

THIS SHEET HAS BEEN REPLACED

1 A Ti/TiN/TiN_x UNDERLAYER WHICH ENABLES
2 A HIGHLY {111} ORIENTED ALUMINUM INTERCONNECT

3 **BACKGROUND OF THE INVENTION**

4 1. Field of the Invention

5 The present invention pertains to a particular (Ti or TiN_x)/TiN/TiN_x
6 barrier/wetting layer structure which enables the warm aluminum filling of high aspect
7 ratio vias while providing an aluminum fill exhibiting a high degree of {111} crystal
8 orientation aluminum.

9 2. Brief Description of the Background Art

10 Titanium nitride layers have been used in semiconductor device structures as
11 barrier layers for preventing the interdiffusion of adjacent layers of materials such as
12 aluminum and silicon, for example. However, the crystal orientation of aluminum
13 deposited over the surface of the titanium nitride barrier layer is typically
14 polycrystalline, and polycrystalline aluminum has poor electromigration resistance.

15 In the formation of integrated circuit interconnect structures, such as a
16 Ti/TiN/TiN_x stack, electromigration of aluminum atoms within the aluminum layer
17 becomes a problem if the aluminum layer is not formed with a high degree of {111}
18 crystal orientation. Electromigration of the aluminum atoms can result in open
19 circuits within the integrated circuit structure, and therefore, such electromigration
20 must be inhibited or eliminated. Electromigration of aluminum atoms can occur
21 within filled vias as well, impairing the conductivity of the contacts.

22 U.S. Patent No. 4,944,961 to Lu et al., issued July 31, 1990, describes a
23 process for partially ionized beam deposition of metals or metal alloys on substrates,
24 such as semiconductor wafers. Metal vaporized from a crucible is partially ionized at



1 the crucible exit, and the ionized vapor is drawn to the substrate by an imposed bias.
2 Control of substrate temperature is said to allow non-conformal coverage of stepped
3 surfaces such as trenches or vias. When higher temperatures are used, stepped
4 surfaces are planarized. The examples given are for aluminum deposition, where the
5 non-conformal deposition is carried out with substrate temperatures ranging between
6 about 150 °C and about 200 °C, and the planarized deposition is carried out with
7 substrate temperatures ranging between about 250 °C and about 350 °C.

8 S.M. Rossnagel and J. Hopwood describe a technique of combining
9 conventional magnetron sputtering with a high density, inductively coupled RF plasma
10 in the region between the sputtering cathode and the substrate in their 1993 article
11 titled "Metal ion deposition from ionized magnetron sputtering discharge", published
12 in the J. Vac. Sci. Technol. B. Vol. 12, No. 1, Jan/Feb 1994. One of the examples
13 given is for titanium nitride film deposition using reactive sputtering, where a titanium
14 cathode is used in combination with a plasma formed from a combination of argon
15 and nitrogen gases.

16 U.S. Patent No. 5,262,361 to Cho et al., issued November 16, 1993 describes a
17 method for forming single crystal aluminum films on the surface of a substrate such
18 as silicon (111). The object is to increase the amount of the aluminum (111) crystal
19 orientation, to improve the electromigration resistance of the aluminum. Electrically
20 neutral aluminum is deposited by a vacuum evaporation technique upon a silicon
21 wafer surface at a temperature ranging between about 300 °C and about 400 °C.

22 U.S. Patent No. 5,543,357 to Yamada et al., issued August 6, 1996, describes a
23 process for manufacturing a semiconductor device wherein a titanium film is used as
24 an under film for an aluminum alloy film to prevent the device characteristics of the
25 aluminum alloy film from deteriorating. The thickness of the titanium film is set to
26 10 % or less of the thickness of the aluminum alloy film and at most 25 nm. In the
27 case of the aluminum alloy film containing no silicon, the titanium film is set to 5%



1 of less of the thickness of the aluminum alloy film. The aluminum film is formed at
2 a substrate temperature of 200 °C or less by a sputtering process, and when the
3 aluminum film or an aluminum alloy film is to fill a via hole, the substrate is heated
4 to fluidize the aluminum. The pressure during the aluminum film formation and
5 during the fluidization is lower than 10^{-7} Torr. A titanium nitride barrier layer may be
6 applied on an interlayered insulating film (or over a titanium layer which has been
7 applied to the insulating film), followed by formation of a titanium film over the
8 titanium nitride film, and finally by formation of the aluminum film over the titanium
9 film. After formation of the titanium nitride barrier layer, the barrier layer is heated
10 to a temperature of about 600 °C to 700 °C in a nitrogen atmosphere using a halogen
11 lamp so that any titanium which is not nitrated will become nitrated. The titanium
12 nitride barrier layer is said to be a poor barrier layer if un-nitrated titanium is present
13 within the layer.

14 U.S. Patent No. 5,571,752 to Chen et al., issued November 5, 1996, discloses a
15 method for patterning a submicron semiconductor layer of an integrated circuit. In
16 one embodiment, titanium or titanium nitride having a thickness of between
17 approximately 300 and 2000 Å is formed by sputter deposition to reach the bottom of
18 a contact opening. The barrier layer may be annealed to form a silicide in the bottom
19 of the opening. A conformal conductive layer of a refractory metal or refractory
20 metal silicide is formed over the titanium or titanium nitride using chemical vapor
21 deposition (CVD). Finally, a second conductive layer, typically aluminum is applied
22 over the surface of the conformal conductive layer. The aluminum is sputtered on,
23 preferably at a temperature ranging between approximately 100 °C and 400 °C. This
24 method is said to make possible the filling of contact openings having smaller device
25 geometry design requirements by avoiding the formation of fairly large grain sizes in
26 the aluminum film.

27 U.S. Patent Application, Serial No. 08/511,825 of Xu et al., filed August 7,



1 1995, assigned to the Assignee of the present invention, and hereby incorporated by
2 reference in its entirety, describes a method of forming a titanium nitride-comprising
3 barrier layer which acts as a carrier layer. The carrier layer enables the filling of
4 apertures such as vias, holes or trenches of high aspect ratio and the planarization of a
5 conductive film deposited over the carrier layer at reduced temperatures compared to
6 prior art methods.

7 U.S. Patent Application, Serial No. 08/753,251 of Ngan et al., filed November
8 21, 1996, describes a method for producing a titanium nitride-comprising barrier layer
9 on the surface of a contact via. For certain contact geometries, when the reactor
10 pressure is reduced during formation of the titanium nitride-comprising barrier layer,
11 the thickness of the barrier layer on the sidewalls of the via increases. This enables
12 an aluminum fill to travel along the sidewalls of the via more easily, resulting in a
13 better fill of the via. In particular, the titanium nitride comprising barrier layer needs
14 to be of a minimum thickness and to have a minimum titanium content so that the
15 barrier layer can react slightly with the Aluminum, to draw the aluminum along the
16 sidewalls of the via.

17 U.S. Patent Application, Serial No. _____ (Attorney Docket No. 1819)
18 of Ngan et al., filed March __, 1997, discloses various process techniques which can
19 be used to control the crystal orientation of a titanium nitride barrier layer as it is
20 deposited. Further, by increasing the {200} crystal orientation of the titanium nitride
21 barrier layer, the resistivity of this layer is decreased.

22 A "traditionally sputtered" titanium nitride-comprising film or layer is
23 deposited on a substrate by contacting a titanium target with a plasma created from an
24 inert gas such as argon in combination with nitrogen gas. A portion of the titanium
25 sputtered from the target reacts with nitrogen gas which has been activated by the
26 plasma to produce titanium nitride, and the gas phase mixture contacts the substrate to
27 form a layer on the substrate. Although such a traditionally sputtered titanium nitride-



1 comprising layer can act as a wetting layer for hot aluminum fill of contact vias, good
2 fill of the via generally is not achieved at substrate surface temperature of less than
3 about 500 °C.

4 To provide for aluminum fill at a lower temperature, Xu et al. (as described in
5 U.S. Patent Application, Serial No. 08/511,825), developed a technique for creating a
6 titanium nitride-comprising barrier layer which can act as a smooth carrier layer,
7 enabling aluminum to flow over the barrier layer surface at lower temperatures (at
8 temperatures as low as about 350 °C, for example). A typical barrier layer described
9 by Xu et al., is a combination of three layers including a first layer of titanium (Ti)
10 deposited over the surface of the via; a second layer of titanium nitride (TiN) is
11 deposited over the surface of the first titanium layer; finally a layer of TiN_x is
12 deposited over the TiN second layer. The three layers are deposited using Ion Metal
13 Plasma (IMP) techniques which are described subsequently herein. Typically the first
14 layer of titanium is approximately 100 Å to 200 Å thick; the second layer of TiN is
15 about 800 Å thick, and the third layer of TiN_x is about 60 Å thick. Although a good
16 fill of contact vias having 0.25 μ diameter through holes having an aspect ratio of
17 about 5 was achieved, the crystal orientation of the aluminum was low in {111}
18 content, resulting in poor electromigration (EM) performance for the aluminum
19 interconnect. Further, the reflectivity of the aluminum, measured by nanoscope, with
20 Si as a reference, was less than about 50 %, so that subsequent lithography indexing
21 was more difficult. It is then desirable to increase the aluminum {111} content for
22 purposes of improving the EM performance and benefiting subsequent lithography
23 process steps.

24 SUMMARY OF THE INVENTION

25 It has been discovered that an improved (Ti or TiN_x)/TiN/ TiN_x barrier layer
26 deposited using IMP techniques can be obtained by increasing the thickness of the
27



1 first layer of Ti or TiN_x to range from greater than about 100 Å to about 500 Å (the
2 feature geometry controls the upper thickness limit); by decreasing the thickness of the
3 TiN second layer to range from greater than about 100 Å to less than about 800 Å
4 (preferably less than about 600 Å); and, by controlling the application of the TiN_x
5 third layer to provide a Ti content ranging from about 50 atomic percent titanium
6 (stoichiometric) to about 100 atomic percent titanium. When the first layer is TiN_x ,
7 the atomic percent of Ti is at least 40 atomic percent. Preferably, the first layer is
8 100 atomic percent Ti. In addition, it is preferred that the TiN_x third layer is formed
9 at the end of the deposition of the TiN second layer and exhibits a Ti content gradient
10 which begins at a stoichiometric Ti content and ends at a Ti content of about 100
11 atomic percent. The thickness of the TiN_x third layer preferably ranges from about 15
12 Å to about 500 Å, with the thickness of the 100 atomic percent Ti component ranging
13 from about 15 Å to about 300 Å. The improved (Ti or TiN_x)/TiN/ TiN_x barrier layer
14 enables the deposit of an aluminum interconnect or an aluminum via fill where the
15 aluminum exhibits a high {111} crystal orientation. Further, the aluminum layer
16 obtained exhibits a reflectivity of 150 percent or greater at 436 nm. A (Ti or
17 TiN_x)/TiN/ TiN_x barrier layer having this structure, used to line a feature, enables
18 complete filling of feature with sputtered aluminum, where the size of the feature is
19 about 0.25 micron and the aspect ratio is as high as about 6 : 1.

20 The TiN_x third layer is deposited at a substrate temperature of ranging from
21 about 50 °C to about 500 °C, preferably at about 200 °C. When the device structure
22 is an interconnect, the TiN_x third layer can be deposited at pressures ranging from
23 about 5 mT to about 40 mT, preferably at about 25 mT. When the device feature is a
24 via, the TiN_x third layer should be deposited at reduced pressures ranging from about
25 5 mT to about 10 mT, preferably at about 10 mT. The aluminum fill is then
26 deposited at a substrate temperature ranging from about 350 °C to about 500 °C,
27 preferably at about 400 °C. The aluminum is deposited at reduced pressures ranging



1 from about 1 mT to about 4 mT, preferably at about 2 mT.

2 BRIEF DESCRIPTION OF THE DRAWINGS

3 Figure 1 shows a schematic of the elements of a process chamber which enable
4 physical vapor deposition augmented by an ion metal plasma (IMP).

5 Figure 2 illustrates a schematic of a conductive contact 218 formed within a
6 high aspect ratio via 213 using the methods and the apparatus described in the present
7 disclosure.

8 Figure 3A shows the X-ray diffraction (XRD) curve for an aluminum film
9 deposited over a Ti/TiN/TiN_x barrier layer of the kind described with reference to
10 U.S. Patent Application, Serial No. 08/511,825, of Xu et al. This aluminum film
11 exhibits a lower {111} crystal orientation content than desired and exhibits a
12 reflectivity of less than about 50 % (measured by nanoscope with Si as a reference).

13 Figure 3B shows the XRD curve for an aluminum film deposited over a
14 Ti/TiN/TiN_x barrier layer created using a first preferred embodiment method described
15 herein. This aluminum film exhibits a high {111} content and exhibits a reflectivity
16 of about 195%.

17 Figure 3C shows the XRD curve for an aluminum film deposited over a
18 Ti/TiN/TiN_x barrier layer using the preferred embodiment method used to create the
19 barrier layer illustrated in Figure 3B, except that the substrate temperature during
20 deposition of the barrier layer was substantially higher. This aluminum film exhibits a
21 lower {111} content than desired and exhibits a reflectivity of less than about 50 %



1 Figure 3D shows the XRD curve for an aluminum film deposited over a
2 Ti/TiN/TiN_x barrier layer created using a second preferred embodiment method
3 described herein. This aluminum film exhibits a high {111} content and exhibits a
4 reflectivity of about 210%.

5
6 Figure 4 is a bar graph showing the relative reflectivities for aluminum films
7 deposited over a Ti/TiN/TiN_x barrier layer having the structure and fabricated using
8 the first and second preferred embodiment methods disclosed herein, in comparison
9 with the reflectivities for aluminum films deposited over a Ti/TiN/TiN_x barrier layer
10 having prior art structures and fabricated using prior art methods.

11 Figure 5A shows an XRD curve for an aluminum film deposited over a
12 Ti/TiN/TiN_x barrier layer having a structure and fabricated using a third preferred
13 embodiment method disclosed herein. This aluminum film exhibits an AFM
14 roughness (rms) of about 78 Å.

15 Figure 5B shows an XRD curve for an aluminum film deposited over a
16 Ti/TiN/TiN_x barrier layer having a structure and fabricated using a fourth preferred
17 embodiment method disclosed herein. This aluminum film exhibits an AFM
18 roughness of about 47 Å.

19
20 Figure 6A illustrates an atomic composition profile for a Ti/TiN/ TiN_x barrier
21 layer having a cross-sectional thickness profile of 100 Å Ti/ 250 Å TiN/ 100 Å TiN_x.

22 Figure 6B illustrates an atomic composition profile for a Ti/TiN/ TiN_x barrier
23 layer having a cross-sectional thickness profile of 200 Å Ti/ 500 Å TiN/ 100 Å TiN_x.



1 Figure 6C illustrates an atomic composition profile for a Ti/TiN/ TiN_x barrier
2 layer having a cross-sectional thickness profile of 200 Å Ti/ 250 Å TiN/ 250 Å TiN_x.

3 DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

4 The present disclosure pertains to a Ti/TiN/TiN_x barrier layer structure and a
5 method of creating that structure. The Ti/TiN/TiN_x barrier layer structure enables the
6 deposition of an overlying aluminum layer having a high {111} crystallographic
7 content and a reflectivity of about 150 % or greater. Further, using the barrier layer
8 structure enables the "warm aluminum" filling of vias having an aperture of 0.25
9 micron or less and having an aspect ratio of about 5 : 1 and greater.

10 I. DEFINITIONS

11 As a preface to the detailed description, it should be noted that, as used in this
12 specification and the appended claims, the singular forms "a", "an", and "the" include
13 plural referents, unless the context clearly dictates otherwise. Thus, for example, the
14 term "a semiconductor" includes a variety of different materials which are known to
15 have the behavioral characteristics of a semiconductor, reference to a "plasma"
16 includes a gas or gas reactants activated by an RF glow discharge, reference to "the
17 contact material" includes aluminum, aluminum alloys, and other conductive materials
18 which have a melting point enabling them to be sputtered over the temperature range
19 described herein.

20 Specific terminology of particular importance to the description of the present
21 invention is defined below.

22 The term "AFM" (Atomic Force Microscope) refers to a technique commonly
23 used to measure film surface roughness, wherein a microprobe in contact with the film
24 surface is drawn across the film and the mechanical movement of the microprobe is
25 translated into a digital signal which is plotted out. A series of plots is compiled and



1 a surface roughness is calculated from the compilation.

2 The term "aluminum" includes alloys of aluminum of the kind typically used
3 in the semiconductor industry. Such alloys include aluminum-copper alloys, and
4 aluminum-copper-silicon alloys, for example. The preferred embodiments described
5 herein were for aluminum comprising about 0.5 % copper.

6 The term "aspect ratio" refers to the ratio of the height dimension to the width
7 dimension of particular openings into which an electrical contact is to be placed. For
8 example, a via opening which typically extends in a tubular form through multiple
9 layers has a height and a diameter, and the aspect ratio would be the height of the
10 tubular divided by the diameter. The aspect ratio of a trench would be the height of
11 the trench divided by the minimal travel width of the trench at its base.

12 The term "feature" refers to contacts, vias, trenches, and other structures which
13 make up the topography of the substrate surface.

14 The term "ion-deposition sputtered" and the term "ion metal plasma (IMP)"
15 refer to sputter deposition, preferably magnetron sputter deposition (where a magnet
16 array is placed behind the target). A high density, inductively coupled RF plasma is
17 positioned between the sputtering cathode and the substrate support electrode, whereby
18 at least a portion of the sputtered emission is in the form of ions at the time it reaches
19 the substrate surface.

20 The term "reactive ion deposition" or "reactive ion metal plasma (IMP)" refers
21 to ion-deposition sputtering wherein a reactive gas is supplied during the sputtering to
22 react with the ionized material being sputtered, producing an ion-deposition sputtered
23 compound containing the reactive gas element.

24 The term "reflectivity" refers to reflectivity measured by nanoscope, with Si as
25 a reference.

26 The term "SEM" refers to a scanning electron microscope.

27 The term "traditional sputtering" refers to a method of forming a film layer on



1 a substrate wherein a target is sputtered and the material sputtered from the target
2 passes between the target and the substrate to form a film layer on the substrate, and
3 no means is provided to ionize a substantial portion of the target material sputtered
4 from the target before it reaches the substrate. One apparatus configured to provide
5 traditional sputtering is disclosed in U.S. Patent No. 5,320,728, the disclosure of
6 which is incorporated herein by reference. In such a traditional sputtering
7 configuration, the percentage of target material which is ionized is less than 10 %,
8 more typically less than 1%, of that sputtered from the target.

9 The term "warm aluminum" refers to aluminum applied using traditional
10 sputtering techniques, where the substrate temperature during application of the
11 aluminum ranges between about 350 °C to about 450 °C.

12 The term "XPS" refers to X-ray Photoelectron Spectroscopy (also known as
13 ESCA (Electron Spectroscopy for Chemical Analysis), an analytical technique wherein
14 a beam of X-ray is illuminated onto a sample. Photoelectrons, characteristic of the
15 sample elemental composition and corresponding binding energy of each element, are
16 emitted which are detected by an electron energy analyzer. Sensitivity factors are
17 used to transform the measured kinetic energy of photoelectrons into atomic percent
18 composition of different elements in the sample. In addition to detecting atomic
19 percent composition at a sample surface, information for the composition of a sample
20 throughout its thickness can be obtained by depth profiling. In that case, the sample
21 material is sputtered away incrementally using inert ions such as argon ions. At each
22 increment, XPS analysis is performed.

23 The term "XRD" (X-ray Diffraction) refers to a technique commonly used to
24 measure crystalline orientation, wherein radiation over particular wavelengths is passed
25 through the material to be characterized, and the diffraction of the radiation, caused by
26 the material through which it passes, is measured. A map is created which shows the
27 diffraction pattern, and the crystal orientation is calculated based on this map.



II. AN APPARATUS FOR PRACTICING THE INVENTION

A process system in which the method of the present invention may be carried out is the Applied Materials, Inc. (Santa Clara, California) Endura® Integrated Processing System. This process system is not specifically shown in the Figures, however, the processing elements shown in Figure 1 can be operated within one of the low pressure process chambers contained within such an Integrated Processing System. The system is shown and described in United States Patents Nos. 5,186,718 and 5,236,868, the disclosures of which are incorporated by reference. With reference to Figure 1, one low pressure process chamber for forming the smooth-surfaced, low resistivity, titanium nitride barrier layer of the present invention employs a standard sputter magnet 110 (to confine the sputtering plasma, enabling an increased sputtering rate) and a sputtering target cathode 112 which operates at power levels up to about 24 kW.

EXAMPLE ONE

To form the Ti/TiN/TiN_x barrier layer structure of the present invention, a titanium target cathode of about 14 inches (35.5 cm) in diameter was used, and a DC power was applied to this cathode over a range from about 4 kW to about 8 kW. The substrate 118, comprising an 8 inch (20.3 cm) diameter silicon wafer, was placed a distance of about 5.5 inches (14 cm) from target cathode 112. A high density, inductively coupled RF plasma was generated in the region between the target cathode 112 and the substrate 118 by applying RF power 116 over a range from about 100 kHz to about 60 MHz (preferably about 2 MHz), at a wattage ranging from about 0.5 kW to about 6 kW (preferably ranging from about 1.5 kW to about 4 kW), to a coil 114 having at least one turn up to about 10 turns (preferably from about 1 to 3 turns). Typically the coil is fabricated from metal tubing which permits water cooling, and



1 has a diameter of about 0.125 inch (0.32 cm). However, the coil can be fabricated
2 from a sheet or ribbon, or other form which provides the desired function. Coil 114
3 surrounded a plasma region between the target 112 and substrate 118. Typically, a
4 substrate bias voltage ranging from 0 to about -300 V DC, preferably about 100 W,
5 is applied to the substrate 118 or the support member 120 to create a D.C. self bias
6 which attracts ions from the plasma to the substrate.

7 Although the preferred apparatus for forming the titanium nitride barrier layer
8 uses a coil to inductively couple the plasma and ionize the sputtered material, other
9 means for ionizing the titanium are contemplated. For example, an ECR source, such
10 as that shown and described in U.S. Patent No. 4,911,814, incorporated herein by
11 reference, or a helicon type coupling device such as that shown in U.S. Patent No.
12 4,990,229, incorporated herein by reference, are also specifically contemplated.
13 Likewise other apparatus which would supply an ionized stream of deposition particles
14 having an ionized percentage of 10 to 100 % are contemplated as being useful to
15 practice the invention. Although the preferred apparatus for forming the titanium
16 atoms which are ionized and reacted with ionized nitrogen to form titanium nitride are
17 preferably formed using sputtering techniques, other means for producing titanium
18 atoms are contemplated. For example titanium evaporation techniques, such as the
19 technique described in U.S. Patent No. 4,944,961 for vaporizing metal or metal alloy
20 in a crucible, incorporated herein by reference, are also specifically contemplated.

21 22 III. THE STRUCTURE 23 OF THE (Ti or TiN_x)/TiN/TiN_x BARRIER LAYER 24

25 We have been able to create a smooth titanium nitride-comprising barrier layer
26 which has been shown to enable electrical contact fabrication in vias, through holes,
27 and trenches of integrated circuits having a feature size as small as about 0.25 micron,
28 having an aspect ratio of about 5 : 1 and greater. The smooth barrier layer of the



1 invention, in addition to permitting the formation of a conductive contact at high
2 aspect ratio, provides a diffusion barrier which prevents the inter-diffusion between
3 the electrical contact material such as aluminum and an underlying semiconductor
4 substrate material such as silicon.

5 The smooth barrier layer structure comprises a stack of three layers. In the
6 more preferred embodiment, the first layer (applied directly over an underlying
7 substrate such as silicon or silicon oxide) is ion-deposited titanium (Ti). The second
8 layer, applied over the first layer, is ion-deposited titanium nitride (TiN). The third
9 layer, applied over the second layer, is an ion-deposited layer, TiN_x , where the
10 composition of the layer varies from about 50 atomic % titanium to about 100 atomic
11 % titanium. Preferably the titanium concentration is a gradient beginning with
12 essentially stoichiometric TiN and progressing toward pure Ti. To reduce the
13 potential for reaction of the TiN_x surface with warm aluminum which is subsequently
14 deposited upon it, it is necessary to control the Ti content at the surface of the layer.
15 The optimal Ti content of the TiN_x layer will depend on whether the application is for
16 a horizontal, conductive interconnect or for filling a high aspect ratio via. A high
17 aspect ratio via requires a higher Ti content in the TiN_x layer to provide for the
18 thermodynamic attraction of aluminum fill into the via. However, too high a Ti
19 content results in the production of $TiAl_3$, which negatively impacts device
20 performance. We have discovered that for high aspect ratio via applications, where
21 the TiN_x layer titanium content is a gradient from an atomic concentration of about
22 50% toward an atomic concentration of 100% Ti, a Ti upper surface thickness of less
23 than about 15 Å is insufficient to draw the warm aluminum into the via in a manner
24 which provides complete fill of the via, while a pure Ti upper surface thickness of
25 more than about 300 Å results in the formation of harmful amounts of $TiAl_3$.

26 An electrical contact or conductive layer is applied over the smooth barrier
27 layer structure. Although the contact or conductive layer described herein is



1 aluminum containing about 0.5% by weight copper, other conductive materials benefit
2 from use of the smooth barrier layer structure described herein. The {111}
3 crystallographic content of a subsequently applied conductive material and its
4 reflectivity can be adjusted using the concepts described herein. With regard to warm
5 aluminum, the preferred embodiment of this disclosure, the aluminum can be applied
6 using methods other than sputtering, such as evaporation, for example. An IMP
7 application of aluminum provides some advantages in via filling, as it provides a
8 particular conformality on a via sidewall; however, traditionally sputtered aluminum is
9 preferred since this is the least expensive method of application for the aluminum.
10 With regard to traditionally sputtered aluminum, applied as warm aluminum (over a
11 temperature range of from about 350 °C to about 450 °C), preferably the aluminum is
12 applied at reduced pressures ranging from greater than about 0.5 mT up to about 50
13 mT, preferably between about 1 mT and 30 mT, and more preferably between about 1
14 mT and 4 mT. The aluminum thickness depends on the application.

15 Figure 2 shows a schematic of a trench or via 213 containing the smooth
16 barrier layer structure 200 of the present invention. The structure 200 was formed on
17 a semiconductor substrate comprising a silicon base 210 having an overlying silicon
18 dioxide layer 211. The via or trench 213 was created by dry etching through the
19 silicon dioxide layer 211 to silicon base 210. Structure 200 comprised three layers:
20 Ti/TiN/TiN_x. The first layer of titanium was ion-deposition sputtered
21 upon the surface of both silicon dioxide layer 211 and silicon base 210; a second layer
22 of reactive ion-deposition sputtered titanium nitride layer 214 was deposited overlying
23 first titanium layer 212; and a third ion-deposition sputtered titanium nitride-
24 comprising layer 216 was deposited overlying titanium nitride layer 214. (Upon ion-
25 sputtering of titanium layer 212, a thin layer of titanium silicide 224 is typically
26 formed at the bottom of via 213 upon high temperature annealing). Structure 200 was
27 then filled with a conductive layer 219. The aspect ratio of via or trench 213 was as



1 illustrated by the ratio of dimension 222 to dimension 220, and was approximately
2 20 : 1, with the dimension of 220 being approximately 0.25 μ .

3 Aluminum was traditionally sputtered upon a structure 200 at a substrate
4 temperature of about 500 °C. In some instances, a cold aluminum nucleation layer
5 was deposited at substrate temperatures of about 50 °C prior to deposition of the
6 remaining aluminum at 500 °C. Due to the improved surface on the sidewall of the
7 barrier layer structure 200 which was obtained using IMP, it was possible to eliminate
8 the need to use cold aluminum nucleation layer, so that only one aluminum deposition
9 chamber was necessary. Further, warm aluminum deposition over a temperature
10 ranging from about 350 °C to about 450 °C became feasible. When the specialized
11 barrier layer structure 200 of the present invention is used, a further benefit of high
12 {111} crystallographic orientation aluminum is obtained. Further, particularly high
13 aspect ratio vias can be filled.

14 EXAMPLE TWO:

15 To obtain an aluminum fill of the via where the crystallographic {111} content
16 was sufficiently high to prevent electromigration, the structure of the Ti/TiN/TiN_x
17 barrier layer 200 was adjusted as follows.

18 The thickness of the first layer 212 of ion-deposition sputtered Ti was about
19 300 Å; the thickness of the second layer 214 of reactive ion-deposition sputtered TiN
20 was about 250 Å; and the thickness of the third layer 216 of reactive ion-deposition
21 sputtered TiN_x was about 250 Å. The composition of the TiN_x layer 216 ranged from
22 50 atomic % Ti initially to toward about 100 atomic % Ti toward the surface of layer
23 216.

24 Aluminum was sputtered over the surface of via 213 using traditional
25 sputtering, at a temperature of about 400 °C and at a pressure of about 2 mT.

26 The {111} crystallographic content of the aluminum fill was about 100 % (no
27 200 was observed on the XRD curve). Further, the reflectivity of the aluminum



1 surface was about 199 %.

2 With reference to Figures 3A through 3D, these figures illustrate the XRD
3 curves for warm aluminum applied over the surface of a Ti/TiN/TiN_x barrier layer
4 overlying a flat silicon oxide surface.

5 Figure 3A is the XRD curve 310 for warm aluminum applied over the surface
6 of a Ti/TiN/TiN_x barrier layer fabricated using the techniques described above, where
7 the Ti first layer is about 100 Å thick; the TiN second layer is about 800 Å thick, and
8 the TiN_x third layer is about 150 Å thick. The process conditions used during
9 fabrication of the barrier layer included a substrate (wafer) temperature of about
10 200 °C during the application of each layer; a DC/RF Power Ratio (this power ratio
11 will be described in more detail subsequently) of about 3.3; a Bias to the substrate of
12 about 100 W; and a process chamber pressure of about 30 mT during application of
13 each of the layers. Warm aluminum was sputtered over the surface of the
14 Ti/TiN/TiN_x barrier layer using traditional sputtering at a substrate temperature of
15 about 400 °C and at a pressure of about 2 mT. The power ratio mentioned above
16 refers to the DC power applied to the sputtering target divided by the RF power to the
17 ionizing device (coil); an example would be 7 kW to the sputtering target and 2 kW
18 to the RF coil, for a power ratio of 3.5.

19 As indicated by XRD curve 310, with the Intensity of diffraction in arbitrary
20 units indicated on the "y" axis and labeled "312", and 2θ (twice the angle of
21 incidence) indicated in degrees on the "x" axis and labeled "314", the crystal
22 orientation content of the aluminum includes both the desired {111} orientation 316
23 and undesired {200} orientation 319 (shown enlarged at 318). The weak signal of the
24 {111} orientation which produced a peak height of only about 3,000 is a further
25 indication of the more polycrystalline nature of the warm deposited aluminum. The
26 presence of the undesired {200} orientation aluminum is attributed to the thickness of
27 the TiN layer being 800 Å.



1 Figure 3B is the XRD curve 320 for warm aluminum applied over the surface
2 of a Ti/TiN/TiN_x barrier layer fabricated using the techniques described above, where
3 the Ti first layer is about 100 Å thick; the TiN second layer is about 250 Å thick, and
4 the TiN_x third layer is about 150 Å thick. The process conditions used during
5 fabrication of the barrier layer included a substrate (wafer) temperature of about
6 200 °C during the application of each layer; a DC/RF Power Ratio of about 3.3; a
7 Bias to the substrate of about 100 W; and a process chamber pressure of about 30 mT
8 during deposition of each layer of the barrier layer structure. Warm aluminum was
9 sputtered over the surface of the Ti/TiN/TiN_x barrier layer using traditional sputtering
10 at a substrate temperature of about 400 °C and at a pressure of about 2 mT.

11 As indicated by XRD curve 320, with the Intensity of diffraction indicated on
12 the "y" axis and labeled "322", and 2θ indicated on the "x" axis and labeled "324",
13 the crystal orientation content of the aluminum is limited to the desired {111}
14 orientation, with none of the undesired {200} orientation (which would appear in
15 enlargement 328). The stronger signal of the {111} orientation, which produced a
16 peak height of about 14,000, is a further indication of the higher degree of {111}
17 content in the warm deposited aluminum.

18 The barrier layer structure which enabled the deposition of the aluminum having the
19 XRD curve shown in Figure 3B is one of the more preferred embodiments of the
20 present invention.

21 Figure 3C is the XRD curve 330 for warm aluminum applied over the surface
22 of a Ti/TiN/TiN_x barrier layer fabricated using the techniques described above, where
23 the Ti first layer is about 100 Å thick; the TiN second layer is about 250 Å thick, and
24 the TiN_x third layer is about 150 Å thick. The process conditions used during
25 fabrication of the barrier layer included a substrate (wafer) temperature of about
26 400 °C during the application of each layer; a DC/RF Power Ratio of about 3.3; a
27 Bias to the substrate of about 100 W; and a process chamber pressure of about 30 mT



1 during application of the each layer of the barrier layer structure. Warm aluminum
2 was sputtered over the surface of the Ti/TiN/TiN_x barrier layer using traditional
3 sputtering at a substrate temperature of about 400 °C and at a pressure of about 2 mT.

4 As indicated by XRD curve 330, with the Intensity of diffraction indicated on
5 the "y" axis and labeled "332", and 2 θ indicated on the "x" axis and labeled "334",
6 the crystal orientation content of the aluminum includes both the desired {111}
7 orientation 336 and undesired {200} orientation 339 (shown enlarged at 338). The
8 extremely weak signal of the {111} orientation, which produced a peak height of only
9 about 34, is an indication that the degree of polycrystallinity of the aluminum is
10 significantly greater than that of the aluminum layer illustrated in Figure 3A. This is
11 further supported by an increased peak height for the {200} crystal orientation fraction
12 of the aluminum. The presence of the undesired {200} orientation aluminum is
13 attributed to the 400 °C deposition temperature used for each of the three barrier
14 layers.

15 Figure 3D is the XRD curve 340 for warm aluminum applied over the surface
16 of a Ti/TiN/TiN_x barrier layer fabricated using the techniques described above, where
17 the Ti first layer is about 300 Å thick; the TiN second layer is about 250 Å thick, and
18 the TiN_x third layer is about 150 Å thick. The process conditions used during
19 fabrication of the barrier layer included a substrate (wafer) temperature of about
20 200 °C during the application of each layer; a DC/RF Power Ratio of about 3.3; a
21 Bias to the substrate of about 100 W; and a process chamber pressure of about 30 mT
22 during application of the Ti and TiN layer, but with the process chamber pressure
23 reduced to about 10 mT during application of the TiN_x layer. Warm aluminum was
24 sputtered over the surface of the Ti/TiN/TiN_x barrier layer using traditional sputtering
25 at a substrate temperature of about 400 °C and at a pressure of about 2 mT.

26 As indicated by XRD curve 340, with the Intensity of diffraction indicated on
27 the "y" axis and labeled "342", and 2 θ indicated on the "x" axis and labeled "344",



1 the crystal orientation content of the aluminum is limited to the desired {111}
2 orientation 346, without the presence of any {200} orientation (which would appear in
3 the enlargement at 348). The very strong signal of the {111} orientation, which
4 produced a peak height of about 27,000, is a further indication of the higher degree of
5 {111} content in the warm deposited aluminum (significantly higher than obtained for
6 the other preferred embodiment of the invention illustrated in Figure 3B, where the
7 TiN_x layer was deposited at 30 mT). The barrier layer structure which enabled the
8 deposition of the aluminum having the XRD curve shown in Figure 3D is one of the
9 more preferred embodiments of the present invention.

10 Figure 4 illustrates the reflectivity for three of the four aluminum/Ti/TiN/TiN_x
11 structures described above and for one different structure. In particular, bar graph 400
12 shows the percent reflectivity (with Si as a reference) on the "y" axis, labeled "410".
13 Each bar on the "x" axis represents a different aluminum/Ti/TiN/TiN_x structure.

14 Bar 412 shows the reflectivity of the aluminum layer for the
15 aluminum/Ti/TiN/TiN_x structure illustrated in the XRD curve shown in Figure 3B.
16 Bar 414 shows the reflectivity of an aluminum layer deposited over a Ti/TiN/TiN_x
17 structure where the method used to produce the entire aluminum/Ti/TiN/TiN_x structure
18 was the same as that used to produce the entire Figure 3B structure, but the thickness
19 of the Ti layer is 300 Å. Bar 416 shows the reflectivity of the aluminum layer for the
20 aluminum/Ti/TiN/TiN_x structure illustrated in the Figure 3A XRD curve. And, Bar
21 418 shows the reflectivity of the aluminum layer for the aluminum/Ti/TiN/TiN_x
22 structure illustrated in the Figure 3C XRD curve.

23 Figure 4 illustrates that, in general, a higher aluminum {111} crystal
24 orientation correlates directly with increased aluminum reflectivity.

25 An even further improvement in reflectivity (and accompanying reduction in
26 surface roughness of the aluminum layer) can be obtained by altering the composition
27 gradient of the third layer TiN_x. We have discovered that by increasing the thickness



1 of the pure Ti which deposits at the end of the TiN_x deposition step, an improvement
2 in reflectivity and a smoother-surfaced aluminum can be obtained. In particular,
3 reflectivity was increased from about 210 % to about 220 % and surface roughness
4 (determined using AFM) was reduced from about 78 Å to about 47 Å.

5 Figures 5 A and 5B represent XRD curves for warm aluminum applied over
6 the surface of a Ti/TiN/TiN_x barrier layers fabricated to have a different thickness of
7 Ti at the end of the TiN_x deposition. The barrier layer structures were fabricated
8 using the techniques described above. In particular, the thickness of the first Ti layer
9 was 300 Å, and the thickness of the second TiN layer was 250 Å in each case. All
10 barrier layers were deposited at a substrate temperature of about 200 °C and at a
11 process chamber pressure of 10 mT.

12 Figure 5A shows the XRD curve 510 for warm aluminum applied over the
13 surface of the Ti/TiN/TiN_x barrier layer where the TiN_x deposition time was about 15
14 seconds, and the resulting thickness of the TiN_x layer was about 180 Å. The
15 diffraction Intensity is shown on the "y" axis and labeled "512"; and, 2 θ is indicated
16 on the "x" axis and labeled "514". Curve 510 exhibits a strong {111} aluminum
17 crystal orientation peak 516 of about 28,000 in intensity and no detectable presence of
18 {200} crystal orientation. The reflectivity for the aluminum surface is about 210 %
19 and the AFM surface roughness is about 78 Å.

20 Figure 5B shows the XRD curve 520 for warm aluminum applied over the
21 surface of the Ti/TiN/TiN_x barrier layer where the TiN_x deposition time was about 30
22 seconds, and the resultant thickness of the TiN_x layer was about 360 Å. The
23 diffraction Intensity is shown on the "y" axis and labeled "522"; and, 2 θ is indicated
24 on the "x" axis and labeled "524". Curve 520 exhibits a strong {111} aluminum
25 crystal orientation peak 526 of about 39,000 in intensity and no detectable presence of
26 {200} crystal orientation. The reflectivity for the aluminum surface is about 220 %
27 and the AFM surface roughness is about 47 Å.



1 IV. THE METHOD OF APPLICATION
2 OF BARRIER LAYERS AND ALUMINUM

3 The apparatus in which the preferred embodiments described herein were
4 fabricated was the Endura® Integrated Processing System previously described, and
5 the elements shown in Figure 1 were present within one of the low pressure process
6 chambers contained within the Integrated Processing System.

7 EXAMPLE THREE:

8 The description which follows pertains to the fabrication of a Ti/TiN/TiN_x
9 barrier layer over the surface of a 0.25 μ feature size via having an aspect ratio of
10 about 5:1.

11 With reference to Figure 2, the via was created by dry etching through the
12 silicon dioxide layer 211 to silicon base 210. Structure 200 comprised three layers:
13 Ti/TiN/TiN_x. The first layer of Ti was ion-deposition sputtered (IMP sputtered) upon
14 the surface of both silicon dioxide layer 211 and silicon base 210; a second layer of
15 reactive ion-deposition sputtered (reactive IMP sputtered) TiN layer 214 was deposited
16 overlying first titanium layer 212; and a third ion-deposition sputtered TiN_x layer 216
17 was deposited overlying titanium nitride layer 214. (Upon ion-sputtering of titanium
18 layer 212, a thin layer of titanium silicide 224 is typically formed at the bottom of via
19 213 upon high temperature annealing). Structure 200 was then filled with a
20 conductive layer 219.

21 To obtain an ion-deposition sputtering rate of about 700 Å per minute of Ti
22 upon the surface of an 8 inch (20.3 cm) diameter substrate, 2 MHz (≈2.5 kW) of RF
23 power was applied to coil 114 while 4 kW of DC power was applied to titanium
24 target cathode 112, and a DC bias of about 100 Watts was applied to substrate platen
25 electrode 120. The Ti deposition was carried out at about 30 mT. This pressure
26 corresponded to an argon feed rate of about 65 sccm. The temperature on the
27 substrate surface was about 200 °C.



1 To obtain a reactive ion-deposition sputtering rate for TiN of about 250 Å per
2 minute over the Ti first layer surface, 2 MHz (≈ 2.5 kW) of RF power was applied to
3 coil 214 while 5 kW of DC power was applied to titanium target cathode 212, and a
4 DC bias of about 100 Watts was applied to substrate platen electrode 220. The TiN
5 deposition was carried out at about 30 mT. This pressure corresponded to an argon
6 feed rate of about 10 sccm and a nitrogen feed rate of about 70 sccm in the Applied
7 Materials Endura ® Integrated Process System. The temperature on the substrate
8 surface was about 200 °C.

9 To obtain a reactive ion-deposition sputtering rate for TiN_x of about 600 Å per
10 minute upon the TiN second layer surface, the process conditions were set at those
11 specified for the TiN second layer; however, at the beginning of the sputtering of the
12 TiN_x layer, the flow of nitrogen gas to the process chamber was discontinued. Since
13 the titanium target becomes nitrided during the TiN deposition, TiN continues to be
14 produced for a limited time period after the nitrogen gas flow is discontinued.
15 Typically, under the operational conditions specified with reference to the TiN
16 deposition, but with the flow of nitrogen gas to the chamber discontinued, the nitrided
17 portion of the target is sputtered away within 5 to 10 seconds. The thicker the TiN
18 second layer, the longer the time period before the nitrided portion of the target is
19 sputtered away. In this particular instance, the TiN_x layer deposition was carried out
20 for a period of about 15 seconds after the flow of nitrogen to the process chamber was
21 discontinued.

22 Subsequently an aluminum layer about 6,000 Å thick was sputtered upon the
23 via surface using traditional sputtering techniques, a substrate temperature of about
24 400 °C, and a pressure of about 2 mT. Complete via fill (with essentially no voids
25 evidenced in the photomicrographs) was achieved for the contact vias using the
26 Ti/TiN/TiN_x barrier/wetting layer fabricated as described above.



1 V. THE COMPOSITION OF INDIVIDUAL LAYERS
2 IN THE BARRIER LAYER STRUCTURE

3 Figures 6A through 6C show the atomic composition of the three barrier layers
4 as analyzed from the top, through the TiN_x third layer, then the TiN second layer, and
5 then the Ti first layer which lies over a silicon oxide substrate.

6 Graph 610 shown in Figure 6A illustrates the composition of the particular
7 barrier layer as the layers are sputtered away during the XPS analytical process. The
8 atomic composition of the material being sputtered using argon ions is represented on
9 the "y" axis, labeled as "612" and is in atomic percent. The time into the analytical
10 process is presented on the "x" axis, labeled "614" and is in seconds. Curve 616
11 represents the Ti content of the layer being analyzed and curve 618 represents the N
12 content of the layer being analyzed. Curve 620 represents oxygen which is an artifact
13 from cratering of the substrate during the analytical process sputtering. Curve, 622
14 represents silicon which is also an artifact of the analytical technique.

15 Graph 610 is representative of a barrier layer having a Ti thickness of about
16 100 Å; a TiN thickness of about 250 Å, and a TiN_x thickness of about 100 Å, where
17 the TiN_x layer deposition was continued for a period of about 8 seconds after the flow
18 of nitrogen gas flow to the deposition process chamber was shut off.

19 Graph 630 shown in Figure 6B illustrates the composition of a second barrier
20 layer structure determined using the analytical technique described with reference to
21 Figure 6A. The atomic composition of the layer being analyzed is represented on the
22 "y" axis, labeled as "632" and is in atomic percent. The time into the analytical
23 process is presented on the "x" axis, labeled as "634" and is in seconds. Curve 636
24 represents the Ti content of the layer being analyzed, and Curve 638 represents the N
25 content of the layer being analyzed. Curve 640 represents oxygen artifact and Curve
26 642 represents the silicon artifact.

27 Graph 630 is representative of a barrier layer having a Ti thickness of about



1 200 Å; a TiN thickness of about 500 Å, and a TiN_x thickness of about 100 Å, where
2 the TiN_x layer deposition was again continued for a period of about 8 seconds after
3 the flow of nitrogen gas to the deposition process chamber was shut off.

4 Graph 650 shown in Figure 6C illustrates the composition of a third barrier
5 layer structure determined using the analytical technique described with reference to
6 Figure 6A. The atomic composition of the layer being analyzed is represented on the
7 "y" axis, labeled as "652" and is in atomic percent. The time into the analytical
8 process is presented on the "x" axis, labeled as "654" and is in seconds. Curve 656
9 represents the Ti content of the layer being analyzed, and Curve 658 represents the N
10 content of the layer being analyzed. Curve 660 represents oxygen artifact and Curve
11 662 represents the silicon artifact.

12 Graph 650 is representative of a barrier layer having a Ti thickness of about
13 200 Å; a TiN thickness of about 250 Å, and a TiN_x thickness of about 250 Å, where
14 the TiN_x layer deposition was continued for a period of about 20 seconds after the
15 flow of nitrogen gas to the deposition process chamber was shut off.

16 These structures are obtainable in a single process chamber, using a single Ti
17 sputtering target, and can be fabricated in a continuous process. The simplicity of
18 producing such barrier layer structures, combined with the excellent functionality of
19 the barrier layers makes them especially attractive for semiconductor device
20 fabrication. In particular, using the barrier layer structures and the fabrication
21 methods disclosed herein enables the formation of aluminum-comprising layers having
22 high aluminum {111} crystal orientation which reduces electromigration problems.
23 Further, using the barrier layer structures and the fabrication methods disclosed herein
24 enables the formation of high reflectivity aluminum-comprising layers which are
25 useful in lithographic indexing.

26 The above described preferred embodiments are not intended to limit the scope



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Attorney Docket No. AM-1808

U.S. Express Mail No. EH734847151US

1 of the present invention, as one skilled in the art can, in view of the present disclosure
2 expand such embodiments to correspond with the subject matter of the invention
claimed below.

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CLAIMS

We claim:

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1. A barrier layer having a particular structure comprising:

a) a first layer of Ti or TiN_x having a thickness ranging from greater than about 100 Å to about 500 Å;

b) a second layer of TiN having a thickness ranging from greater than about 100 Å to less than about 800 Å; and

c) a third layer of TiN_x having a thickness ranging from about 15 Å to about 500 Å.

2. The barrier layer of Claim 1, where the Ti content of said first layer of TiN_x is at least 40 atomic percent.

3. The barrier layer of Claim 1, wherein the thickness of said second layer of TiN is less than about 500 Å.

4. The barrier layer of Claim 3, wherein the thickness of said third layer of TiN_x is less than about 400 Å.

5. The barrier layer of Claim 1 wherein the Ti content of said third layer of TiN_x ranges from about 50 atomic percent to about 100 atomic percent and follows a gradient where the first portion of said third layer is at said 50 atomic percent and the last portion of said third layer approaches said 100 atomic percent Ti.

6. The barrier layer of Claim 5, wherein the thickness of said 100 atomic percent Ti component ranges from about 15 Å to about 300 Å.





1 7. A structure comprising the barrier layer of Claim 1 and having an aluminum-
2 comprising conductive layer applied over a surface of said third layer of TiN_x .

1 8. A structure comprising the barrier layer of Claim 5 and having an aluminum-
2 comprising conductive layer applied over a surface of said third layer of TiN_x .

1 9. The structure of Claim 7, wherein said aluminum-comprising layer contains at least
2 90 atomic percent aluminum.

1 10. The structure of Claim 8, wherein said aluminum-comprising layer contains at
2 least 90 atomic percent aluminum.

1 11. The structure of Claim 9, wherein said aluminum-comprising layer exhibits
2 essentially no {200} crystallographic content.

1 12. The structure of Claim 10, wherein said aluminum-comprising layer exhibits
2 essentially no {200} crystallographic content.

1 13. The structure of Claim 11 wherein said aluminum-comprising layer exhibits a
2 reflectivity in excess of 150 percent.

1 14. The structure of Claim 12, wherein said aluminum-comprising layer exhibits a
2 reflectivity in excess of 190 percent.

1 15. a method of producing an aluminum-comprising layer/Ti/TiN/TiN_x structure,
2 wherein a crystal orientation of said aluminum-comprising layer is essentially {111},





3 said method comprising:

4 a) using IMP techniques to apply a first layer of Ti or TiN_x having a thickness
5 ranging from about 100 Å to about 500 Å;

6 b) using reactive IMP techniques to apply a second layer of TiN having a
7 thickness ranging from about 100 Å to less than about 800 Å; and

8 c) using reactive IMP techniques to apply a third layer of TiN_x having a
9 thickness ranging from about 15 Å to about 500 Å.

1 16. The method of Claim 15, wherein the nitrogen content during said reactive IMP
2 deposition of said third layer of TiN_x is controlled to produce a 100 atomic percent Ti
3 component at the surface of said TiN_x layer surface.

1 17. The method of Claim 16, where the thickness of said 100 atomic percent Ti
2 component ranges from about 15 Å to less than about 300 Å.

1 18. The method of Claim 15, wherein said barrier layers are deposited at a substrate
2 temperature of less than about 400°C.

1 19. The method of Claim 18, wherein said barrier layers are deposited at a substrate
2 temperature of about 200 °C or lower.

1 20. The method of Claim 18, wherein said third layer of TiN_x is applied at process
2 chamber pressures ranging from about 0.5 mT to about 30 mT.

1 21. The method of Claim 20, wherein said pressure is about 10 mT or less.

