

# United States Patent [19]

### Sellers

### [54] PREFERENTIAL SPUTTERING OF INSULATORS FROM CONDUCTIVE TARGETS

- [75] Inventor: Jeff C. Sellers, Palmyra, N.Y.
- [73] Assignee: ENI, Rochester, N.Y.
- [21] Appl. No.: 261,988
- [22] Filed: Jun. 17, 1994
- [52]
- 204/298.03; 204/298.08 [58] Field of Search ...... 204/192.12, 192.13,
  - 204/298.03, 298.08

#### [56] **References** Cited

### U.S. PATENT DOCUMENTS

4,103,324	7/1978	Vandervelden et al 204/298.08 X
4,936,960	6/1990	Siefkes et al 204/298.41 X
5,241,152	8/1993	Anderson et al 204/298.08 X
5,286,360	2/1994	Szczyrbrowski et al 204/298.08

## US005651865A

#### 5,651,865 [11] Patent Number:

#### [45] **Date of Patent:** Jul. 29, 1997

5,300,205	4/1994	Fritsche 204/298.08 X
5,303,139	4/1994	Mark 204/298.08 X
5,427,669	6/1995	Drummond 204/298.08

### FOREIGN PATENT DOCUMENTS

0 591 675	8/1993	European Pat. Off
0 564 789 A1	10/1993	European Pat. Off
0 636 702	7/1994	European Pat. Off
WO87/05053	8/1987	WIPO .

Primary Examiner-Nam Nguyen Attorney, Agent, or Firm-Trapani & Molldrem

#### ABSTRACT [57]

Pulses of positive voltage are applied to the target of a dc sputtering process to create a reverse bias. This charges insulating deposits on the target to the reverse bias level, so that when negative sputtering voltage is reapplied to the target, the deposits will be preferentially sputtered away. The reverse bias pulses are provided at a low duty cycle, i.e., with a pulse width of 0.25-3 microseconds at a pulse rate of about 40-100 KHz. This technique reduces sources for arcing during a reactive sputtering process.

### 5 Claims, 4 Drawing Sheets







FIG.2

















### PREFERENTIAL SPUTTERING OF INSULATORS FROM CONDUCTIVE TARGETS

### BACKGROUND OF THE INVENTION

This invention relates to the deposition of thin films and is more concerned with a reactive sputtering process wherein atoms of a target material are freed from a conductive target and are reacted with a reactive gas to form a coating which is deposited on a substrate. This process can be employed, for example, in creating dielectric insulating layers on electrical parts, or wear-resistant layers on mechanical parts.

The invention is more specifically directed to a dc sputtering process in which the dielectric coating material that becomes lodged on the conductive target is removed, thus avoiding a major cause of arcing.

Sputtering is a vacuum deposition process in which a sputtering target is bombarded with ions, typically an ion- 20 ized noble gas, and the atoms of the target material are mechanically freed by momentum transfer. The target material then coats a nearby substrate.

In a reactive sputtering process a reactive gas is introduced into the deposition chamber, and the freed target 25 material reacts with the reactive gas to form a coating material. For example, the target material can be aluminum, and oxygen can be introduced as the reactive gas to produce a coating of aluminum oxide. A carbonaceous gas, e.g. acetylene can be used as the reactive gas to produce carbide 30 coatings such as SiC. WC, etc., and ammonia can be introduced to produce a nitride coating such as TiN. In any event, the conductive target atoms and the reactive gas react in plasma in the chamber to produce the compound that serves as a coating. In a typical example, aluminum atoms 35 freed from an aluminum target enter plasma of argon and oxygen to produce a deposition of aluminum oxide.

DC sputtering is a random process and the insulating coating material is deposited on all available surfaces. This means that not only does the insulating material coat the article in question, but it also coats other surfaces in the chamber as well, including the target. Thus, in a reactive sputtering process for depositing aluminum oxide, molecules of Al<sub>2</sub>O<sub>3</sub> land on the surface of the aluminum target. 45 This deposition of an insulator on the target causes severe problems, including a reduction of sputtering rate and a propensity to induce arcing.

Contamination of the target can also result, even in conventional dc sputtering, due to atmospheric gases, water 50 droplets, inclusions, and other contaminants. Each of these can be a source for arcing, and their presence will also reduce the deposition rate over time because of a reduced active sputtering area on the target. Accordingly, these problems necessitate a frequent cleaning of the target surface.

This problem has been known to exist for some time, but its causes have not been completely appreciated. Procedures to deal with these problems, such as arc control in reactive sputtering have not been completely satisfactory.

60

A standard approach involves sensing the presence of arcing, and then interrupting current flow. This will control the arcing, but does nothing about the insulating coating that continues to cover the target.

One early attempt to deal with arcing in a blind fashion 65 involves periodically interrupting the current flow between the dc power supply and the plasma generator in which the

sputtering occurs. Here, shutting off the dc power serves to extinguish early arcing. This means that unipolar pulses of power at a fixed duty cycle are fed to the target. This does have the beneficial effect of permitting charge to build up only partially across the dielectric deposition on the target, so that arcing is less likely to occur, and also can lead to a small amount of resputtering of the deposition. However, this system while slowing down the rate of insulating deposit on the target, does not reverse the deposition.

Another system previously proposed is called a low energy small package arc repression circuit. In that system, an electronic switch cycles at a rate of about 2 KHz to cut out the current to the target. This in effect reverses the voltage on the target to several volts positive, and draws some electrons from the plasma to the front surface of the insulative deposition. This neutralizes the anions on the front surface of the deposition, to discharge the voltage buildup across the layer, thereby greatly reducing the occurrence of dielectric breakdown and arcing. Also, discharge of the front surface of the insulative layer lowers the surface potential to approximately that of the target. Discharging of the dielectric deposition also permits the argon ions in the plasma to collide with the insulating dielectric material. This does result in some resputtering of the molecules of the deposited material, thus slowing the rate of deposit on the target.

However, this approach does not resputter the molecules of the deposited compound as effectively as the atoms of the target material, and so this approach has not been totally effective in removing deposits from the target during reactive sputtering processes.

Different materials require different voltages to be applied to the targets to effect sputtering. For example, because a gold atom is a much heavier atom than an aluminum atom, it requires a much more energetic ion to free it from the target. Typically, in a process that employs an aluminum target, the applied voltage needed is about -450 volts, while in a similar process that employs a gold target the applied voltage has to be about -700 volts.

Considering that an aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) molecule is significantly heavier than an aluminum atom, one can understand that a higher potential would be required to energize the argon ion enough to resputter the coating. This is, of course, true for other materials as well.

Another approach to solve this problem involves a pair of sputtering targets, with one serving as cathode and the other as the anode. The applied electrical voltage is periodically reversed so that the sputtering occurs first from the one target and then from the other. This process also reverses the charge on the deposited insulating material as well, which reduces the possibility of arcing and also resputters some of the insulating material on the targets. However, this arrangement, requiring plural targets, can be cumbersome and expensive to employ.

These previous solutions, which employ unipolar pulsing or alternately cycled targets, have been effective in reducing <sup>55</sup> voltage stress on insulating films redeposited on the targets, but have not been effective in removing the redeposit or in preventing it. None of these approaches sputters off the insulator from the beginning before it has a chance to accumulate, and none of these techniques has been effective in eliminating or halting the redeposition of insulating film on the target.

### OBJECTS AND SUMMARY OF THE INVENTION

It is an object of this invention to enhance reactive sputtering in a fashion which avoids accumulations of insulating deposition on the conductive target.

It is another object to carry out reactive sputtering under conditions that eliminate sources of arcing and which maximizes the rate at which sputtering can take place.

According to an aspect of the present invention, a reactive dc sputtering process is carried out in a plasma chamber by 5 applying an electric potential to a conductive target so that target material is sputtered from the target and is reacted with a reactive gas that is introduced into the chamber. A dc voltage of a suitable level e.g. -500 volts, is applied from a power supply to the sputtering target, which serves as 10 cathode, with a conductive surface in the plasma chamber being held at ground to serve as anode. A noble gas, e.g., argon, present in the chamber is ionized and creates a plasma, e.g., argon anions and free electrons. The electrons are drawn off in the anode, and the positive argon ions are 15 accelerated towards the cathode, that is, towards the conductive target. The argon ions knock atoms of target material free from the target by momentum transfer. The argon ions pick up electrons from the negatively charged target, and migrate back to the plasma. The freed target atoms enter the 20 plasma and react with a reactive gas that has been introduced into the chamber. The reactive gas can be, for example, oxygen, borane, acetylene, ammonia, silane, arsene, or various other gases. The reaction product is deposited on a substrate positioned adjacent the plasma. The substrate can 25 be a masked semiconductor wafer on which a compound such as a Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, or another insulator or dielectric is to be deposited. In some processes, the substrate can be a drill bit, wear plate, valve spindle, or other mechanical part on which a wear resistant coating, such as WC or TiN, is 30 deposited.

As mentioned before, the reaction product of the reactive sputtering process is deposited randomly, and not only coats the workpiece substrate, but also coats other surfaces including walls of the chamber and the sputtering target. Accumulation of the insulating coating can induce arcing, and also reduces the available area of the sputtering target, thereby reducing the sputtering rate over time.

In this invention the negative dc voltage that is applied to the target is interrupted periodically by reverse-bias pulses of a positive dc voltage relative to the anode. Preferably, the reverse bias pulses are at a level of 50 to 300 volts above ground potential, and these are applied at a pulse frequency of 40 KHz to 100 KHz with a pulse width of 1 µsec to 3 µsec. This results in low duty cycle pulses (about 10% or less). The reverse bias creates a reversal of the charge across the insulating material. These accumulations behave as a capacitor, with the conductive target being one plate and the conductive plasma being the other plate. The reverse voltage is applied long enough (e.g. 2 µsec) for the polarity of the capacitive charge to be reversed, up to -300 volts, on the plasma side of the deposition.

Then when the normal or negative sputtering voltage is again applied, the argon ions in the plasma are accelerated preferably towards the reverse-charged dielectric material. <sup>55</sup> These ions are accelerated to an increased energy due to the additional potential difference. As a result, the molecules of the deposition are re-sputtered off the target. This process keeps the target clear of depositions, and thereby eliminates the main sources of arcing. This process also permits the active sputtering surface of the target to remain as large as possible.

This process also cleans out other insulative contaminants from the surface of the target whether used for reactive sputtering or conventional sputtering.

The above and many other objects, features, and advantages of this invention will become more apparent from the ensuing description of a preferred embodiment, which should be read in conjunction with the accompanying Drawing.

### BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a schematic view of a dc sputtering arrangement for explaining the principles of reactive dc sputtering.

FIG. 2 is a Passione curve of voltage versus current density, showing the various regions of plasma behavior in a sputtering operation.

FIGS. 3 to 5 are schematic views for explaining the adverse effects of film accumulations on the sputtering target.

FIGS. 6 and 7 are schematic representations of an ideal sputtering process and one in which an arc discharge condition occurs.

FIGS. 8A and 8B are schematic views illustrating equivalency of an insulative film accumulation on the sputtering target with a capacitor.

FIG. 9 is a chart showing a pulse waveform for unipolar dc sputtering according to the prior art.

FIG. 10 is a chart showing a pulse waveform for low duty cycle reverse bias dc sputtering, according to this invention.

FIG. 11 is a waveform chart showing the behavior of electric potential at the plasma-facing surface of an insulator film accumulation on the conductive target, according to an embodiment of this invention.

FIG. 12 is a schematic view of an arrangement for practicing reactive dc sputtering according to the principles of this invention.

### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

With reference to the Drawing and initially to FIG. 1, a dc sputtering arrangement 10 employs a plasma chamber 12 and a regulated dc power supply 14. The plasma chamber is evacuated by a vacuum pump (not shown) and is supplied with a controlled amount of a noble gas, typically argon. A conductive target 16 is located within the chamber 12 and serves as a cathode. The target 16 is connected to a negative terminal of the power supply 14. The target 16 can be a flat plate of aluminum, tungsten, silicon or another conductive material. An anode 18 disposed within the chamber is coupled to the positive terminal of the power supply 14. The anode can have a relatively large surface area with respect to the target 16. A gas conduit 20 brings a reactive gas at a controlled rate into the chamber to react with atoms of the target material that become freed from the target. This reaction produces a reaction product that is deposited as a film on surfaces within the chamber. A plasma 22 is formed in the noble gas in a zone between the anode 18 and the target 16, and a substrate 24 to be coated is positioned in the chamber adjacent the plasma 22. The substrate is passive in this process, and can be any part or element on which a coating is desired. For example, the substrate can be a steel mechanical part, such as a bearing, on which the reactive sputtering process deposits a layer of titanium nitride as a hard wear coating. As another example, the substrate can be a semiconductor wafer on which a dielectric layer such as aluminum oxide or nitride is deposited.

The sputtering arrangement can also incorporate suitable 65 means for removing heat from the target.

Actual deposition of a film requires ignition of the plasma 22. The plasma is created by applying a voltage to the space

between the anode 18 and the cathode or target 16 high enough to cause ionization of at least a small portion of the sputtering gas, i.e., the argon.

The intense electric field strips outer electrons from the gas atoms, creating anions, e.g.,  $Ar^+$  and electrons. This <sup>5</sup> leads to a steady state which is fed by a steady state applied current from the dc power supply 14.

The positive ions Ar<sup>+</sup> are accelerated by the steady state field towards the target **16** or cathode, where they recombine with electrons in the target. To achieve a sputtering effect, <sup>10</sup> the ions are made energetic enough so that their kinetic energy will knock atoms of the target material off the target **16** when the ions collide with it. The freed target atoms then enter the plasma and react with the reactive gas that is introduced into the chamber. The product of this reaction deposits itself on any available surface, and preferably on the substrate **24**. The reaction product also redeposits on the target **16**, as well as other surfaces in the plasma chamber. Accumulations of insulating deposit on the target can lead to arcing and other problems. <sup>20</sup>

Control of the sputtering process requires control of voltage, current flow, and current density.

Current flow is required for sputtering, and all else being equal, higher current produces proportionately more collisions of the  $Ar^+$  ions onto the target. Therefore a high deposition rate requires higher current flow.

Different target materials require different levels of applied voltage. For example, a gold atom is significantly heavier than an aluminum atom, and therefore requires a much more energetic ion to knock it free from the target. In a typical dc sputtering process that can use either a gold target or an aluminum target, a voltage of about 450 volts is required for gold. There is thus a lower limit at which plasma exists, but no sputtering takes place because the ions are not sufficiently energetic. Each process will have a lower limit on sputtering voltage depending on the target material.

Current density is an important factor and has to be controlled. In the sputtering process it is desirable to maximize the deposition rate but it is also imperative to avoid arc discharges.

The upper limit of deposition rate occurs when the current density of the plasma is in a so-called superglow region. This can be explained with reference to FIG. 2, which shows the  $_{45}$  relation of current density in plasma to the voltage drop across the plasma. This curve is the so-called Passione curve.

In an initial region shown at the extreme left side of FIG. 2, before the plasma is ignited there, is a zero current density 50 and the entire voltage necessary for ignition appears across the gaseous medium. For low current densities, there is a negative-resistance region indicated as the ignition region, where the voltage across the plasma drops with increased current density, until a generally flat region, i.e. a glow 55 region, is reached. As current density increases, a superglow region is reached. Here the plasma becomes more energetic and thus more resistive to the movement of argon ions across it; hence the voltage increases across the plasma. Beyond this region the current density becomes so high that the 60 plasma self-heats the ions to the point that the plasma generates thermal electrons and photons which then cause runaway ionization. This drastically increases the number of ions, i.e., the number of charge carriers in the plasma. The plasma impedance drops, and current flow increases. This 65 produces a sharp negative resistance region leading to an arcing region where the impedance is very small and current

is limited only by the output impedance of the power source. The arcing that is produced here creates deleterious effects on the target, such as pitting, flaking and cracking, as well as localities of extreme heat. Moreover, as soon as arcing occurs, sputtering stops.

Therefore, in a sputtering process, a number of factors come into play: deposition rates are directly related to current flow; applied voltages are determined by the characteristics of target materials; and arc discharging directly follows from excess current density conditions.

The problems that arise from redeposition of insulator reaction products onto the target 16 can be explained with reference to FIGS. 3, 4, and 5. As shown in FIG. 3, the potential difference between the positive anode 18 and the negative target 16 produces electric field lines from the anode to the target. An applied voltage VAPP, typically on the order of about 300 to 500 volts dc, is applied from the dc power supply 14 to the anode 18 and target 16. In this example, the target is made of aluminum, the plasma 22 is formed of argon ions Ar<sup>+</sup>, and oxygen O<sub>2</sub> is introduced as a reactive gas. Initially, the electric field lines are distributed more or less uniformly over the surface of the target, so that the argon ions bombard the target 16 over its entire surface. Wherever the argon ion impacts, aluminum atoms are knocked free by momentum transfer. The argon ions pick up electrons from the negative target 16, and electrons in the plasma are absorbed into the anode 18.

The freed aluminum atoms enter the plasma 22 and react with oxygen in the plasma, forming alumina  $Al_2O_3$ . The alumina molecule is neutral and non-conducting, and deposits itself on any convenient surface which it contacts. Some of the alumina lands on the target 16 and produces as a deposition an insulating layer 26, as shown in FIG. 4.

Because the insulating deposition 26 is a dielectric, i.e., an insulator, the surface that faces the plasma, that is the upper surface in FIG. 4, accumulates a positive change. This deflects the electric field lines around the deposition 26. The ions tend to follow the field lines and thus are accelerated towards the uncoated aluminum areas of the target. The argon ions collide with and sputter aluminum, but do not resputter the Al<sub>2</sub>O<sub>3</sub> material. Consequently, the alumina continues to coat the exposed surface of the target, while the relative area of aluminum metal that is still available for sputtering shrinks. The result is to increase the current density while reducing the overall current flow, thus reducing the deposition rate. Eventually nearly the entire surface of the target 16 becomes coated, and only a small area of the target is uncovered and available for sputtering. This causes the electric field lines to converge on localized spots as shown in FIG. 5. Ions impact these spots at a high current density, leading to arc discharge.

The adverse effects of insulative redeposit on the target can be explained with reference to FIGS. 7 and 8. Under glow or superglow conditions, the plasma 22 can be considered to function as thousands of high-value resistances connected in parallel, with leads positioned uniformly over the face of the target 16. In other words, the plasma can be considered as several thousand-megohm resistors as shown in FIG. 6. As the coating of insulating material develops on the target, the available area on the target is diminished, ultimately to small uncoated gaps 28. The current density at the gaps 28 enters the negative resistance zone of the Passione curve (FIG. 2) and the current path becomes restricted. The effective resistance drops to a very small value, e.g., 0.01 ohm, as illustrated in FIG. 7. Under these conditions, arcing occurs. The above-described problem comes about because the insulating film 26 forms a dielectric, sandwiched between the electrodes formed by the conductive plasma 22 and the conductive target 16, respectively. The top surface, i.e., the plasma side of the insulating film 26, charges up to the applied voltage  $+V_{APP}$  because of charge received from the positively charged argon ions.

Because of electrostatic repulsion between the positively charged ions, the argon ions  $Ar^+$  are steered away from the film-coated areas of the target toward the remaining <sup>10</sup> uncoated metal areas. This raises the current density on those areas. Therefore the total current flow has to be reduced to keep the current density within the superglow range and this of course reduces the deposition rate. Also, because the argon ions are steered away from the coated <sup>15</sup> areas of the target **16**, there is little resputtering of the insulating film **26**. Consequently, the redeposition of the insulating film continues unabated, until the sputtering process has to be terminated.

The zone of insulating film can be considered a capacitor <sup>20</sup> to which a dc voltage is applied. The insulating film **26** on the target **16** charges up to the applied voltage  $V_{APP}$ , when the capacitor is charged, no further current can flow, i.e., the area covered by the insulating film is unavailable for sputtering. Plasma current is directed to the remaining areas and <sup>25</sup> the current density increases to the remaining uncovered gaps **28** on the target. Eventually, the current density will creep up to the arc discharge region of the Passione curve (FIG. **2**) which establishes a limit for reactive dc sputtering.

Another problem that can arise from the accumulation of <sup>30</sup> the dielectric layer **26** is that the dielectric layer may not be strong enough to tolerate the entire applied voltage  $V_{APP}$ . In that case, the dielectric layer will punch through and fail. When this happens the failure region will be flooded with charge causing a radical increase in local conductance, and <sup>35</sup> a consequent increase in local current density. This can produce local arcing, and can bring the entire plasma **22** to an arc discharge condition.

Returning to the Passione curve of FIG. 2, in the glow and superglow regions the plasma 22 has a positive resistance and exhibits stable behavior. The arc discharge region is entered whenever the current density, even for a local area, exceeds the superglow limit. The arc discharge behaves like a short from anode to target, and exhibits a negative resistance so the more that current flows, the lower the resistance becomes. To stop the arc discharge, the current density in the plasma has to drop to the point where it crosses back to the glow or superglow regions.

The most reliable way to do this is to remove all current  $_{50}$  flow as quickly as possible. However, when the current flow resumes, there is nothing to prevent the arc conditions from recurring.

Modern power supplies for dc sputtering often incorporate arc detecting circuitry which shuts the current off when 55 an arc is detected. The current must remain off long enough for the arc current to decay. This can reduce effective net power to the plasma chamber 12.

One attempt to deal with this problem of arc prevention has been the introduction of unipolar pulsing. This technique 60 involves shutting off the power supply, creating pulses at a fixed rate and duty cycle. This produces alternate application of negative and ground voltage to the target, which allows the positively charged dielectric material of the layer **26** to be partly charged, and then partly discharged around a bias point. This reduces the voltage stress on the insulating layer, and does permit some re-sputtering of the insulating layer.

This system can be understood with reference to FIGS. 8A, 8B and 9. As shown in FIGS. 8A and 8B, the plasma chamber can be considered as a capacitor circuit in which the target 16 serves as one electrode or plate, the deposition of insulating material 26 serves as the capacitor's dielectric layer, and the plasma 22 serves as a series resistance 22a and also as the other (positive) electrode or plate 22b of the capacitor. The voltage applied to the target  $V_T$  can be a pulsed dc voltage alternating between the applied potential V<sub>APP</sub> and ground potential, i.e., 0 volts. The anode 18 is considered to be at ground potential. In this prior-art unipolar pulsed power system, the upper plate 22b of the capacitor, i.e., the dielectric layer 26 is permitted to decay back towards target potential. In FIG. 8B, the voltage at the upper capacitor plate is represented as V<sub>CAP</sub>. Typically, the applied voltage is interrupted for periods of 10 microseconds or more with a pulse rate of about 2 kilohertz, i.e., a pulse period of 500 microseconds. This technique is intended to prevent high-voltage build-up on the dielectric or insulating layer 26, but does not actually remove the positive charges. Consequently, even though some resputtering of the layer may occur from ion bombardment, the net effect is simply to slow the rate at which the insulting layer accumulates on the target.

The technique of this invention can be explained with reference to FIGS. 10 and 11. In this technique, pulses 30 of reverse bias voltage  $+V_{REV}$  are inserted onto the negative dc sputtering voltage  $-V_{APP}$ . The effect of this is to bring the target voltage  $V_T$  above ground potential to the reverse bias voltage  $+V_{REV}$ . This lifts the capacitor voltage  $V_{CAP}$ , i.e. the potential of the upper surface of the dielectric layer 26 to a high potential so that the layer 26 absorbs free electrons from the plasma 22 until it achieves ground potential, i.e., corresponding to the potential of the anode 18. This decay is shown at curves 32 in FIG. 11.

As shown in FIG. 10, the reverse pulses create asymmetric bipolar pulsed dc. The positive reverse voltage has excursions of smaller magnitude than the negative sputtering voltage, e.g.,  $+V_{REV}$ =+150 volts vs.  $-V_{APP}$ =-300 to -700 volts. As also shown, the pulse duty cycle is quite small, that is, the pulse width of the positive reverse voltage  $+V_{REV}$  is small relative to its period, e.g., about 2 µsec.

Then the applied negative voltage is resumed at the end of the pulse. This brings the lower electrode of the capacitor, i.e., the target 16 to the negative voltage  $-V_{APP}$ .

The upper, or plasma-facing surface of the dielectric deposition layer 26 is then brought to a very negative potential, more negative than the target potential by the amount  $-V_{PFV}$ .

For a practical example of a reactive sputtering system. e.g., Al to produce Al<sub>2</sub>O<sub>3</sub>, the applied voltage V<sub>APP</sub> can be -400 vdc, and the reverse bias voltage can be +150 volts. In this case, the voltage  $V_{CAP}$  which appears on the upper surface of the layer 26 follows the voltage wave form of FIG. 11. At the onset of applications of -400 volts to the target to the target 16, the dielectric voltage drops to -550 volts. This is more negative than the target potential, and tends to attract the positively charged argon ions towards the accumulations of Al<sub>2</sub>O<sub>3</sub> on the target. Because of the greater potential, i.e. 550 volts, the ions that bombard the Al<sub>2</sub>O<sub>3</sub> are more energetic than those that strike the uncoated target. These more energetic ions are able to free the relatively heavy oxide molecules from the insulating layer 26, and these molecules deposit themselves elsewhere. Thus, the reverse bias pulses serve to clean the target of the insulating deposition, so that sputtering can continue unabated until the target is consumed.

The reverse bias pulses are also effective in removing impurities and inclusions from the target 16. This technique can also be employed to prepare or "condition" an aluminum target which has been exposed to the atmosphere. In a standard aluminum reactive sputtering technique, a separate 5 conditioning phase is required, and this can damage the aluminum target due to pitting and stressing. This preconditioning stress damage can actually produce arcing during a subsequent sputtering process. On the other hand, the reverse bias pulsing technique of this invention sputters off 10 oxides and impurities with little or no damage to the target and without arcing.

A principal benefit of this technique is that it removes sources for arcing, rather than simply interrupting or limiting arcing. This technique permits a reactive sputtering <sup>15</sup> operation to run as close as possible to the high end of the superglow region, which maximizes deposition rates without risk of encountering arc discharge conditions.

In a preferred mode, the reverse bias pulse can be provided at 150 volts, at period of 2 µsec and a pulse repetition rate of 50 KHz. The duty cycle should be kept small so that sputtering is not unduly interrupted. The pulse period should be of sufficient length to allow the dielectric layer the reverse biased, and this period can be between 0.25 µsec and 3 µsec. The pulse repetition rate can be between 40 KHz and 200 KHz. The positive reverse bias voltage is tailored to the particular sputtering operation, as is the negative applied voltage. That is the applied voltage  $V_{APP}$  can be between -300 and -700 volts, and the reverse bias voltage can be between +50 and +300 volts.

A practical embodiment of a reactive dc sputtering arrangement, with a reverse bias pulsing feature of this invention, is shown in FIG. 12. Here the chamber 12 has the target 16 coupled to a negative terminal of the power supply 14 and its anode 18 coupled to the positive terminal of the power supply 14. The substrate 24 is situated within the plasma chamber; argon or another suitable sputtering gas in introduced through a gas conduit 38, while the reactive gas, e.g., oxygen is delivered through a reactive gas conduit 20. The chamber is continuously evacuated by a vacuum pump (not shown).

A reverse-bias pulse arrangement 40 is installed between the dc power supply 14 and the plasma chamber 12. This arrangement includes a dc power supply 42 that is connected in series with a controlled switch 44. The power supply 42 can be a switch mode power supply that is variable up to 300 volts. There is a series pair of inductances 46 coupled between the negative terminal of the power supply 14 and the target 16, and another series pair of inductances 48 coupled between the positive terminal of the power supply 14 and the anode 18. The series circuit formed by the power supply 42 and the switch 44 is connected between the junction of the one pair of inductances 46 and the junction of the other pair 48.

A pulse-width and a pulse-rate control circuit **50** generates signals that actuate the switch **44** to create the reverse bias pulses of suitable duty cycle and pulse frequency. The switch closure times are controllable by the control circuit **50**, and the output voltage of the power supply **42** is also  $_{60}$  controllable, e.g., from 50 volts up to 300 volts.

When the switch 44 closes, the reverse bias power supply 42 applies a positive voltage to the target 16, and applies a negative voltage to the anode 18. Then when the switch 44 opens, the power supply 42 is cut out, and the potential at the 65 target reverts to the negative applied voltage  $V_{APP}$  from the power supply 14.

The elements including the reverse bias pulse arrangement 40 and the pulse-rate and pulse-width control circuit 50 can be made as a compact, self contained unit that can be coupled between a standard power supply 14 and a standard plasma chamber 12.

While this invention has been described with reference to a preferred embodiment, it should be appreciated that a wide variety of embodiments would present themselves to those skilled in the art without departing from the principles of this invention, as defined in the appended claims.

What is claimed is:

1. Asymmetric bipolar pulsed dc reactive sputtering process wherein a target material is sputtered from a conductive target in a plasma chamber and is reacted with a reactive gas in the chamber to form a compound that is then deposited on a substrate, comprising the steps of:

- applying a dc forward sputtering current between said target and an anode in said chamber at an applied forward voltage level that is negative to said target relative to said anode and so that said forward sputtering current causes noble gas ions to impinge upon said target with sufficient energy to free atoms of said target material from said target;
- introducing said reactive gas into said chamber to react with the free atoms of said target material; and
- applying, onto said forward sputtering current, pulses of a reverse voltage to said target that is positive relative to said anode at a suitable pulse rate and pulse width so that accumulations of said compound which have deposited on said target are bombarded by said noble gas ions with sufficient energy to free molecules of said compound from said target;
- wherein said forward applied voltage is between negative 300 and negative 700 volts and said reverse voltage is provided at a substantially smaller level between positive 50 volts and positive 150 volts; and wherein said pulse rate is between about 40 KHz and 200 KHz with said pulse width being between 0.25 µsec and 3 µsec.

2. Asymmetric bipolar dc plasma arrangement for reactive dc sputtering wherein a target material is reacted with a reactive gas to form a compound that is deposited on a substrate, comprising:

- a dc plasma chamber housing a sputtering target formed of said target material and serving as a cathode, an anode formed in said chamber, and means for introducing said reactive gas into said chamber;
- a dc power supply providing to said target a sputtering current at an applied voltage level that causes noble gas ions in said chamber to impinge upon said target with sufficient energy to free atoms of said target material from said target such that the free atoms react with said reactive gas to form said compound; and
- reverse bias means interposed in circuit between said de power supply and said plasma chamber for passing said sputtering current to said target at said predetermined level relative to said anode, and applying to said sputtering current pulses of a positive reverse voltage relative to said anode at a suitable pulse rate and a pulse width so that accumulations of said compound which have been deposited on said target are bombarded by said noble gas ions with sufficient energy to free molecules of said compound from said target, wherein said positive reverse voltage is below about 150 volts and of significantly smaller magnitude than said negative voltage, said pulse rate is above 40 KHz, and said pulse width is between 0.25 microsec and microsec.

**3.** Asymmetric bipolar dc plasma arrangement according to claim **2**, wherein said dc power supply furnishes said negative voltage at a level between minus 300 and minus 700 volts, and said reverse bias means provides said pulses of positive reverse voltage at a level between 50 and 150 5 volts.

4. Asymmetric bipolar dc plasma arrangement according to claim 2, wherein said reverse bias means provide said pulses with their pulse width between 0.25 µsec and 3 µsec, and with the pulse rate between 40 KHz and 200 KHz. 10

5. Asymmetric bipolar dc sputtering process wherein a target material is sputtered from a conductive target in a plasma chamber to be deposited on a substrate, comprising the steps of

applying a dc sputtering current to said target at an applied <sup>15</sup> negative voltage level, relative to an anode in said

chamber, that causes noble gas ions to impinge upon said target with sufficient energy to free atoms of said target material from said target; and

- applying pulses of a positive reverse voltage to said target relative to said anode at a suitable pulse rate and pulse width so that accumulations of insulating compound on said target are bombarded by said noble gas ions with sufficient energy to free molecules of said compound from said target;
- said pulse rate being between 40 KHz and 200 KHz, said pulse width being between 0.25 µsec and 3 µsec, and said positive reverse voltage being between about positive 50 volts and positive 150 volts.

\* \* \* \* \*

# UNITED STATES PATENT AND TRADEMARK OFFICE CERTIFICATE OF CORRECTION

PATENT NO. :5,651,865 DATED :July 29, 1997 INVENTOR(S) :Jeff C. Sellers

It is certified that error appears in the above-indentified patent and that said Letters Patent is hereby corrected as shown below:

Column 10, Line 67, "0.25 microsec and microsec," should read --0.25 microsec and 3 microsec--.

> Signed and Sealed this Seventh Day of October, 1997

Since Tehman

BRUCE LEHMAN
Commissioner of Patents and Trademarks

Attesting Officer

Attest: