Substrate Biasing in Pulsed DC Reactive Sputtering of Dielectrics

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ABSTRACT

DC reactive sputter deposition of dielectrics can be done using dc power pulsed in the range of 5-350 kHz. Although pulsed dc reactive sputtering (PDRS) does not solve the disappearing anode problem, it provides a deposition process without arcing. Pulsing plasma creates unusual conditions at the substrate, and requires a special approach to biasing. In this work, plasma properties, as well as substrate effects under various biasing conditions, were investigated. Deposition of alumina is used as an example of PDRS.

INTRODUCTION

Pulsed DC reactive sputtering is an established technology to sputter-deposit dielectrics [1-7]. This technology, even though it does not solve the disappearing anode problem [8], creates non-arcing conditions for reactive sputtering of dielectrics [7]. The structure of sputter-deposited thin dielectric films depends on the energy delivered to the growing film. This energy is supplied to the film either by heating the substrate and/or by bombardment of the growing film with neutral and charged particles. Heating, required for getting dense structures of various dielectric films, lies very often in the range of many hundreds of degrees C, and is not always desirable.

In reactive sputtering, a growing film is bombarded by energetic (neutralized and reflected from the target) particles, and by much less energetic sputtered atoms of the target material. The intensity of this bombardment is determined by the sputtering conditions, mainly by power and pressure. In spite of apparent simplicity of varying these deposition parameters, it is not necessarily the best way to control the energy flux.

If the substrate is conductive, and the film is either conductive or thin, the growing film is also bombarded by ions that are extracted from the plasma surrounding the substrate and accelerated by a biasing voltage. Ion current to the substrate is determined by plasma density near the substrate, which, in turn, depends on the usually fixed magnetic field configuration of the magnetron (balanced/unbalanced) and the power supplied to the magnetron. The last one is fixed by the chosen deposition rate. Thus, the best way to control the electron and ion bombardment of a growing film is to vary the biasing voltage. The statement is correct if the substrate biasing does not redistribute plasma near the substrate and the substrate is neither an additional anode, nor a cathode of an additional glow discharge.

DC biasing works only for electrically conductive films. In the pulsed DC sputtering of dielectrics, biasing is accomplished by applying pulsed DC power [9-13]. It has been shown that applying pulsed DC biasing improves substantially the quality of various thin films. In spite of many valuable experimental results obtained, several questions remain. What are, for example, biasing parameters such as frequency and phase shift, duty cycles, range of voltage pulses, etc., vs. the same for the magnetron?

Pulsed DC biasing can be considered, in the first approximation, an alternating (positive and negative) DC biasing. It seemed useful, therefore, to include its exploration into our work on the pulsed DC bias. In this first part of our work, the focus was on the effect of biasing on the magnetron plasma, comparison of DC and pulsed DC biasing conditions, and formulation of a number of biasing parameters for pulsed DC reactive sputtering of dielectrics.

EXPERIMENT TECHNIQUE

Experiments were done in a box-coater, equipped with a diffusion pump, at a base pressure of about 2.5×10^{-4} Pa. An HRC-873 planar magnetron (BOC Coating Technology) was modified to have an unbalanced magnetic field (Figure 1). Advanced Energy Industries, Inc. pulsed power supplies were used to power the magnetron (MDX-10 combined with Sparcle-V) and for the substrate bias (MDX-1 and Pinnacle Plus). Power supplies were run in a constant power or voltage modes, power in the range 0.5-2 kW, and voltage in the range 0-300 volts. Frequencies applied ranged from 20 to 350 kHz, with various reverse times. A digital oscilloscope, Tektronics TDS 3034, was used to record cathode and substrate voltages and currents, registered by Tektronics differential voltage probes (P5200 and P5205) and Tektronics current probes (TCP 202 and A6303 with A503 amplifier).

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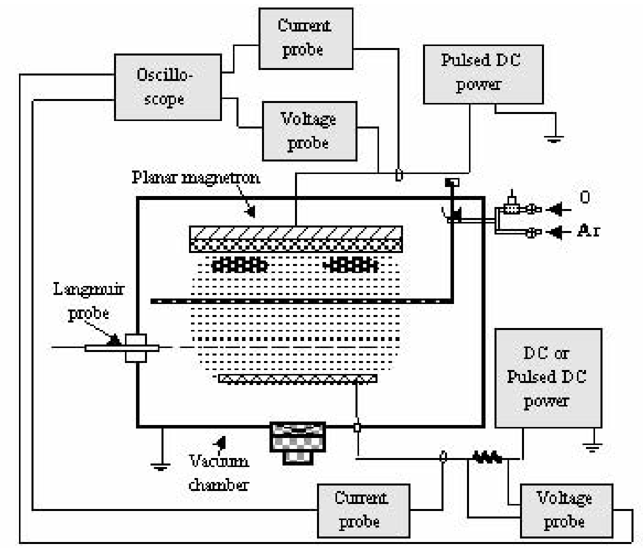

Figure 1: Experimental set-up of deposition system for reactive pulsed DC sputtering with DC and pulsed DC biasing.

Plasma parameters were measured using a Langmuir probe (Scientific Systems SmartProbe). Time-average Langmuir probe measurements were done at 2 cm above the substrate. Substrates used were stainless steel and glass.

Methodology of the non-conducting substrate (Al_2O_3) investigation was the following. A layer of aluminum (2-kA thick) was initially deposited on the glass samples. Subsequently, this layer was connected to the biasing power supply, and layers of alumina deposited on top. Substrate and magnetron currents and voltages were measured at time intervals corresponding to the defined thicknesses of the coating. During this process, the DC or pulsed DC was applied to the growing substrate.

The Al_2O_3 thin films were prepared with a 99.999% Al target and 99.995% pure argon and 99.98% pure oxygen gases, at 4 mTorr pressure. Refractive indices of the Al_2O_3 films were characterized by using AutoEL II ellipsometer at the wavelength of 632 nm. The film thickness was determined via the Tencor stylus profilometer and/or the ellipsometer.

RESULTS AND DISCUSSION

Biasing Conditions

Appropriate biasing conditions could be defined as conditions under which biasing provides controlled ion bombardment of the substrate, but does not influence the magnetron discharge; i.e., it does not redistribute substantially the plasma created in the discharge and used as a source of ions. Such conditions are created when (Figure2):

$$I_s << I_a \approx I_c \quad (1)$$

$$V_a - V_s > 0 \quad (2)$$

and $$|V_c - V_s| \geq 0 \quad (3)$$

where I_a, I_c, and I_s are respectively anode, cathode and substrate currents, but V_a, V_c and V_s are respectively anode, cathode and substrate voltages. The first equation precludes substrate from functioning as an additional anode; i.e., whatever potential is applied to the substrate, its current should be much less than the anode current. For that, the substrate potential should be more negative than the anode's (Equation 2). When a negative voltage is applied to the substrate, it is working as a second cathode (in a Townsend-type discharge), and collecting an ion current on the order of a tenth or less of mA/cm^2. But if the Equation 3 is not satisfied (case of a substrate biased by a very high negative potential) an additional discharge near the substrate could be initiated, and the plasma density near the substrate could be changed.

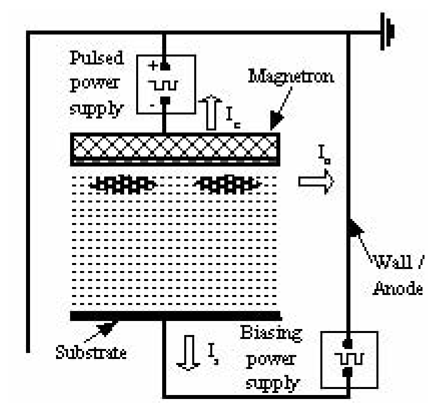

Figure 2: Schematic of the electrical circuitry of a pulsed DC reactive sputtering system.

To verify that in pulsed DC reactive sputtering with a conductive substrate the appropriate biasing conditions exist, we have measured time-averaged plasma parameters (density and electron temperature) in the immediate vicinity of the DC or pulsed DC biased substrate, with a Langmuir probe. Representative results, for plasma density and electron temperature, are plotted respectively in Figure 3 and Figure 4. They show that, within the limit of error, plasma parameters do not depend on the biasing, if the absolute values of the negative (relative to the ground) voltage exceed 30-60 V. So, in the range from -60 V to -300 V of the biasing voltage, we have had appropriate biasing conditions. The plasma density was in the range of (3-5) $x10^9$ cm^{-3}, while electron temperature was in the range 3-5 eV. These values are typical for low-power unbalanced magnetron plasma.

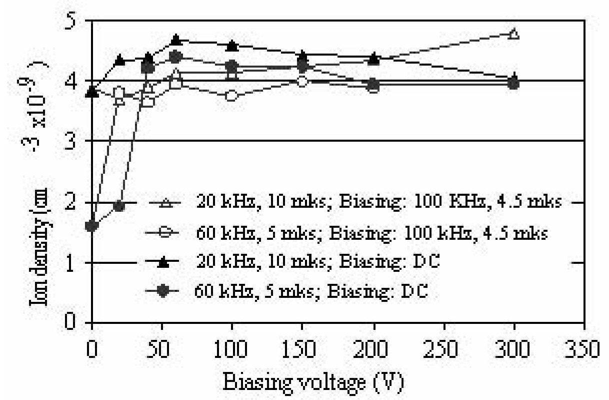

Figure 3: Plasma density measured near the substrate surface versus DC and pulsed DC biasing voltage at different main power pulsing frequency and reverse time.

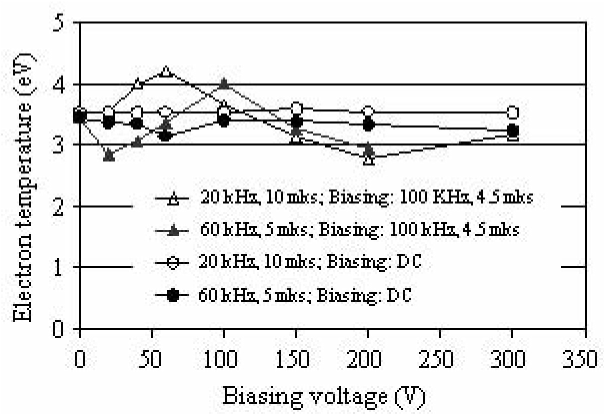

Figure 4: Electron temperature measured near the substrate surface versus DC and pulsed DC biasing voltage at different main power pulsing frequency and reverse time.

Plasma density increases, while increasing the absolute values of negative voltages from zero to about -60 V, were observed in a few cases (Figure 3). At these voltages, the substrate can still be working as an additional anode, and therefore, cause some plasma redistribution near its surface (anode effect) [14].

DC BIASING

Conductive Substrate and Film

Pulsed DC biasing can be considered, to a first approximation, an alternating negative and positive DC biasing. Therefore, it makes sense to start the analysis of the pulsed DC biasing with the analysis of DC biasing while the magnetron is run by pulsed DC power.

Typical oscillograms of cathode current and substrate currents, at different DC biasing voltages, are shown in Figure 5. The oscillograms were recorded using a conductive substrate and conductive film (steel substrate and Al film). During the on-time, when the discharge was stabilized and -100 V was applied to the substrate, the substrate was collecting mainly a steady ion current whose absolute value was much less than the cathode current. These ions were extracted from the plasma surrounding the substrate. As the biasing voltage became less negative (-60 V and then -20 V), the substrate current absolute values decreased. During the off-time, the substrate current also decreased as the biasing voltage became less negative. But during the off-time, at all biasing voltages, there was also a strong initial current peak followed by a steady current decay with one or two time constants. The strong initial peak decayed in about 1 μs or less.

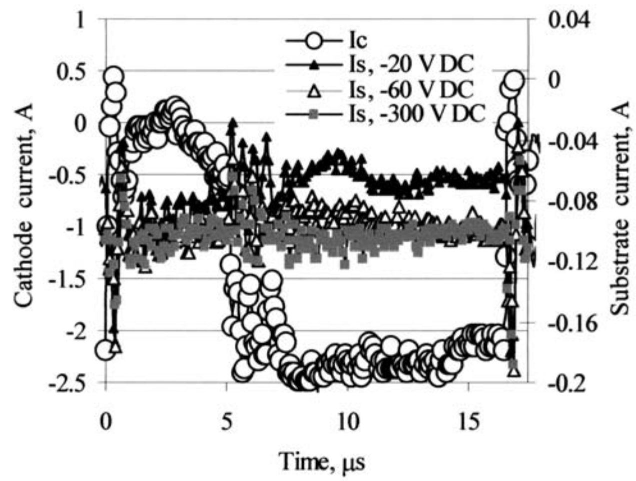

Figure 5: Oscillograms of cathode current and substrate currents recorded at different DC biasing voltages.

During the off-time plasma is decaying and the substrate current is decreasing. The obtained strong initial peak is related to the substrate conditions. At the beginning of the off-time, the substrate becomes suddenly (voltage switching time) the only one negatively charged electrode in the plasma. Therefore, the initial substrate current peak could be very strong, and so the ion current through the substrate, during the off-time, can be even bigger than during the on-time.

The decrease in the absolute values of the substrate current, while the biasing voltage becomes less negative, could be due to a decrease of the ion current itself, or an addition of electron current. At low negative biasing voltages, the negative voltage between the substrate and plasma could be insufficient to stop all electrons from reaching the substrate. Average substrate current measurements vs. biasing voltage support this conclusion (Figure 6). At 0 V (substrate was grounded and worked as an anode) the electron current to the substrate was on the order of 0.5 A. Thus, at low negative voltages, the substrate was still collecting some electron current. In the range from -10 V to about -30 V, a biasing voltage could be found at which the total current to the substrate would be zero (electron current is equal to the ion current). At about -60 V and more negative voltages, the substrate showed characteristics of a cathode in a Townsend discharge.

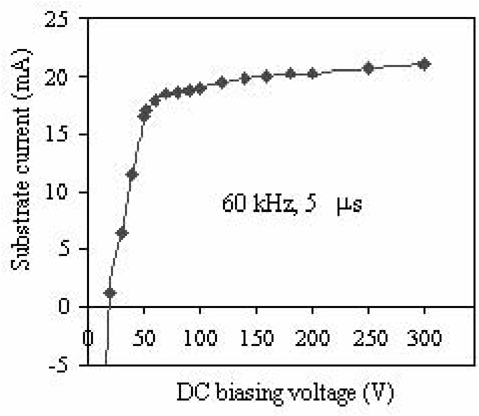

Figure 6: Average substrate current versus DC biasing voltage.

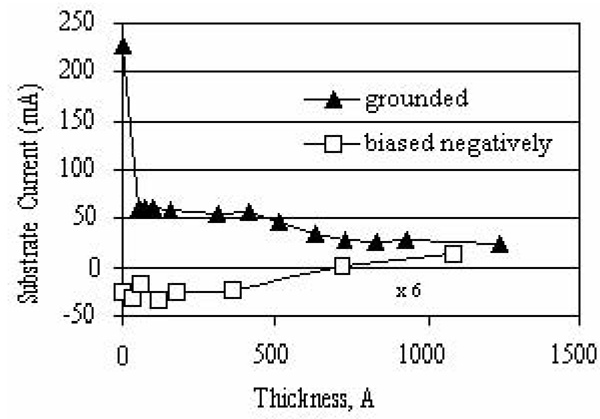

Figure 8: Average substrate currents versus DC biasing voltages for a grounded and biased to -100 V substrates.

The electron current heats the substrate (as it heats the anode). Such substrate heating could be undesirable. If this is the case, the DC biasing voltage should always be more negative than -30 V ÷ -50 V.

Conductive Substrate and Non-Conductive Film

Deposition of an Al_2O_3 film on a conductive aluminum substrate results in covering it with a non-conductive coating. When biasing voltage of -100 V was used, the ion current through the substrate was diminishing with increasing thickness of the alumina coating (Figure 7). Eventually, when a thicker film was deposited, the current through the substrate changed its sign and became mainly an electron current. This could be due to the well-known effect of a self-biasing film-capacitor. Similar variations of the substrate current with the thickness of alumina coating were obtained when an average current was measured (Figure 8). When a thicker film was deposited, the current through the substrate changed its sign and became mainly an electron current. This could be due as well to the effect of a self-biasing film-capacitor.

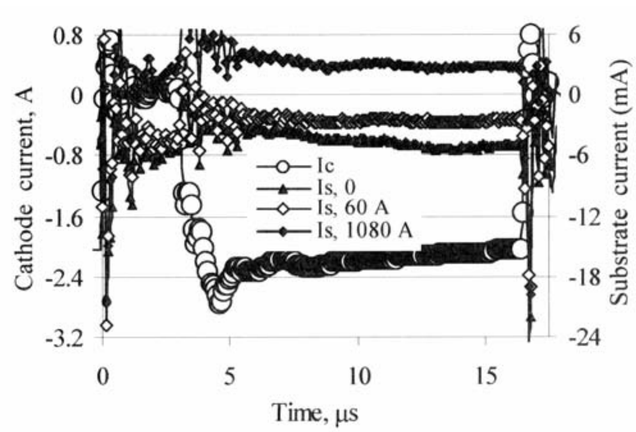

Figure 7: Oscillograms of cathode and substrate currents recorded at different thicknesses of alumina layer on the substrate. DC biasing voltage: -100 V.

PULSED DC BIASING

Conductive Substrate and Conductive Film

Oscillograms of the cathode and substrate currents, using two different pulsed DC biasing frequencies, are shown in Figure 9. In both cases, pulsing DC biasing voltage was applied to a conductive substrate covered with a conductive film (steel substrate and Al film). DC biasing was pulsed from -100 V to about +10V. When the discharge is on (on-time), and the biasing voltage is negative against the ground, an ion current is impinging on the substrate. This current is small in comparison with the cathode current. When a small positive voltage is applied to the substrate, a large electron current passes through it. The substrate is working now as an additional anode having a potential even higher than the "regular" anode (chamber walls). The electron current is heating the substrate in the same way as it is heating the "regular" anode. When the discharge is off (off-time), and the biasing voltage is negative against the ground, a decaying ion current is collected by the substrate. During the off-time, when a small positive voltage is applied to the substrate, a large but decaying electron current passes through the substrate and results in its additional heating up.

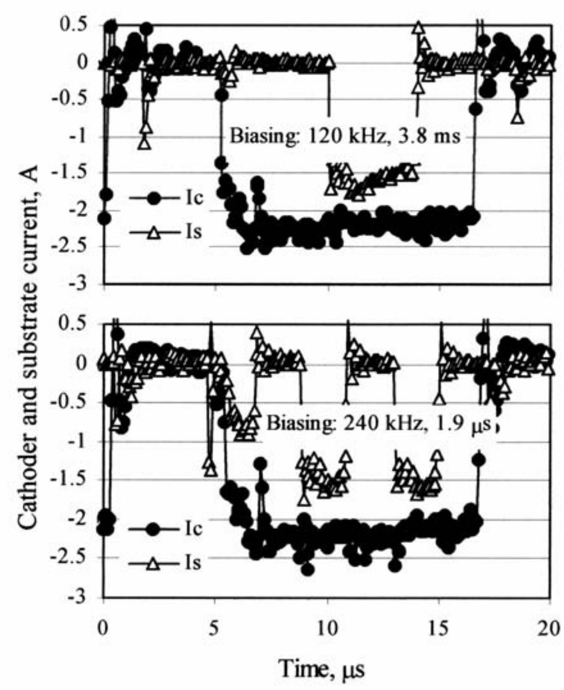

Figure 9: Oscillograms of cathode and substrate currents recorded at different pulsed DC biasing frequencies and reverse times.

If substrate heating has to be reduced, the substrate biasing voltage should be negative during the on-time. It means that pulsed DC voltage applied to the magnetron and pulsed DC biasing should be fully synchronized: frequencies should be the same and the phase shift should be zero. If synchronization is used, during the off-time the biasing voltage could still be negative in order to collect only ion current from the decaying plasma. This depends, however, on the necessity to discharge a dielectric coating if such one is being deposited on the conductive substrate.

Conductive Substrate and Non-Conductive Film

A typical oscillogram, obtained from the pulsed DC biasing of a non-conducting sample, is shown in Figure 10. Substrate currents, at two different thicknesses, are compared in the figure. The same effect, as we have seen in the DC biasing, manifests itself here—values of the current decrease with the increasing thickness of the deposited alumina. They approach the floating substrate conditions of the dielectric-capacitor. This case is still under investigation.

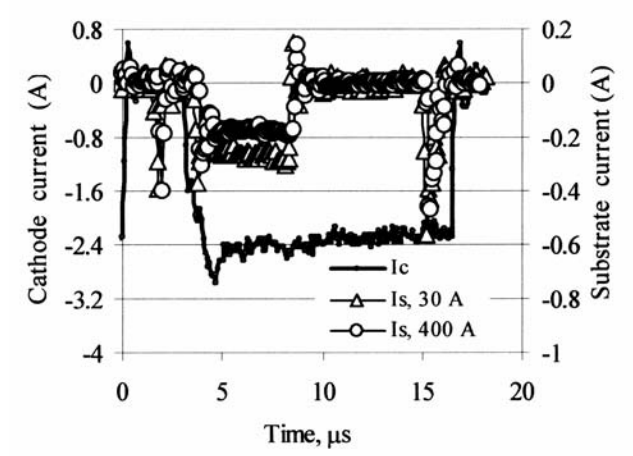

Figure 10: Oscillograms of cathode and substrate currents recorded at different thicknesses of alumina layer on the substrate. DC biasing voltage: -100 V.

CONCLUSION

Experimental data obtained in this work indicate that biasing (DC or pulsed DC), within the above-described limits, does not affect the essential plasma properties, like density and electron temperature, in the vicinity of a substrate.

DC biasing results show that when the substrate bias is negative ($V_s < 0$), a small ion current flows through the substrate. If the substrate bias is positive ($V_s \geq 0$), the substrate is working as an anode and a huge electron current passes through it.

The following applications of pulsed DC biasing in plasma processing were arrived at:

1. If only ion bombardment but no electron heating is desired (which is the case of the temperature sensitive substrates), then:
   - Substrate should be always negative with respect to plasma; pulsing should be between about -20 and -100 VDC
   - Power supplies of the magnetron and the substrate should be synchronized (they should be in-phase, and have the same frequency).
2. If ion bombardment and heating are desired, then:
   - Substrate bias should be pulsed in the way similar to pulsing the magnetron power: between the ground or small positive voltage and about -100 V or higher
   - Synchronization of the power supplies should be avoided, and asynchronous mode, with optimized frequency and duty factor, should be applied.

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