simultaneously while the

plasma is ignited. For example, in one embodiment, a mixture of oxygen and argon is introduced to the substrate while the plasma is ignited. Example inert gases include helium and argon. In some embodiments, the inert gas acts as a carrier gas to deliver the process gases to the substrate and is diverted upstream of the chamber. For deposition by ALD or PEALD, the silicon-containing precursor and the reactant are introduced sequentially in pulses, which may be separated by purging operations. Such examples are further described below with respect to Figure 7.

[0085] In embodiments where a combination of any of ALD, PEALD, CVD, and PECVD are used to deposit the dielectric material in operation 604, the same reactants and precursors may be used during both techniques. In some embodiments, different precursors may be selected depending on the technique. For example, in some embodiments, ALD may be performed using a halosilane, followed by PECVD using silane as the silicon-containing precursor. In some embodiments, a plasma is ignited during one or more of the techniques used to deposit the dielectric material.

[0086] In operation 607, a sacrificial helmet is deposited on a field surface of the substrate. A helmet is an overburden of material deposited preferentially on the field surface of the substrate relative to the interior of features on the substrate. In various embodiments, the helmet is a sacrificial material used to cushion subsequent etching processes so as to protect the underlying feature profile from being etched or damaged.

[0087] In various embodiments, the helmet material grows on dielectric material that was deposited in operation 604 that remained on the field surface of the substrate. Thus, the sacrificial helmet does not close over the feature opening and the feature opening remains open. In various embodiments, the sacrificial helmet is deposited by PECVD. The sacrificial helmet functions as a layer used to protect the underlying substrate and feature profile during subsequent etching operation 608. The sacrificial helmet may be the same or different material as the dielectric material deposited into the features. For example, in some embodiments, the sacrificial helmet is a silicon oxide material deposited by PECVD while the material to be deposited into the features is also silicon oxide.

[0088] In some embodiments, the helmet is deposited to a thickness between about 10 Å and about 500 Å. A helmet may be deposited using ALD, CVD, or a combination of both. In various embodiments, the helmet is deposited by PECVD. In various embodiments, the features are exposed to deposition precursors for depositing a helmet (such as a silicon-containing precursor and a nitrogen-containing precursor for depositing a silicon nitride helmet) for a duration sufficient to form the helmet. The duration depends on the size of the feature opening,

the depth of the feature opening, the technique used to deposit the material, whether the material is the same as or different than the material deposited into the feature, and, if the material is the same as the material being deposited into the feature, the amount of material, if any, already deposited into the feature.

5 **[0089]** In some embodiments where the helmet material and the material being deposited into the feature are the same, the deposition of operation 604 may continue in cycles to deposit the sacrificial helmet for operation 607. In various embodiments, the deposition of operation 604 is performed by PEALD or ALD, while operation 607 is performed by PECVD. Where the materials of the helmet and material to be deposited into the features are the same, the same
10 precursors may be used in both operation 604 and operation 607. For example, any of the precursors and reactants described above with respect to operation 604 may be used for depositing a silicon oxide helmet in operation 607.

[0090] In some embodiments, the sacrificial helmet is a different material as the material deposited into the features. For example, in some embodiments, the sacrificial helmet is a silicon
15 nitride material deposited by PECVD while the material to be deposited into the features is silicon oxide. In various embodiments, the sacrificial helmet has etch selectivity to the material to be deposited when exposed to etchants used in operation 608, such that etching in operation 608 does not damage the sacrificial helmet and the helmet can therefore withstand many cycles of deposition and etch of the material to be deposited into the feature. In some
20 embodiments, the helmet is a silicon nitride material deposited by PECVD or PEALD using a silicon precursor and nitrogen plasma, simultaneously or in alternating pulses respectively. In various embodiments, formation of a silicon nitride helmet is performed after silicon oxide is used to deposit the first amount of silicon oxide in the feature, and silicon nitride is deposited on the field surface of the substrate. In various embodiments, silicon nitride used as the helmet
25 increases etch selectivity to the oxide during etching. For deposition by CVD, the substrate may be exposed to deposition precursors (such as a silicon-containing precursor and an oxidant for depositing silicon oxide) for a duration between about 2 seconds and about 120 seconds. In some embodiments, the helmet is deposited using a plasma-enhanced process, such as PEALD or PECVD.

30 **[0091]** For deposition of a silicon nitride helmet by PECVD, a nitrogen-containing gas such as nitrogen is flowed to the process chamber with a silicon-containing precursor to form silicon nitride. In various embodiments, nitrogen is ignited with a plasma to form silicon nitride.

[0092] In one example, silane is used as the silicon-precursor for performing PECVD of silicon nitride and/or silicon oxide. During deposition by PECVD, the precursor and reactants

are flowed at various flow rates. For example, for depositing silicon oxide, silane may be flowed with nitrogen and/or nitrous oxide. Silane may be introduced at a flow rate between about 50 sccm and about 200 sccm, such as about 75 sccm. Nitrogen may be introduced at a flow rate between about 1000 sccm and about 15000 sccm, such as about 3000 sccm. Nitrous oxide may be introduced at a flow rate between about 5000 sccm and about 25000 sccm, such as about 20000 sccm.

[0093] In operation 608, the dielectric material is etched from feature openings to leave a partially etched dielectric material in the feature. Etching may be performed for a duration sufficient to widen the feature opening such that subsequent deposition is capable of reaching the bottom of the feature. For example, in some embodiments etching is performed for a duration between about 100 seconds and about 400 seconds, such as about 115 seconds, or about 200 seconds, or about 300 seconds, or about 400 seconds. In various embodiments, etching is performed for a duration sufficient to open the feature opening without removing material underlying the layers of the dielectric material deposited into the feature in operation 604. In various embodiments, etching is performed with etch selectivity to the helmet material. For example, in some embodiments, nitrogen trifluoride (NF_3) is the etchant used during operation 608 and the etch selectivity of silicon oxide to a sacrificial silicon nitride helmet is between about 3:1 and about 5:1.

[0094] In some embodiments where operation 604 involves deposition of an overburden or helmet, etching is performed such that the feature opening exposed while consuming some but not all of the helmet of sacrificial dielectric material on the field surface, thereby leaving a partially etched dielectric layer in the feature. Some of the helmet may be consumed even if the helmet is a different material than that of the material to be deposited. However, in some embodiments, a helmet of a different material having etch selectivity to the material being deposited into the feature allows for the helmet to withstand longer etching times without being consumed as quickly as a helmet of the same material as the material being deposited would be etched under the same process conditions. In some embodiments, a helmet of a different material having etch selectivity to the material being deposited into the feature allows a thinner helmet to be deposited while still mitigating etching of the feature profile.

[0095] The etchant selected depends on the material to be etched. For example, for etching silicon oxide, etching may be performed using a fluorine-containing etch chemistry, such as by flowing nitrogen trifluoride (NF_3). Example etchants for etching silicon oxide include nitrogen trifluoride, fluoroform (CHF_3), octafluorocyclobutane (C_4F_8), tetrafluoromethane (CF_4), and combinations thereof. Example etchants for etching silicon carbide, silicon nitride, silicon,

tungsten, ruthenium, copper, cobalt, and molybdenum for feature fill using these materials include hydrobromic acid (HBr), fluoromethane (CH₃F), chlorine (Cl₂), silicon tetrafluoride (SiF₄), tetrafluoromethane (CF₄), boron trichloride (BCl₃), fluoroform (CHF₃) and combinations thereof.

5 **[0096]** In another example, for etching silicon carbide where silicon carbide is to be deposited into the features, etching may be performed using hydrobromic acid, or CH₃F.

[0097] In another example, for etching silicon nitride where silicon nitride is to be deposited into the features, etching may be performed using CH₃F.

10 **[0098]** In various embodiments, etchants may be flowed with one or more carrier gases, such as oxygen, nitrogen, and/or argon.

[0099] In various embodiments, a plasma is ignited during operation 608 to enhance etching. In some embodiments, the plasma is ignited using radio frequency plasma. In some embodiments, a self-bias bias may be applied to a powered pedestal holding the substrate during etch. In various embodiments, the plasma is ignited while flowing the nitrogen trifluoride gas
15 using a plasma power between about 1000 W and about 5000W. In various embodiments, the plasma is generated in-situ. In some embodiments, plasma may be generated remotely in a remote plasma chamber prior to delivering the process chamber housing the substrate.

[0100] In some embodiments, operations 604 and 608 are performed in different chambers. In some embodiments, operations 604 and 608 are performed in the same chamber. In some
20 embodiments, operations 604 and 608 are performed without breaking vacuum. For example, in some embodiments operations 604 and 608 are performed in separate stations in a multi-station chamber without breaking vacuum. Disclosed embodiments promote efficiency since deposition and etching may be performed in the same chamber or in the same tool.

[0101] In some embodiments, operation 608 may be performed to smooth the sidewalls.
25 Disclosed embodiments may also be suitable for smoothening sidewalls during deposition into features having sidewall topography such that etching performed between depositions of dielectric material is used to even out the surface and reduce the presence of stubs on the sidewalls of a feature. The etching may be performed by modulated the duration and plasma power to etch for a duration sufficient to smooth out the dielectric material deposited onto the
30 sidewalls of the feature without exposing the underlying material on the substrate. Smoothening of sidewalls may be performed by etching for a duration of about 200 seconds or less than about 200 seconds, or for a duration shorter than which to open a feature opening.

[0102] In operation 610, a second amount of dielectric material is deposited over the partially etched dielectric material. In various embodiments, the second amount of dielectric material is

deposited by ALD, PEALD, CVD, PECVD, or any combination thereof. Deposition may be performed using any suitable precursors and reactants. For example, for depositing a second amount of silicon oxide, any silicon-containing precursor described above with respect to operation 604 may be used for operation 610. Likewise, any suitable reactant for reacting with the precursor may be used. For example, for depositing silicon oxide, an oxidant such as oxygen or nitrous oxide may be used to react with the silicon-containing precursor. Any oxidant described above with respect to operation 604 may be used in operation 610.

[0103] In some embodiments, the process chamber is purged between operation 604 and operation 608. In some embodiments, the process chamber is purged between operation 608 and 610. In some embodiments, the process chamber is purged after operation 610. Purging the chamber may involve flowing a purge gas or a sweep gas, which may be a carrier gas used in other operations or may be a different gas. Example purge gases include argon, nitrogen, hydrogen, and helium. In various embodiments, the purge gas is an inert gas. Example inert gases include argon, nitrogen, and helium. In some embodiments, purging may involve evacuating the chamber. In some embodiments, purging may include one or more evacuation subphases for evacuating the process chamber. Alternatively, it will be appreciated that purging may be omitted in some embodiments. Purging may be performed for any suitable duration, such as between about 0.1 seconds and about 2 seconds.

[0104] In some embodiments, the second amount of the dielectric material fills the feature. In some embodiments, further operations are performed until the features are filled. For example, in some embodiments, operations 604, 608, and 610 are repeated in cycles. In some embodiments, after performing operation 604, operations 608 and 610 are repeated sequentially.

[0105] In some embodiments, operation 604 includes depositing a first amount of dielectric features by ALD and depositing a helmet by PECVD and etching in operation 608 is performed after deposition of the helmet. In some embodiments, the helmet is a different material than the dielectric used to fill the feature. For example, in some embodiments, silicon oxide is deposited in the features but silicon nitride is deposited as the helmet prior to etching. In some embodiments, using silicon nitride as the helmet where the material to be deposited into the feature is silicon oxide may be used to achieve high etch selectivity of the silicon nitride sacrificial helmet material relative to the silicon oxide material during etching to prevent removal of the material deposited in the feature. Additionally, use of a silicon nitride helmet may allow etching to be performed for a longer duration for less deposited silicon nitride, relative to using silicon oxide as a helmet. For example, for the same deposited thickness of a silicon nitride helmet versus a silicon oxide helmet, the silicon oxide helmet will etch faster than a silicon

nitride helmet. Thus, a thicker silicon oxide helmet is used to achieve a specified etching duration used to open a feature where a thinner silicon nitride material is used.

[0106] Figure 7 provides an example process flow diagram of an example method performed in accordance with certain disclosed embodiments. The example process in Figure 7 includes repetitions of various operations such as operations 604, 608, and 610 of Figure 6.

[0107] In operation 701, a substrate having features with sidewall topography is provided to a process chamber. Although the example described herein refers to features having sidewall topography, it will be understood that such embodiments can apply to substrates having features in a multi-laminate stack, features with positively sloped sidewalls, features with negatively sloped sidewalls, and features with re-entrant profiles. Operation 701 may correspond to embodiments of operation 602 described above with respect to Figure 6.

[0108] In operations 702a-1, 702b-1, 702c-1, and 702d-1, a first amount of dielectric material may be deposited into the features, and such operations may, in some embodiments, correspond to embodiments of operation 604 described above with respect to Figure 6. In this specific example, operations 702a-1 through 702d-1 may constitute one ALD cycle. In operation 702a-1, the substrate is exposed to a silicon-containing precursor, such as any of those described above with respect to operation 604, to adsorb the precursor onto the surface of the feature. In various embodiments, this operation is self-limiting. In some embodiments, the precursor adsorbs to less than all of the active sites on the surface of the feature. In operation 702b-1, the process chamber is optionally purged to remove any unadsorbed silicon-containing precursors. In operation 702c-1, the substrate is exposed to an oxidant and a plasma is ignited to form a first silicon oxide layer in the feature. In various embodiments, this layer is the first amount of dielectric material deposited in features referred to above in operation 604 with respect to Figure 6. In various embodiments, operation 702c-1 converts the adsorbed silicon-containing precursor layer to silicon oxide. In operation 702d-1, the process chamber is optionally purged to remove byproducts from the reaction between the silicon-containing precursor and the oxidant. Operations 702a-1 through 702d-1 may be optionally repeated for two or more cycles as desired to deposit silicon oxide to a desired thickness in the feature.

[0109] In operation 708a, the first amount of silicon oxide is partially etched from the feature. In various embodiments, this corresponds to operation 608 of Figure 6. Operation 708a may be performed for a duration sufficient to open the feature opening. For example, in some embodiments, cycles of operations 702a-1 through 702b-1 may deposit silicon oxide into the feature until pinch-off, upon which operation 708a is performed to open the feature opening and thereby allow subsequent deposition. Any etching chemistry as described above with respect to

operation 608 may be used. In some embodiments, a plasma is ignited to facilitate etching. It will be understood that the etching chemistry and the plasma conditions depend on the material being deposited in the features. For example, for deposition of silicon oxide, operation 708a may involve flowing nitrogen trifluoride and igniting a plasma at a power between about 1000W and about 5000W to etch silicon oxide.

[0110] In operation 710, the substrate is exposed to a silicon-containing precursor and an oxidant to form a second amount of silicon oxide by PECVD. This may correspond to operation 610 of Figure 6 as described above. In some embodiments, the silicon-containing precursor used is the same as in operation 702a-1. In some embodiments, the silicon-containing precursor used is different from the silicon-containing precursor used in 702a-1. The selection of a silicon-containing precursor depends on the oxidant used and the technique (ALD, PEALD, CVD, PECVD, etc.) used. The oxidant may also be the same as or different from the oxidant used in operation 702c-1. The second amount of silicon oxide may be used to deposit a helmet on the field surface of the substrate.

[0111] In operation 708b, the silicon oxide may be etched from the feature opening. This may correspond to a repeated operation of operation 608 of Figure 6 as described above. In some embodiments, the helmet deposited in operation 710 protects the material underlying the silicon oxide and the feature profile such that the helmet may be etched while etching the feature opening relative to the interior of the feature. For example, etching the feature opening may involve etching the top 5% or top 10% of the depth of the feature. In some embodiments, due to the high aspect ratio of the features, etching species may not reach the bottoms of the features and thus, only the top up to about 50% of the material deposited in the feature is etched. Such etching may be modulated by using a plasma power between about 1000W and about 5000W, for a duration between 2 seconds and about 200 seconds.

[0112] Operations 702a-2 through 702d-2 may constitute an ALD cycle. Such example shows the repeating of, for example, operation 604 of Figure 6. Operation 702a-2 may be the same as or involve the same precursors and conditions as operation 702a-1, operation 702b-2 may be the same as or involve the same purging conditions as operation 702b-1, operation 702c-2 may be the same as or involve the same oxidant and/or plasma conditions as operation 702c-1, and operation 702d-2 may be the same as or involve the same purging conditions as operation 702d-1. During operation 702a-2, the substrate is exposed to a silicon-containing precursor to adsorb the precursor to the feature surface, which includes silicon oxide previously deposited and/or etched in prior operations. In some embodiments, operations 702a-1, 702b-1, 702c-1, 702d-1, 708a, 710, 708b, 702a-2, 702b-2, 702c-2, and 702d-2 may be optionally repeated until

the features are filled.

[0113] Figures 8A-8D show an example of a substrate 804a having a feature 802a with a re-entrant feature defined by liner 806a and a bottom of the feature 812a undergoing feature fill operations in accordance with certain disclosed embodiments. In Figure 8B, a first amount of silicon oxide 808b is deposited over the substrate to form a helmet. While silicon oxide is depicted as an example, in some embodiments, the helmet is silicon nitride, or silicon oxynitride. Due to the re-entrant profile, a void 810b is formed. This may correspond to deposition in operation 604 of Figure 6. In Figure 8C, the substrate is etched back as shown in the etched silicon oxide 808c. This may correspond to operation 608 of Figure 6. In Figure 8D, the substrate etch back is complete and the feature is opened as shown with the exposed void 810d, with no material loss 816d given the sacrificial helmet that was consumed during etch back. The feature 802d may now be filled with subsequent ALD operations to deposit silicon oxide without having a void, as compared to Figure 3D.

[0114] Figures 9A-9D show an example of a substrate 904a having a feature 902a with a feature profile defined by liner 906a with stubs 916a and a bottom of the feature 912a undergoing feature fill operations in accordance with certain disclosed embodiments. In Figure 9B, a first amount of silicon oxide 908b is deposited over the substrate to form a helmet as shown in the overburden deposited over the field surface of the liner 906b. Due to the sidewall topography, there is space 910b between the sidewalls of deposited silicon oxide. This deposition may correspond to deposition in operation 604 of Figure 6. In Figure 9C, the substrate is etched back as shown in the etched silicon oxide 908c, resulting in smoothed surface 914c. This may correspond to operation 608 of Figure 6. In Figure 9D, the substrate etch back is complete and the feature is opened and the sidewalls at 914d and 916d are both smoothed as shown, with no material loss 916d given the sacrificial helmet that was consumed during etch back. The feature 902d may now be filled with subsequent ALD operations to deposit silicon oxide without having a void, as compared to Figure 4D.

[0115] Figures 9E-9H show an example of a substrate 904e having a feature 902e with a feature profile defined by liner 906e with stubs 916e and a bottom of the feature 912e undergoing feature fill operations in accordance with certain disclosed embodiments. In Figure 9F, a first amount of silicon oxide 908f is deposited into the feature 902f, and a silicon nitride helmet 999f is deposited over the substrate as shown in the overburden deposited over the field surface of the liner 906f. This deposition may correspond to deposition in operations 604 and 607 of Figure 6. Due to the sidewall topography, there is space 910b between the sidewalls of deposited silicon oxide. In Figure 9G, the substrate is etched back as shown in the etched

silicon oxide 908g, resulting in smoothened surface 914g. This may correspond to operation 608 of Figure 6. Note that silicon nitride has etch selectivity to silicon oxide, so much of the silicon oxide remains and the silicon nitride helmet can withstand the etching operation for a longer duration; however since etch selectivity here is not necessarily infinite, some silicon nitride may be removed during the etching process. In Figure 9H, the substrate etch back is complete and the feature is opened and the sidewalls at 914h and 916h are both smoothened as shown, with no material loss 916h given the sacrificial helmet 999h having etch selectivity relative to silicon oxide. Note that in some embodiments, some of the helmet 999h may be removed but not as much as silicon oxide would, given the same etching conditions. The feature 902h may now be filled with subsequent ALD operations to deposit silicon oxide without having a void, as compared to Figure 4D.

[0116] Figures 10A-10C show an example of a substrate with multi-laminate stack having two materials 1004a and 1014a with having a feature 1002a undergoing feature fill operations in accordance with certain disclosed embodiments. A first amount of silicon oxide 1009b is deposited over the substrate to form a helmet. This may correspond to deposition in operation 604 of Figure 6. In Figure 10B, the substrate is etched back as shown in the etched silicon oxide 1008b. This may correspond to operation 608 of Figure 6. In Figure 10C, the feature 1002c is filled with subsequent ALD operations to deposit silicon oxide without causing damage to the feature profile, as compared to Figure 5C.

[0117] Figure 11 is a timing sequence diagram of example pulses in accordance with an example of repeated operations of Figure 6 using a combination of PEALD and PECVD for depositing silicon oxide during deposition operations. Figure 11 shows phases in an example process 1100, for various process parameters, such as carrier gas or purge gas flow, silicon-containing precursor flow, oxidant flow, plasma power, and etchant flow. The lines indicate when the flow or plasma power is turned on and off. Other process parameters not shown in Figure 11 may also be relevant for modulating certain disclosed embodiments; such parameters include, but are not limited to, flow rates for precursor, inert, reactant, and etchant gases, substrate temperature, and process chamber pressure.

[0118] Process 1100 includes ALD cycles 1102-1, etch phase 1108-1, ALD cycle 1110-1, PECVD exposure phase 1110-2, long etch back phase 1108-2, and ALD cycles 1102-2. Although a specific sequence of example operations are shown in Figure 11, it will be understood that this is an example and other variations of ALD, PECVD, and etching may be used for substrates with features of various types and for various materials. Further, while Figure 11 shows silicon precursor and oxidant flows for depositing silicon oxide, it will be

understood that embodiments may be suitable for depositing any desired material such as described elsewhere herein.

[0119] ALD cycles 1102-1 include silicon-containing precursor exposure phase 1102a, purge phase 1102b, oxidant exposure phase 1102c, and purge phase 1102d. In silicon-containing precursor exposure phase 1102a, argon is flowed as a carrier gas with silicon-containing precursor gas flow on, while oxidant and etchant flows are off and the plasma is turned off. In purge phase 1102b, argon gas remains on while silicon-containing gas, oxidant gas, and etchant gas flows are off and plasma is off. In oxidant exposure phase 1102c, oxidant and purge gas flows are on, while plasma is turned on and silicon-containing precursor and etchant gas flows are off. In purge phase 1102d, argon gas is on while silicon-containing gas flow, oxidant gas flow, and etchant gas flows are off and plasma is turned off. While one ALD cycle is depicted, it will be understood that multiple cycles may be performed in some embodiments.

[0120] Etch phase 1108-1 includes one etching phase 1108a whereby argon gas and etchant gas flows are on and plasma is turned on, while silicon-containing precursor and oxidant gas flows are off.

[0121] In purge phase 1153a, argon acts as a purge gas and argon gas flow is turned on, while silicon-containing precursor gas, oxidant gas, and etchant gas flows are off and the plasma is turned off.

[0122] ALD cycles 1110-1 are performed to deposit more silicon oxide material after etching in etch phase 1108-1. ALD cycles 1110-1 include repeating operations from ALD cycles 1102-1 such that ALD cycles 1110-1 includes silicon-containing precursor exposure phase 1102a, purge phase 1102b, oxidant exposure phase 1102c, and purge phase 1102d. In silicon-containing precursor exposure phase 1102a, argon is flowed as a carrier gas with silicon-containing precursor gas flow on, while oxidant and etchant flows are off and the plasma is turned off. In purge phase 1102b, argon gas remains on while silicon-containing gas, oxidant gas, and etchant gas flows are off and plasma is off. In oxidant exposure phase 1102c, oxidant and purge gas flows are on, while plasma is turned on and silicon-containing precursor and etchant gas flows are off. In purge phase 1102d, argon gas is on while silicon-containing gas flow, oxidant gas flow, and etchant gas flows are off and plasma is turned off. While one ALD cycle is depicted, it will be understood that multiple cycles may be performed in some embodiments.

[0123] In this example, a combination of ALD cycles 1110-1 and PECVD exposure 1110-2 are performed to deposit a second amount of silicon oxide after etching in etch phase 1108-1. During PECVD exposure phase 1110-2, only one operation of PECVD exposure 1110 is depicted, whereby argon is flowed, silicon-containing precursor gas is flowed, oxidant gas is

flowed, and a plasma is ignited to deposit silicon oxide. It will be understood that the silicon-containing precursor may be the same as or different from the silicon-containing precursor used in ALD cycles 1102-1 and 1110-1. PECVD exposure 1110-2 may correspond to formation of a helmet on a field surface of a substrate.

5 [0124] In long etch back phase 1108-2, a longer duration of etching 1108b is depicted whereby argon is flowed, etchant gas is flowed, and plasma is ignited to facilitate etching, while silicon-containing precursor and oxidant gas flows are turned off. This may correspond to opening of a feature opening by etching after forming a sacrificial helmet on the substrate.

[0125] Purge phase 1153b involves flowing argon gas, while silicon-containing gas, oxidant gas, and etchant gas flows are turned off and plasma is off. ALD cycles 1102-2 include repeating operations from ALD cycles 1102-1 such that ALD cycles 1110-2 includes silicon-containing precursor exposure phase 1102a, purge phase 1102b, oxidant exposure phase 1102c, and purge phase 1102d. In silicon-containing precursor exposure phase 1102a, argon is flowed as a carrier gas with silicon-containing precursor gas flow on, while oxidant and etchant flows are off and the plasma is turned off. In purge phase 1102b, argon gas remains on while silicon-containing gas, oxidant gas, and etchant gas flows are off and plasma is off. In oxidant exposure phase 1102c, oxidant and purge gas flows are on, while plasma is turned on and silicon-containing precursor and etchant gas flows are off. In purge phase 1102d, argon gas is on while silicon-containing gas flow, oxidant gas flow, and etchant gas flows are off and plasma is turned off. While one ALD cycle is depicted, it will be understood that multiple cycles may be performed in some embodiments.

APPARATUS

[0126] Figure 12 depicts a schematic illustration of an embodiment of an atomic layer deposition (ALD) process station 1200 having a process chamber body 1202 for maintaining a low-pressure environment. Such station may be used for performing certain disclosed embodiments, including deposition by ALD, PEALD, CVD, PECVD, as well as etch back operations. A plurality of ALD process stations 1200 may be included in a common low pressure process tool environment. For example, Figure 13 depicts an embodiment of a multi-station processing tool 1300. In some embodiments, one or more hardware parameters of ALD process station 1200 including those discussed in detail below may be adjusted programmatically by one or more computer controllers 1250.

[0127] ALD process station 1200 fluidly communicates with reactant delivery system 1201a for delivering process gases to a distribution showerhead 1206. Reactant delivery system 1201a

includes a mixing vessel 1204 for blending and/or conditioning process gases, such as an silicon-containing precursor gas, or oxidant gas (e.g., oxygen or nitrous oxide), inert gases, etchant gases (e.g., nitrogen trifluoride) for delivery to showerhead 1206. One or more mixing vessel inlet valves 1220 may control introduction of process gases to mixing vessel 1204.

5 Nitrogen trifluoride and/or oxidant plasma may also be delivered to the showerhead 1206 or may be generated in the ALD process station 1200.

[0128] As an example, the embodiment of Figure 12 includes a vaporization point 1203 for vaporizing liquid reactant to be supplied to the mixing vessel 1204. In some embodiments, vaporization point 1203 may be a heated vaporizer. The saturated reactant vapor produced from
10 such vaporizers may condense in downstream delivery piping. Exposure of incompatible gases to the condensed reactant may create small particles. These small particles may clog piping, impede valve operation, contaminate substrates, etc. Some approaches to addressing these issues involve purging and/or evacuating the delivery piping to remove residual reactant. However, purging the delivery piping may increase process station cycle time, degrading process station
15 throughput. Thus, in some embodiments, delivery piping downstream of vaporization point 1203 may be heat traced. In some examples, mixing vessel 1204 may also be heat traced. In one non-limiting example, piping downstream of vaporization point 1203 has an increasing temperature profile extending from approximately 100°C to approximately 150°C at mixing vessel 1204.

20 [0129] In some embodiments, liquid precursor or liquid reactant may be vaporized at a liquid injector. For example, a liquid injector may inject pulses of a liquid reactant into a carrier gas stream upstream of the mixing vessel. In one embodiment, a liquid injector may vaporize the reactant by flashing the liquid from a higher pressure to a lower pressure. In another example, a liquid injector may atomize the liquid into dispersed microdroplets that are subsequently
25 vaporized in a heated delivery pipe. Smaller droplets may vaporize faster than larger droplets, reducing a delay between liquid injection and complete vaporization. Faster vaporization may reduce a length of piping downstream from vaporization point 1203. In one scenario, a liquid injector may be mounted directly to mixing vessel 1204. In another scenario, a liquid injector may be mounted directly to showerhead 1206.

30 [0130] In some embodiments, a liquid flow controller (LFC) upstream of vaporization point 1203 may be provided for controlling a mass flow of liquid for vaporization and delivery to process station 1200. For example, the LFC may include a thermal mass flow meter (MFM) located downstream of the LFC. A plunger valve of the LFC may then be adjusted responsive to feedback control signals provided by a proportional-integral-derivative (PID) controller in

electrical communication with the MFM. However, it may take one second or more to stabilize liquid flow using feedback control. This may extend a time for dosing a liquid reactant. Thus, in some embodiments, the LFC may be dynamically switched between a feedback control mode and a direct control mode. In some embodiments, this may be performed by disabling a sense tube of the LFC and the PID controller.

[0131] Showerhead 1206 distributes process gases toward substrate 1212. In the embodiment shown in Figure 12, the substrate 1212 is located beneath showerhead 1206 and is shown resting on a pedestal 1208. Showerhead 1206 may have any suitable shape, and may have any suitable number and arrangement of ports for distributing process gases to substrate 1212.

[0132] In some embodiments, pedestal 1208 may be raised or lowered to expose substrate 1212 to a volume between the substrate 1212 and the showerhead 1206. It will be appreciated that, in some embodiments, pedestal height may be adjusted programmatically by a suitable computer controller 1250. In some embodiments, the pedestal is powered and power may be applied to generate a bias to the pedestal.

[0133] In another scenario, adjusting a height of pedestal 1208 may allow a plasma density to be varied during plasma activation cycles in the process in embodiments where a plasma is ignited. At the conclusion of the process phase, pedestal 1208 may be lowered during another substrate transfer phase to allow removal of substrate 1212 from pedestal 1208.

[0134] In some embodiments, pedestal 1208 may be temperature controlled via heater 1210.

In some embodiments, the pedestal 1208 may be heated to a temperature of between about 50°C and about 650°C. In some embodiments, the pedestal is set at a temperature between about 50°C and about 500°C, such as at a temperature between about 200°C and about 275°C. In some embodiments, the pedestal is set at a temperature between about 50°C and about 300°C. In some embodiments, the pedestal is set at a temperature between about 200°C and about 275°C.

[0135] Further, in some embodiments, pressure control for process station 1200 may be provided by butterfly valve 1218. As shown in the embodiment of Figure 12, butterfly valve 1218 throttles a vacuum provided by a downstream vacuum pump (not shown). However, in some embodiments, pressure control of process station 1200 may also be adjusted by varying a flow rate of one or more gases introduced to the process station 1200.

[0136] In some embodiments, a position of showerhead 1206 may be adjusted relative to pedestal 1208 to vary a volume between the substrate 1212 and the showerhead 1206. Further, it will be appreciated that a vertical position of pedestal 1208 and/or showerhead 1206 may be varied by any suitable mechanism within the scope of the present disclosure. In some embodiments, pedestal 1208 may include a rotational axis for rotating an orientation of

substrate 1212. It will be appreciated that, in some embodiments, one or more of these example adjustments may be performed programmatically by one or more suitable computer controllers 1250.

[0137] In some embodiments where plasma may be used as discussed above, showerhead 1206 and pedestal 1208 electrically communicate with a radio frequency (RF) power supply 1214 and matching network 1216 for powering a plasma. In some embodiments, the plasma energy may be controlled by controlling one or more of a process station pressure, a gas concentration, an RF source power, an RF source frequency, and a plasma power pulse timing. For example, RF power supply 1214 and matching network 1216 may be operated at any suitable power to form a plasma having a desired composition of radical species. Examples of suitable powers are included above. Likewise, RF power supply 1214 may provide RF power of any suitable frequency. In some embodiments, RF power supply 1214 may be configured to control high- and low-frequency RF power sources independently of one another. Example low-frequency RF frequencies may include, but are not limited to, frequencies between 0 kHz and 500 kHz. Example high-frequency RF frequencies may include, but are not limited to, frequencies between 1.8 MHz and 2.45 GHz, or greater than about 13.56 MHz, or greater than 27 MHz, or greater than 40 MHz, or greater than 60 MHz. It will be appreciated that any suitable parameters may be modulated discretely or continuously to provide plasma energy for the surface reactions. Plasma power may vary depending on whether the station is performing deposition or etching. For example, example etching plasma powers include 1000W to 5000W.

[0138] In some embodiments, the plasma may be monitored in-situ by one or more plasma monitors. In one scenario, plasma power may be monitored by one or more voltage, current sensors (e.g., VI probes). In another scenario, plasma density and/or process gas concentration may be measured by one or more optical emission spectroscopy sensors (OES). In some embodiments, one or more plasma parameters may be programmatically adjusted based on measurements from such in-situ plasma monitors. For example, an OES sensor may be used in a feedback loop for providing programmatic control of plasma power. It will be appreciated that, in some embodiments, other monitors may be used to monitor the plasma and other process characteristics. Such monitors may include, but are not limited to, infrared (IR) monitors, acoustic monitors, and pressure transducers.

[0139] In some embodiments, instructions for a controller 1250 may be provided via input/output control (IOC) sequencing instructions. In one example, the instructions for setting conditions for a process phase may be included in a corresponding recipe phase of a process recipe. In some cases, process recipe phases may be sequentially arranged, so that all

instructions for a process phase are executed concurrently with that process phase. In some embodiments, instructions for setting one or more reactor parameters may be included in a recipe phase. For example, a first recipe phase may include instructions for setting a flow rate of an inert and/or a reactant gas (e.g., the first precursor such as a silicon-containing precursor),
5 instructions for setting a flow rate of a carrier gas (such as argon), and time delay instructions for the first recipe phase. A second, subsequent recipe phase may include instructions for modulating or stopping a flow rate of an inert and/or a reactant gas, and instructions for modulating a flow rate of a carrier or purge gas and time delay instructions for the second recipe phase. A third recipe phase may include instructions for modulating a flow rate of a second
10 reactant gas such as oxygen, instructions for modulating the flow rate of a carrier or purge gas, and time delay instructions for the third recipe phase. A fourth, subsequent recipe phase may include instructions for modulating or stopping a flow rate of an etchant gas, and instructions for modulating a flow rate of a carrier or purge gas and time delay instructions for the fourth recipe phase. A fifth recipe phase may include instructions for modulating a flow rate of a silicon-
15 containing gas as well as a nitrogen-containing or oxygen-containing gas for depositing a silicon nitride or oxide helmet respectively, instructions for modulating the flow rate of a carrier or purge gas, and time delay instructions for the fifth recipe phase. It will be appreciated that these recipe phases may be further subdivided and/or iterated in any suitable way within the scope of the disclosed embodiments. In some embodiments, the controller 1250 may include any of the
20 features described below with respect to system controller 1350 of Figure 13.

[0140] As described above, one or more process stations may be included in a multi-station processing tool. Figure 13 shows a schematic view of an embodiment of a multi-station processing tool 1300 with an inbound load lock 1302 and an outbound load lock 1304, either or both of which may include a remote plasma source. A robot 1306 at atmospheric pressure is
25 configured to move wafers from a cassette loaded through a pod 1308 into inbound load lock 1302 via an atmospheric port 1310. A wafer is placed by the robot 1306 on a pedestal 1312 in the inbound load lock 1302, the atmospheric port 1310 is closed, and the load lock is pumped down. Where the inbound load lock 1302 includes a remote plasma source, the wafer may be exposed to a remote plasma treatment in the load lock prior to being introduced into a processing
30 chamber 1314. Further, the wafer also may be heated in the inbound load lock 1302 as well, for example, to remove moisture and adsorbed gases. Next, a chamber transport port 1316 to processing chamber 1314 is opened, and another robot (not shown) places the wafer into the reactor on a pedestal of a first station shown in the reactor for processing. While the embodiment depicted in Figure 13 includes load locks, it will be appreciated that, in some

embodiments, direct entry of a wafer into a process station may be provided.

[0141] The depicted processing chamber 1314 includes four process stations, numbered from 1 to 4 in the embodiment shown in Figure 13. Each station has a heated pedestal (shown at 1318 for station 1), and gas line inlets. It will be appreciated that in some embodiments, each process station may have different or multiple purposes. For example, in some embodiments, a process station may be switchable between an ALD and plasma-enhanced ALD process mode. Additionally or alternatively, in some embodiments, processing chamber 1314 may include one or more matched pairs of ALD and plasma-enhanced ALD process stations. While the depicted processing chamber 1314 includes four stations, it will be understood that a processing chamber according to the present disclosure may have any suitable number of stations. For example, in some embodiments, a processing chamber may have five or more stations, while in other embodiments a processing chamber may have three or fewer stations.

[0142] Figure 13 depicts an embodiment of a wafer handling system 1390 for transferring wafers within processing chamber 1314. In some embodiments, wafer handling system 1390 may transfer wafers between various process stations and/or between a process station and a load lock. It will be appreciated that any suitable wafer handling system may be employed. Non-limiting examples include wafer carousels and wafer handling robots. Figure 13 also depicts an embodiment of a system controller 1350 employed to control process conditions and hardware states of process tool 1300. System controller 1350 may include one or more memory devices 1356, one or more mass storage devices 1354, and one or more processors 1352. Processor 1352 may include a CPU or computer, analog, and/or digital input/output connections, stepper motor controller boards, etc.

[0143] In some embodiments, system controller 1350 controls all of the activities of process tool 1300. System controller 1350 executes system control software 1358 stored in mass storage device 1354, loaded into memory device 1356, and executed on processor 1352. Alternatively, the control logic may be hard coded in the controller 1350. Applications Specific Integrated Circuits, Programmable Logic Devices (e.g., field-programmable gate arrays, or FPGAs) and the like may be used for these purposes. In the following discussion, wherever “software” or “code” is used, functionally comparable hard coded logic may be used in its place. System control software 1358 may include instructions for controlling the timing, mixture of gases, gas flow rates, chamber and/or station pressure, chamber and/or station temperature, wafer temperature, target power levels, RF power levels, substrate pedestal, chuck and/or susceptor position, and other parameters of a particular process performed by process tool 1300. System control software 1358 may be configured in any suitable way. For example, various process tool

component subroutines or control objects may be written to control operation of the process tool components used to carry out various process tool processes. System control software 1358 may be coded in any suitable computer readable programming language.

5 [0144] In some embodiments, system control software 1358 may include input/output control (IOC) sequencing instructions for controlling the various parameters described above. Other computer software and/or programs stored on mass storage device 1354 and/or memory device 1356 associated with system controller 1350 may be employed in some embodiments. Examples of programs or sections of programs for this purpose include a substrate positioning program, a process gas control program, a pressure control program, a heater control program, and a plasma control program.

[0145] A substrate positioning program may include program code for process tool components that are used to load the substrate onto pedestal 1318 and to control the spacing between the substrate and other parts of process tool 1300.

15 [0146] A process gas control program may include code for controlling gas composition (e.g., silicon-containing gases, oxidant gases, etchant gases, carrier gases, and purge gases as described herein) and flow rates and optionally for flowing gas into one or more process stations prior to deposition in order to stabilize the pressure in the process station. A pressure control program may include code for controlling the pressure in the process station by regulating, for example, a throttle valve in the exhaust system of the process station, a gas flow into the process station, etc.

20 [0147] A heater control program may include code for controlling the current to a heating unit that is used to heat the substrate. Alternatively, the heater control program may control delivery of a heat transfer gas (such as helium) to the substrate.

[0148] A plasma control program may include code for setting RF power levels applied to the process electrodes in one or more process stations in accordance with the embodiments herein.

25 [0149] A pressure control program may include code for maintaining the pressure in the reaction chamber in accordance with the embodiments herein.

[0150] In some embodiments, there may be a user interface associated with system controller 1350. The user interface may include a display screen, graphical software displays of the apparatus and/or process conditions, and user input devices such as pointing devices, keyboards, touch screens, microphones, etc.

30 [0151] In some embodiments, parameters adjusted by system controller 1350 may relate to process conditions. Non-limiting examples include process gas composition and flow rates, temperature, pressure, plasma conditions (such as RF bias power levels), etc. These parameters may be provided to the user in the form of a recipe, which may be entered utilizing the user

interface.

[0152] Signals for monitoring the process may be provided by analog and/or digital input connections of system controller 1350 from various process tool sensors. The signals for controlling the process may be output on the analog and digital output connections of process tool 1300. Non-limiting examples of process tool sensors that may be monitored include mass flow controllers, pressure sensors (such as manometers), thermocouples, etc. Appropriately programmed feedback and control algorithms may be used with data from these sensors to maintain process conditions.

[0153] System controller 1350 may provide program instructions for implementing the above-described deposition processes. The program instructions may control a variety of process parameters, such as DC power level, RF bias power level, pressure, temperature, etc. The instructions may control the parameters to operate in-situ deposition of film stacks according to various embodiments described herein.

[0154] The system controller 1350 will typically include one or more memory devices and one or more processors configured to execute the instructions so that the apparatus will perform a method in accordance with disclosed embodiments. Machine-readable media containing instructions for controlling process operations in accordance with disclosed embodiments may be coupled to the system controller 1350.

[0155] In some implementations, the system controller 1350 is part of a system, which may be part of the above-described examples. Such systems can include semiconductor processing equipment, including a processing tool or tools, chamber or chambers, a platform or platforms for processing, and/or specific processing components (a wafer pedestal, a gas flow system, etc.). These systems may be integrated with electronics for controlling their operation before, during, and after processing of a semiconductor wafer or substrate. The electronics may be referred to as the “controller,” which may control various components or subparts of the system or systems. The system controller 1350, depending on the processing conditions and/or the type of system, may be programmed to control any of the processes disclosed herein, including the delivery of processing gases, temperature settings (e.g., heating and/or cooling), pressure settings, vacuum settings, power settings, radio frequency (RF) generator settings, RF matching circuit settings, frequency settings, flow rate settings, fluid delivery settings, positional and operation settings, wafer transfers into and out of a tool and other transfer tools and/or load locks connected to or interfaced with a specific system.

[0156] Broadly speaking, the system controller 1350 may be defined as electronics having various integrated circuits, logic, memory, and/or software that receive instructions, issue

instructions, control operation, enable cleaning operations, enable endpoint measurements, and the like. The integrated circuits may include chips in the form of firmware that store program instructions, digital signal processors (DSPs), chips defined as application specific integrated circuits (ASICs), and/or one or more microprocessors, or microcontrollers that execute program instructions (e.g., software). Program instructions may be instructions communicated to the system controller 1350 in the form of various individual settings (or program files), defining operational parameters for carrying out a particular process on or for a semiconductor wafer or to a system. The operational parameters may, in some embodiments, be part of a recipe defined by process engineers to accomplish one or more processing steps during the fabrication of one or more layers, materials, metals, oxides, silicon, silicon dioxide, surfaces, circuits, and/or dies of a wafer.

[0157] The system controller 1350, in some implementations, may be a part of or coupled to a computer that is integrated with, coupled to the system, otherwise networked to the system, or a combination thereof. For example, the system controller 1350 may be in the “cloud” or all or a part of a fab host computer system, which can allow for remote access of the wafer processing. The computer may enable remote access to the system to monitor current progress of fabrication operations, examine a history of past fabrication operations, examine trends or performance metrics from a plurality of fabrication operations, to change parameters of current processing, to set processing steps to follow a current processing, or to start a new process. In some examples, a remote computer (e.g. a server) can provide process recipes to a system over a network, which may include a local network or the Internet. The remote computer may include a user interface that enables entry or programming of parameters and/or settings, which are then communicated to the system from the remote computer. In some examples, the system controller 1350 receives instructions in the form of data, which specify parameters for each of the processing steps to be performed during one or more operations. It should be understood that the parameters may be specific to the type of process to be performed and the type of tool that the system controller 1350 is configured to interface with or control. Thus as described above, the system controller 1350 may be distributed, such as by including one or more discrete controllers that are networked together and working towards a common purpose, such as the processes and controls described herein. An example of a distributed controller for such purposes would be one or more integrated circuits on a chamber in communication with one or more integrated circuits located remotely (such as at the platform level or as part of a remote computer) that combine to control a process on the chamber.

[0158] Without limitation, example systems may include a plasma etch chamber or module, a

deposition chamber or module, a spin-rinse chamber or module, a metal plating chamber or module, a clean chamber or module, a bevel edge etch chamber or module, a physical vapor deposition (PVD) chamber or module, a chemical vapor deposition (CVD) chamber or module, an ALD chamber or module, an atomic layer etch (ALE) chamber or module, an ion
5 implantation chamber or module, a track chamber or module, and any other semiconductor processing systems that may be associated or used in the fabrication and/or manufacturing of semiconductor wafers.

[0159] As noted above, depending on the process step or steps to be performed by the tool, the system controller 1350 might communicate with one or more of other tool circuits or modules,
10 other tool components, cluster tools, other tool interfaces, adjacent tools, neighboring tools, tools located throughout a factory, a main computer, another controller, or tools used in material transport that bring containers of wafers to and from tool locations and/or load ports in a semiconductor manufacturing factory.

[0160] An appropriate apparatus for performing the methods disclosed herein is further
15 discussed and described in U.S. Patent Application Nos. 13/084,399 (now U.S. Patent No. 8,728,956), filed April 11, 2011, and titled “PLASMA ACTIVATED CONFORMAL FILM DEPOSITION”; and 13/084,305, filed April 11, 2011, and titled “SILICON NITRIDE FILMS AND METHODS,” each of which is incorporated herein in its entirety.

[0161] The apparatus/process described herein may be used in conjunction with lithographic
20 patterning tools or processes, for example, for the fabrication or manufacture of semiconductor devices, displays, LEDs, photovoltaic panels and the like. Typically, though not necessarily, such tools/processes will be used or conducted together in a common fabrication facility. Lithographic patterning of a film typically includes some or all of the following operations, each operation enabled with a number of possible tools: (1) application of photoresist on a workpiece,
25 i.e., substrate, using a spin-on or spray-on tool; (2) curing of photoresist using a hot plate or furnace or UV curing tool; (3) exposing the photoresist to visible or UV or x-ray light with a tool such as a wafer stepper; (4) developing the resist so as to selectively remove resist and thereby pattern it using a tool such as a wet bench; (5) transferring the resist pattern into an underlying film or workpiece by using a dry or plasma-assisted etching tool; and (6) removing the resist
30 using a tool such as an RF or microwave plasma resist stripper.

EXPERIMENTAL

EXPERIMENT 1

[0162] An experiment was conducted on a substrate with a 15:1 feature having a depth of 5

microns with recessed tungsten and silicon oxide stubs and re-entrancy at the top of the feature near the feature opening. Conventional atomic layer deposition of silicon oxide over such a substrate performed on the substrate, which resulted in voids where the oxide stubs come together on the sidewalls. Re-entrancy at the top of the feature also resulted in top voids.

- 5 Another similar substrate was exposed to 300 cycles of ALD, etch back using NF_3 , multiple cycles of ALD, deposition of a helmet using PECVD for 60 seconds, a long etch back, and ALD deposition to fill the features. The substrate resulted in no voids, and no removal of material from the corners at the feature opening. These results suggested viability of certain disclosed dep-etch-dep operations used for void-free gapfill of dielectric material in high aspect ratio
- 10 features with sidewall topography.

EXPERIMENT 2

- [0163] An experiment was conducted on a substrate with recessed features having a depth of 160nm where the surface of the recessed features was silicon nitride where the feature opening at the top of the feature is 25nm and the sidewalls narrow depth wise to the bottom of the feature.
- 15 Silicon oxide was deposited into the features using 40 cycles of ALD using an aminosilane precursor and $\text{N}_2\text{O}/\text{O}_2$ with plasma. Following ALD, a silicon oxide helmet was deposited by PECVD to a thickness of 125Å. After helmet deposition, the substrate was etched using NF_3 for a duration of 32 seconds. The helmet was consumed entirely during the etch. Subsequent ALD was performed to complete fill of the features. The feature was filled with little to no void
- 20 formation.

- [0164] An experiment was conducted on a substrate with recessed features having a depth of 160nm where the surface of the recessed features was silicon nitride where the feature opening at the top of the feature is 25nm and the sidewalls narrow depth wise to the bottom of the feature. Silicon oxide was deposited into the features using 40 cycles of ALD using an aminosilane
- 25 precursor and $\text{N}_2\text{O}/\text{O}_2$ with plasma. Following ALD, a silicon nitride helmet was deposited by PECVD using an aminosilane precursor and a nitrogen plasma to a thickness of 125Å. After helmet deposition, the substrate was etched using NF_3 for a duration of 32 seconds. Greater than 50% of the helmet remained after etch. Subsequent ALD was performed to complete fill of the features. Etch selectivity of the silicon nitride helmet to the silicon oxide deposited by ALD
- 30 allowed for longer etch duration to open the feature.

[0165] An experiment was conducted on a substrate with recessed features having a depth of 160nm where the surface of the recessed features was silicon nitride where the feature opening at the top of the feature is 25nm and the sidewalls narrow depth wise to the bottom of the feature.

Silicon oxide was deposited into the features using 40 cycles of ALD using an aminosilane precursor and $\text{N}_2\text{O}/\text{O}_2$ with plasma. Following ALD, a silicon nitride helmet was deposited by PECVD using an aminosilane precursor and a nitrogen plasma to a thickness of 65Å. After helmet deposition, the substrate was etched using NF_3 for a duration of 32 seconds. The helmet was consumed entirely during the etch. Subsequent ALD was performed to complete fill of the features. Due to the etch selectivity of the silicon nitride helmet to the silicon oxide deposited by ALD, a thinner helmet was able to be used and still achieve void-free feature fill.

CONCLUSION

[0166] Although the foregoing embodiments have been described in some detail for purposes of clarity of understanding, it will be apparent that certain changes and modifications may be practiced within the scope of the appended claims. It should be noted that there are many alternative ways of implementing the processes, systems, and apparatus of the present embodiments. Accordingly, the present embodiments are to be considered as illustrative and not restrictive, and the embodiments are not to be limited to the details given herein.

CLAIMS

1. A method of filling a feature on a substrate, the method comprising:
5 providing the substrate comprising the feature to a process chamber, the feature comprising a feature opening and sidewall topography, the sidewall topography comprising stubs on the sidewall of the feature;
depositing a first amount of silicon oxide for a duration insufficient to fill the feature using a silicon-containing precursor and an oxidant;
10 exposing the first amount of the silicon oxide to an etchant to etch at least some of the first amount of the silicon oxide; and
after etching the first amount of the silicon oxide, depositing a second amount of the silicon oxide over the etched first amount of the silicon oxide.
2. The method of claim 1, wherein the stubs have a dimension perpendicular to a planar
15 surface of the sidewall of between 100 Å and about 300 Å.
3. The method of claim 1, wherein the duration insufficient to deposit the first amount of silicon oxide into the feature forms an overburden of silicon oxide on a field surface of the substrate.
4. The method of claim 3, wherein exposing the first amount of the silicon oxide to the
20 etchant comprises etching the at least some of the first amount of the silicon oxide at or near the feature opening relative to the inside of the feature.
5. The method of claim 1, wherein the second amount of the silicon oxide is deposited by plasma-enhanced chemical vapor deposition.
6. The method of claim 5, further comprising after depositing the second amount of the
25 silicon oxide, exposing the second amount of the silicon oxide to the etchant for a duration longer than the duration used for exposing the first amount of the silicon oxide to the etchant.
7. The method of claim 1, wherein the sidewalls of the feature comprise two or more materials layered in a stack.

8. The method of claim 1, wherein the depositing of the first amount of the silicon oxide and the exposing of the first amount of the silicon oxide to the etchant are performed without breaking vacuum.
9. The method of claim 1, wherein the exposing of the first amount of the silicon oxide to the etchant and the depositing of the second amount of the silicon oxide are performed without breaking vacuum.
10. The method of any of claims 1-9, wherein the first amount of the silicon oxide is deposited by one or more cycles of atomic layer deposition, each cycle of atomic layer deposition comprising alternating pulses of the oxidant and the silicon-containing precursor.
11. The method of claim 10, wherein a plasma is ignited during pulses of the oxidant.
12. The method of any of claims 1-9, wherein the etchant is selected from the group consisting of nitrogen trifluoride, fluoroform (CHF_3), octafluorocyclobutane (C_4F_8), tetrafluoromethane (CF_4), and combinations thereof.
13. The method of any of claims 1-9, wherein the feature has a depth of at least 5 microns.
14. The method of any of claims 1-9, wherein the feature has an aspect ratio of at least 15:1.
15. A method of filling a feature on a substrate, the method comprising:
 - providing the substrate comprising the feature to a process chamber, the feature comprising a feature opening and sidewall topography, the sidewall topography comprising stubs on the sidewall of the feature;
 - depositing a first amount of silicon oxide for a duration insufficient to fill the feature using a silicon-containing precursor and an oxidant;
 - after depositing the first amount of silicon oxide and prior to exposing the first amount of silicon oxide to an etchant, depositing a sacrificial helmet forming an overburden on a field surface of the substrate;
 - exposing the substrate to the etchant to etch at least some of the first amount of the silicon oxide; and
 - after etching the first amount of the silicon oxide, depositing a second amount of the oxide over the etched first amount of the silicon oxide to at least partially fill the feature.

16. The method of claim 15, wherein sacrificial helmet is deposited by plasma enhanced chemical vapor deposition.
17. The method of any of claims 15 and 16, wherein the sacrificial helmet comprises silicon nitride.
- 5 18. The method of any of claims 15 and 16, wherein the sacrificial helmet comprises silicon oxide.
19. A method of filling a feature on a substrate, the method comprising:
providing the substrate comprising the feature to a process chamber, the feature
comprising a feature opening and sidewalls having one or more reentrant surfaces;
10 depositing a first amount of a material for a duration insufficient to fill the feature;
exposing the first amount of the material to an etchant to etch at least some of the first
amount of the material within the feature; and
after etching the first amount of the material, depositing a second amount of the material
over the etched first amount of the material,
15 wherein the material is selected from the group consisting of silicon carbide, silicon
nitride, silicon, tungsten, ruthenium, copper, cobalt, and molybdenum.
20. The method of claim 19, wherein the duration sufficient to deposit the first amount of the material into the feature forms an overburden of the material on a field surface of the substrate.
- 20 21. The method of claim 20, wherein exposing the first amount of the material to the etchant comprises etching the at least some of the first amount of the material at or near the feature opening relative to the inside of the feature.
22. The method of claim 21, wherein the second amount of the material is deposited by plasma-enhanced chemical vapor deposition.
- 25 23. The method of claim 22, further comprising after depositing the second amount of the material, exposing the second amount of the material to the etchant for a duration longer than the duration used for exposing the first amount of the material to the etchant.
24. The method of any of claims 19-23, wherein the sidewalls of the feature comprise two or more materials layered in a stack.

25. The method of any of claims 19-23, wherein the depositing of the first amount of the material and the exposing of the first amount of the material to the etchant are performed without breaking vacuum.

26. The method of any of claims 19-23, wherein the exposing of the first amount of the material to the etchant and the depositing of the second amount of the material are performed without breaking vacuum.

27. A method of filling a feature on a substrate, the method comprising:

providing the substrate comprising the feature to a process chamber, the feature

comprising a feature opening and sidewall topography, the sidewall topography

comprising stubs on the sidewall of the feature;

depositing a first amount of a first material for a duration insufficient to fill the feature;

after depositing the first amount of a first material and prior to exposing the first amount

of the first material to an etchant, depositing a sacrificial helmet forming an

overburden on a field surface of the substrate, the sacrificial helmet comprising a

second material;

exposing the substrate to the etchant to etch at least some of the first amount of the first material; and

after etching the first amount of the first material, depositing a second amount of the

material over the etched first amount of the first material to at least partially fill the

feature.

28. The method of claim 27, wherein the first material is different from the second material.

29. The method of claim 27, wherein the first material is compositionally the same as the second material.

30. An apparatus for processing a semiconductor substrate, the apparatus comprising:

(a) at least one process chamber, the at least one process chamber comprising a pedestal for holding the semiconductor substrate;

(b) at least one outlet for coupling to a vacuum;

(c) one or more process gas inlets coupled to one or more process gas sources; and

(d) a controller for controlling operations in the apparatus, comprising machine-readable instructions for:

(i) causing introduction of a silicon-containing precursor and an oxidant to deposit a

first amount of silicon oxide on the semiconductor substrate for a duration insufficient to fill a feature on the semiconductor substrate, the feature having a feature opening and sidewall topography, the sidewall topography having stubs on the sidewall of the feature;

- 5 (ii) causing introduction of an etchant to the at least one process chamber for a duration to etch at least some of the first amount of the silicon oxide; and
- (iii) after causing introduction of the etchant to the at least one process chamber, causing introduction of the silicon-containing precursor and the oxidant to deposit a second amount of silicon oxide over the etched first amount of the
- 10 silicon oxide.

31. The apparatus of claim 30, wherein the controller further comprises machine-readable instructions for causing the duration of (iii) to be longer than the duration of (i).

32. The apparatus of claim 30, wherein the controller further comprises machine-readable instructions for causing performance of (i) and (ii) without breaking vacuum.

15 33. The apparatus of claim 30, further comprising a plasma generator for generating a plasma.

34. The apparatus of claim 30, wherein the controller further comprises instructions for causing generation of a plasma when the oxidant is introduced.

35. An apparatus for processing a semiconductor substrate, the apparatus comprising:

- 20 (a) at least one process chamber, the at least one process chamber comprising a pedestal for holding the semiconductor substrate;
- (b) at least one outlet for coupling to a vacuum;
- (c) one or more process gas inlets coupled to one or more process gas sources; and
- (d) a controller for controlling operations in the apparatus, comprising machine-readable
- 25 instructions for:

- (i) causing introduction of a silicon-containing precursor and an oxidant to deposit a first amount of silicon oxide on the semiconductor substrate for a duration insufficient to fill a feature on the semiconductor substrate, the feature having a feature opening and sidewall topography, the sidewall topography having stubs
- 30 on the sidewall of the feature;
- (ii) causing introduction of one or more process gases for depositing a sacrificial

helmet forming an overburden on a field surface of the semiconductor substrate;

(iii) causing introduction of an etchant to the at least one process chamber for a duration to etch at least some of the first amount of the silicon oxide; and

(iv) after causing introduction of the etchant to the at least one process chamber, causing introduction of the silicon-containing precursor and the oxidant to deposit a second amount of silicon oxide over the etched first amount of the silicon oxide.

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36. The apparatus of claim 35, wherein the controller includes instructions for causing delivery of a second silicon-containing precursor and a nitrogen-containing reactant during (ii) to deposit the sacrificial helmet, the sacrificial helmet comprising silicon nitride.

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