Parameter Optimization in Pulsed DC Reactive Sputter Deposition of Aluminum Oxide

D. Carter, H. Walde, G. McDonough, and G. Roche,
Advanced Energy Industries, Fort Collins, CO

Key Words: Reactive deposition; Sputter deposition; Pulsed DC; Mass flow control

ABSTRACT

Pulsed dc power supplies provide enabling capability to dielectric reactive sputtering applications due to their ability to reduce or even eliminate arcing during the deposition process. As this technology matures there has been increasing interest in the semiconductor, optical and industrial coating industries to use pulsed-dc sputter supplies for difficult to deposit films. The fundamental behavior of reverse-voltage pulsing and its ability to reduce arcing has been studied [1,2] but little work has been published on optimizing the available parameters to provide maximum rate, minimum arc activity and maximized process stability and system utilization. Previous studies have shown that in the range of 20 to 100 kHz there is a strong relationship between arc activity and reverse pulse time. In one study a critical duty factor was identified at a pulsing frequency of 60 kHz where arc free operation was obtained over an extended operating period [1]. This paper intends to expand upon these findings to explore whether an optimum duty factor may be established throughout the available frequency spectrum of today's generation pulsed-dc power supplies. Furthermore, little is understood of how arc intensity, i.e. energy released during the arc, can affect process stability and longevity. Recent technological advances in pulsed-dc power supplies have allowed the range of pulsing frequencies to extend beyond 300 kHz and system architecture now allows for arc intensity discrimination. This study capitalizes on these capabilities as well as the more traditional controls of reverse time (duty factor) and reverse voltage in an effort to characterize their relationship to arc density, and long term process reliability during the reactive deposition of dielectric Al_2O_3.

INTRODUCTION

The sputter deposition of dielectrics has been an active area of research for many years. Because of the issues associated with the deposition of insulating layers, however, alternatives to dc magnetron sputtering are required [3]. High frequency (rf) ac has been used to avoid the issues associated with dc coupling through an insulator and is still one of the most common approaches employed for sputter depositing dielectrics. Unfortunately, cost, low deposition rates and reliability issues plague this approach and deter its acceptance in many high volume production arenas. Reactive sputtering from metallic targets has emerged recently as a preferred technology eliminating the need to use ceramic sources and high cost rf. But while this technique has the promise of high efficiencies [4,5,6,7] the approach presents new control problems for the process or system designer that must be addressed before its potential can be realized.

Process control in reactive sputtering of dielectrics can be problematic. Arc suppression and target condition control issues can result in poor film quality or process stability problems. Due to the nature of these processes, control issues may not be evident at the onset of a deposition. Throughout the process, otherwise conductive surfaces become covered with the dielectric deposit, and the processing environment changes. As a result, the stability experienced at 5 minutes into a run can be lost at 30 minutes or beyond.

This paper looks specifically at arc behavior in extended run reactive depositions as a means to assess process stability. Although there are more aspects to a stable process than arc prevention, arc suppression can be one of the most difficult issues to address but one that can be characterized to identify general trends and relationships transferable to most systems and process architectures.

Arcing typically results from a charge building up on insulating layers formed during deposition. Since dielectric build-up takes place over time, arcing may not be evident early in a process. As surfaces become insulating, however, the propensity for arcing can increase dramatically. The onset of arcing is a major cause for process failure in industrial coating applications. Not only can arcs create damaging defects in delicate thin films but arcs themselves can inject instabilities and disruptions in the power being applied to the sputtering process causing at the least momentary fluctuations and in the extreme a complete loss of power regulation.

Arc suppression has been effectively addressed through the innovation of pulsed-dc power delivery in arc prone processes. It has been shown [1,2] that reverse voltage pulsing on a cathode can be effective in dissipating an accumulated charge on the target and thus reduce or eliminate the creation of arcs during the sputtering process. With recent advances in pulsed power supply technology, a broad matrix of available

© 2002 Society of Vacuum Coaters 505/856-7188
45th Annual Technical Conference Proceedings (2002) ISSN 0737-5921

pulsing conditions, such as pulse frequency and off times (or alternatively, duty factor) are possible. Optimization of these parameters has been studied recently [1] but across only a subset of the parameters now available. In the Kelly study, arc free operation was achieved at 70% and lower duty factors up to a pulsing frequency of 100 kHz. With the advent of higher frequency pulsing power supplies, this study intends to determine if this guideline holds at higher frequencies up to and beyond 300 kHz.

Beyond the primary factors of frequency and duty factor, modern day pulsed-dc supplies offer voltage reversal during the "off-pulse". Typically this reversal is on the order of 10% to as high as 20% of the average sputtering voltage. This voltage reversal has the primary function to efficiently "scrub" the target surface of an accumulated (positive) charge that results during the sputtering ("on") portion of the pulse. The advantage of increasing reverse voltage has been debated but little literature exists to prove or disprove its utility. Reverse voltages of 10% and 20% are included in this work in an effort to understand the impact this parameter has on arc behavior.

As discussed, continuous pulsing by itself is not always adequate to prevent arcs from occurring. Therefore, modern pulsed-dc power supplies are also featured with active arc handling circuitry. The purpose of arc handling is to extinguish a detected arc event that the pulsing action did not prevent. Typically arc detection is based on the low voltage characteristic of an arc. Once the power supply senses a rapid fall in target voltage a preset detection limit (typically between 50 and 150V) senses the arc and initiates a reverse voltage pulse in an attempt to quickly extinguish the arc. An arc that is extinguished with a single reverse pulse is typically called a "micro-arc". One that requires multiple reverse pulses to extinguish is called a "hard-arc".

The nature of the arc event itself is an interesting element in the reactive sputtering system. Traditionally, power supply manufacturers have been driven toward "low stored energy" output in order to minimize the energy released in an arc event. In the case where an arc is initiated by a discrete defect or surface imperfection on a target, however, effective elimination of the arc may require the permanent elimination of the defect itself. Following this supposition, a small number of high energy arcs may be more effective at suppressing subsequent events than a greater number of lower energy events. Milde, et al. [8] found arc rates during target conditioning to be strongly influenced by the delay time prior to arc response. To further investigate this behavior, we studied arc rates in an otherwise stable process as a function of response delay. Arc response delay is defined as the amount of time an arc is allowed to persist prior to its elimination. Varying delay time is used here as a means to control the total energy released in an arc event. Here we attempt to determine if there is a benefit to process longevity gained through increased arc energy.

EXPERIMENTAL

Process control

In order to evaluate arc behavior over an extended processing run we needed a technique to provide stable operation of the reactive process for hours at a time. To accomplish this target "condition" had to be controlled through the duration of the process. Target condition refers to the electrical character of the target while it is being sputtered. The electron emission character of the surface is of primary concern since this determines, in part, the sputtering potential and can significantly influence the sputtering efficiency. In the aluminum/oxygen system, a largely metallic aluminum surface sputters at a much higher voltage than a surface that has been converted to a dielectric in an oxygen rich environment.

This target poisoning process is depicted in the characteristic hysteresis curve shown in Figure 1. Here target voltage is plotted against oxygen flow as constant power is regulated to the cathode. As the flow into the reaction chamber increases the target condition and operating voltage change very little as long as the sputtered aluminum production is adequate to getter the majority of the reactive gas. As the introduction rate of oxygen begins to exceed the liberation rate of aluminum, the process quickly becomes unstable and the target poisons, or converts to a dielectric condition. The poisoned aluminum target is characterized by behavior more similar to that of the oxide, causing the discharge voltage to drop dramatically and consequently the deposition rate. The optimum condition for maximum rate and good quality dielectric deposit occurs where the target begins to transition from a metallic to an oxidized state at or near the knee of this curve.

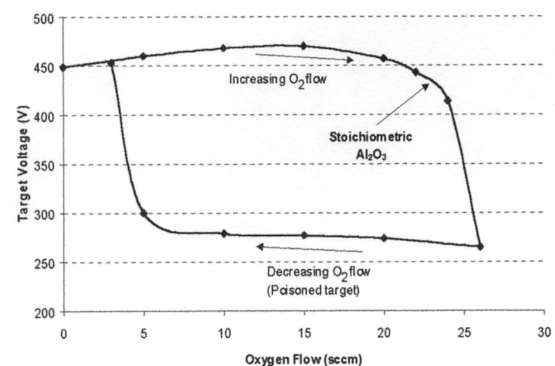


Figure 1: Hysteresis behavior of target voltage in the reactive aluminum oxide sputtering.

Based on this behavior, a closed-loop control system was devised using target voltage as an indicator of target condition. This technique has been discussed elsewhere [9] and is very similar to optical emission techniques used in similar studies [1,10]. The key difference in this apparatus is the use of a piezo based MFC as the gas regulating device in place of a fast piezo valve that is typically used elsewhere. The

advantage of using the piezo MFC is its ease of installation and its compatibility with other traditional control electronics. High-speed piezo valves typically require 0 – 150V where the MFC used in this work requires only a 0 – 5 V input signal.

Figure 2 provides a graphical schematic of the vacuum and control system used in our study. The piezo based flow controller is an EMCO Mach1 MFC operated through a PCU04 PID controller manufactured by Von Ardenne. The target voltage measured by the power supply is routed to the PCU04 and compared to a pre-defined set point. The control signal input for the Mach1 MFC is then provided in the form of a 0 – 5 V output from the PCU04.

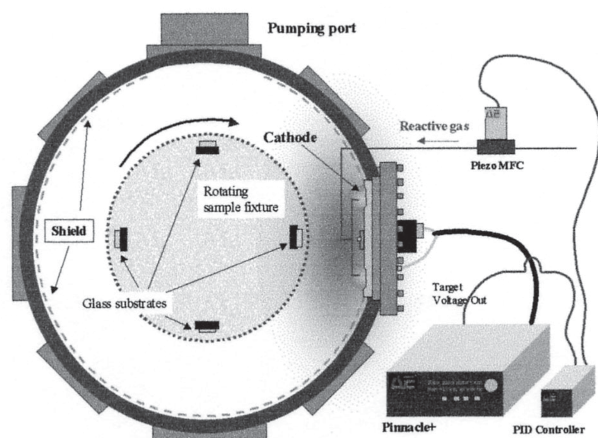


Figure 2: Deposition system schematic.

Figure 3 shows a result of the active control loop, beginning at time 0 when the process was started in the metallic mode. At approximately 30 seconds the target was poisoned with an excess flow of oxygen. This set the metallic and dielectric voltages used to calculate the turn-down set point. In this case a 50% turn-down was calculated and after a brief cleaning period in metallic mode the closed loop control was initiated at time ~ 90 seconds. At this point oxygen was introduced into the chamber at a pre-defined flow level and the PID control loop became active regulating the flow in order to bring the target voltage to the set-point value. After a momentary overshoot, the process then became stable at approximately 110 seconds. For the following studies the target voltage was set to either 25% or 50% "turn-down" defined as a proportion of the full-scale drop in target voltage from fully metallic condition to fully poisoned condition.

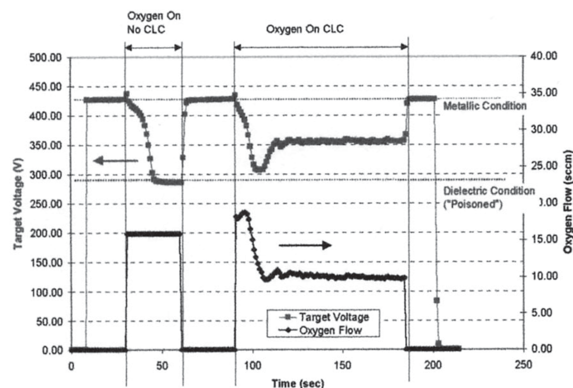


Figure 3: Example of process operating under closed-loop-control (CLC).

Method

All tests were performed in an open volume cylindrical (~30" dia. x 18" deep) vacuum chamber equipped with a Leybold Turbovac 1000 turbomolecular pump backed by an Edwards EH250/E2M40 blower. A six-inch aluminum (99.995%) target mounted on a Torus 10 balanced-field magnetron served as the sputter source. Power to the cathode was applied using an Advanced Energy Pinnacle*plus*™ 5-kW pulsed-dc power supply with a pulsing frequency range from 0 to 350 kHz and pulse reversal times from 0.4 to 5.0 microseconds.

Argon for the sputtering process was fed to the chamber via a dedicated gas line positioned behind the vacuum chamber shielding. Oxygen was delivered through a dedicated delivery manifold distributing the reactive flow slightly toward the surface of the target. Argon flow was regulated using a Unit 1200 series mass flow controller.

All tests were performed at 3.5 mTorr chamber pressure after the system was pumped to a base less than 5.0×10^{-6} Torr. Arc counts were downloaded directly from the Pinnacle*plus*™ using a proprietary logging routine. Arc trip limits were set to 50V unless otherwise stated. In-situ deposition rates were measured using a Sycon STM-100 quartz crystal deposition monitor. Films deposited on cleaned glass substrates were measured on a Tencor P200 profilometer while optical properties were measured on a Gaertner L116 ellipsometer and a Filmetrics F20 spectrometer. Current and voltage applied to the cathode were monitored using a Tektronix TDS 3054 digital oscilloscope.

Table 1 gives the matrix of pulsing conditions tested. We refer to on-time as the portion of a pulsing period during which the voltage is negative or during which sputtering takes place. Conversely, the off-time is the portion during which the voltage is reversed or positive. We define duty factor as the on-time divided by the period of the entire pulse. Not all duty factors are available for all frequencies. Maximum reverse

time is 5 microseconds, minimum reverse is 0.4 microseconds. Minimum duty factor is limited to 55% to 60%. In all cases the process was initiated in the metal mode, and oxygen was introduced to bring the target to the 25% turn-down condition. The deposition was then allowed to run for 2 hours minimum with some tests running for 5 hours or more. Arc counts were logged throughout the run and tabulated or charted accordingly.

Table 1: Pulsed-dc Frequency matrix tested.

	Frequency (kHz)				
D.F.	40*	60	150	250	350
Range	Reverse Times (μs)/Duty Factors Tested				
95%+		0.4/98%	n/a	n/a	n/a
90-94%	2.5/90%	1.0/94%	0.4/94%	0.4/90%	n/a
85-89%		2.0/88%	1.0/85%		1.0/ 86%
80-84%	5.0/80%	3.0/82%			
75-79%	n/a	4.0/76%	1.4/80%		
70-74%	n/a	5.0/70%	2.0/70%		
<70 %	n/a	n/a			

The effects of reverse voltage were studied at 50 and 100 kHz at duty factors intended to produce a significant arc rate based on the frequency/duty factor matrix. Arc data were collected over a ten-minute period for each condition tested allowing for average arc rates to be calculated and plotted for each condition (see Table 2).

Table 2: Reverse voltages tested.

	Frequency	
50 kHz	100 kHz	
Reverse Time (us)	Reverse voltage tested	
0.4	10%, 20%	10%, 20%
0.5	10%, 20%	10%, 20%
0.6	10%, 20%	10%, 20%
0.7	10%, 20%	10%, 20%
0.8	10%, 20%	10%, 20%

Arc rates were compared for different arc energies manipulated by adjusting the delay time in the arc response circuit. Again, conditions favorable for arc production were used, i.e., low frequency and high duty factors. The impact of arc response delay was then plotted for the conditions of 1 microsecond and 5 microsecond delays (see Table 3).

Table 3: Arc response delays tested.

	Frequency
	50 kHz
Reverse Time (μs)	Arc delay tested
0.4	1.0, 5.0 us
0.5	1.0, 5.0 us
0.6	1.0, 5.0 us
0.7	1.0, 5.0 us
0.8	1.0, 5.0 us

All tests in this study were performed at constant 750 watts regulated by the Pinnacle*plus*™. During the course of our testing it was found that initial target condition was a significant factor influencing arc behavior. If the target was not adequately cleaned at the onset of a test we determined that results were inconsistent and non-repeatable. To avoid this, all runs were preceded by a pre-conditioning of the target in argon only for a period of up to 45 minutes. Adequate cleaning was establish by monitoring the target voltage to confirm that no voltage drift occurred over the final five minutes leading up to the initiation of a test pass.

RESULTS AND DISCUSSION

Deposition rates were measured through the target transition to assess the impact of increasing levels of poisoning. Figure 4 shows the influence target poisoning has on deposition rates for the 50 kHz, 1 microsecond reverse condition. Maximum rates are achieved in the metallic and near metallic region while in the dielectric mode the rate falls to less than one-tenth the original value. Based on this, most runs were completed at 25% turn down in order to maximize deposition rates during the process. Films deposited at 60, 150, 250 and 350 kHz at 70% duty factors showed no discernible difference in transmission or refractive index. Refractive index ranged from 1.57 to 1.65. Transmission values were measured from 96 to 99% in the visible range. Measurements were taken on films nominally 4000 Angstroms thick. This apparent insensitivity to frequency and duty factor has been reported elsewhere [1,11].

Cumulative arc counts generated at 60 kHz for duty factors from 70% to 94% are given in Figure 5. It is shown that shorter reverse times associated with higher duty factors prove inadequate for arc prevention. Longer reverse times provide adequate charge scrubbing to allow the process to operate virtually arc free for over 5 hours in the 70% and 76% duty factor conditions.

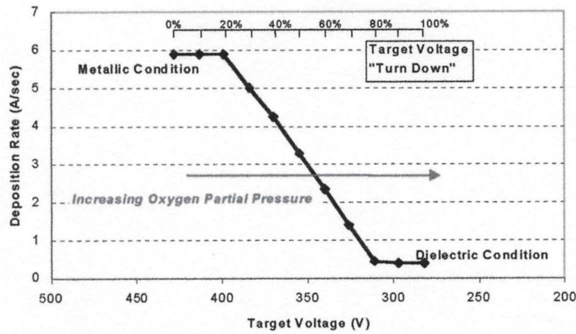
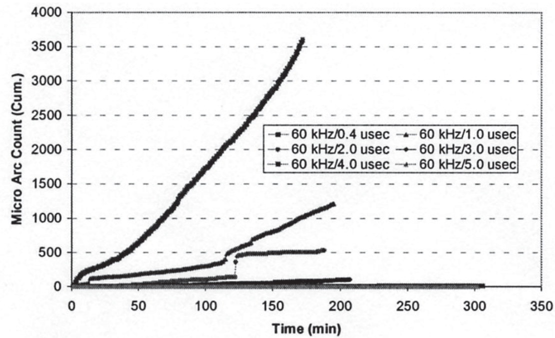
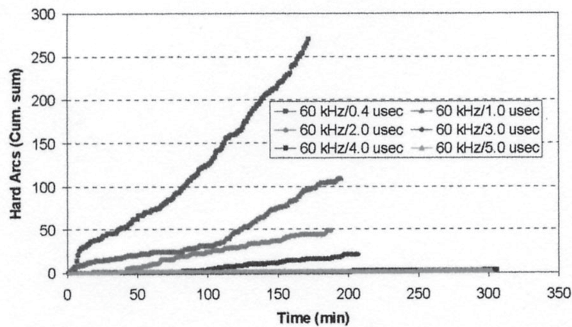


Figure 4: Deposition rates at 50 kHz/95% under closed loop control for increasing levels of target poisoning.



(a)



(b)

Figure 5: Cumulative a) micro-arc and b) hard-arc counts at 60 kHz and varying duty factors.

An explanation for the duty factor (reverse time) dependence is presented in Figure 6. Here the reverse current trace is plotted for 50 kHz, 0.4, 1.0 and 1.6 microsecond reverse times. Upon voltage reversal the current is seen to rise rapidly and peak in this case near a maximum of five amps. Once the "fast" electrons are depleted from near the surface of the target the current collapses to near or even slightly below zero. This transition occurs over a period of 100 to 150 nano-seconds at which time the current is seen to "ring" back to a more positive value and slowly stabilize to a few tenths of an amp. This current drawn to the surface of the cathode continues at a relatively stable value for the duration of the reverse pulse.

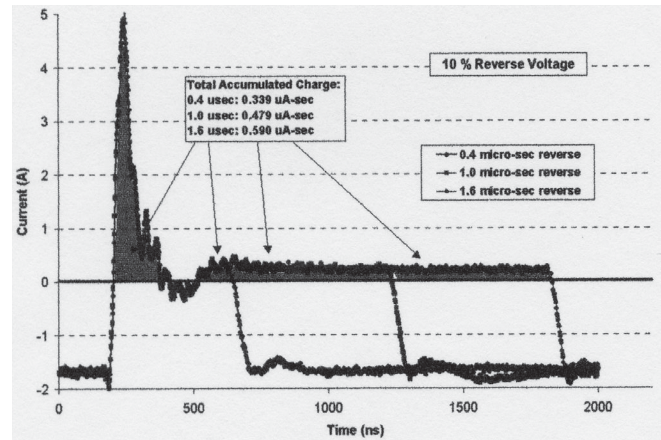


Figure 6: Reverse-pulse current waveforms for 50 kHz, 0.4, 1.0 and 1.6 microsecond reversals (10% reverse voltage) with accumulated charge calculated for each condition.

This reverse current observed through the off pulse was used to approximate a total accumulation of charge brought to the cathode surface during the voltage reversal. The 0.4 microsecond pulse in this case resulted in 0.338 microamp-sec of charge accumulation, while the 1.6 microsecond pulse brought 0.59 microamp-sec of charge to the cathode. This increase in accumulated charge is proposed as the factor driving the behavior seen in Figures 5a and 5b. Higher accumulated charge provides more adequate "scrubbing" of the target. This is accomplished by increasing reverse time and taking advantage of the residual current draw present through the duration of the off pulse.

Similar behaviors were seen at all frequencies tested with shorter off times producing higher arc counts. Arc counts were tabulated for all runs after two hours of continuous operation. A tabulation of these results demonstrated that the point at which arc-free operation occurs is frequency dependent. Table 4 summarizes these results showing that as frequencies increase the duty factor providing arc free performance tends also to increase. This behavior may be explained in part by previous work [11] indicating a fall in deposition rates with increasing pulsing frequency. Lower deposition rates would result in less buildup and re-deposition on exposed surfaces, potentially leading to less arc activity over a fixed duration run. It is not likely, however, that a fractional decrease in rate alone is solely responsible for the behavior seen. The 90% duty, 250 kHz trial was allowed to operate for over five hours yielding no additional arcs. This suggests other phenomena may be at play such as a disproportional decrease in charge accumulation or enhancement in charge scrubbing brought on at the high frequencies.

Table 4: Summary of arc count results.

	Frequency (kHz)				
40*	60	150	250	350	
Duty factor	Micro-arc count after 120-minute deposition				
95% +		2137	n/a	n/a	n/a
90-94%	200+	528	188	1	n/a
85-89%		132	67	0	1
80-84%	20+	43		Arc Free region	
75-79%	n/a	0	0		
70-74%	n/a	0	0		
<70 %	n/a	n/a			

*40 kHz runs stopped after 60 minutes

Based on the arc count results, reverse voltage and delay time tests were carried out at high duty factor conditions at 50 and 100 kHz to ensure that sufficient arc activity would be present to allow for comparisons.

Figure 7 shows the results generated comparing 10% and 20% reverse voltage (Vrev). For very short reverse times the arc activity is seen to decrease for the 20% Vrev condition. The effect, however, diminishes at longer reverse times, to the point where little influence is seen at the 0.8 microsecond condition.

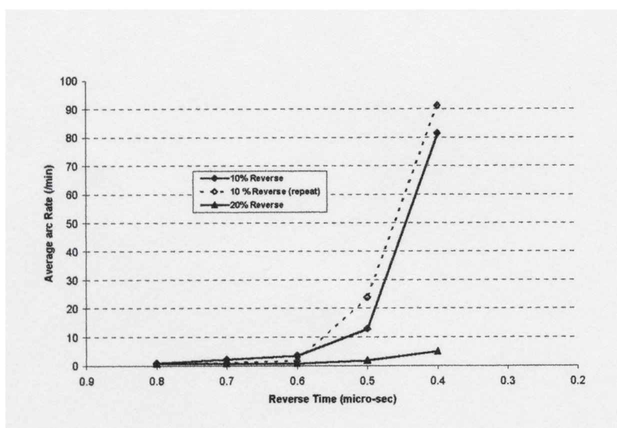


Figure 7: Arc rate at 50 kHz for short reverse times with 10% and 20% reverse voltage.

Figure 8 graphically illustrates this observation, where the magnitude of the arc rate reduction is plotted for both the first trail (1) and the repeated trial (2) seen in Figure 7. For this illustration duty factor is substituted for reverse time on the x-axis. It is noteworthy that the most dramatic effect is only seen at the highest duty factor (shortest reverse time). Below the 98% duty factor condition (0.4 microsecond reverse time) the observed benefit derived from the increased voltage falls quickly to become only a modest improvement at the 96% duty factor. This can then be extrapolated to near zero at 95% and below. Based on this the practical utility of this observed effect is somewhat questionable.

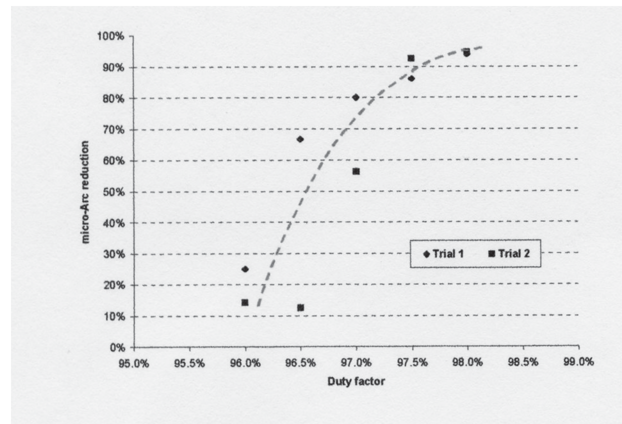


Figure 8: Comparative reduction in arc rates at 50 kHz pulsing for 10% to 20% increase in reverse voltage.

Reverse current waveforms for the 20% Vrev condition are given in Figure 9. Upon comparison to Figure 6 it is apparent that the higher Vrev draws additional current to the cathode through the reverse pulse. The charge accumulated at the cathode during the reverse pulse is calculated to show increases for each condition tested. However, based on the argument above and the chart in Figure 8 the benefit derived is limited to the highest duty factors where inadequate reverse time is provided. For typical duty factors of 60% to 80% adequate reverse time is provided at most frequencies (see Table 4). In these cases the additional current is likely drawn through the conductive regions of the cathode and does not play a significant role in arc suppression.

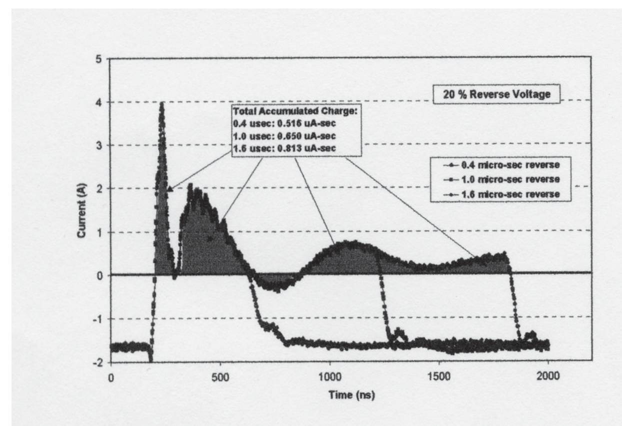


Figure 9: Reverse-pulse current waveforms for 50 kHz, 0.4, 1.0 and 1.6 microsecond reversals and 20% reverse voltage.

Typical arc traces are shown in Figure 10 for 1 and 5 microsecond arc response delays. The same matrix of conditions used for the reverse voltage test was employed here at 50 kHz to generate the delay time results shown in Figure 11. The data plotted are for short cycle (10 minute) runs and are given as average arc rates over the duration of the run.

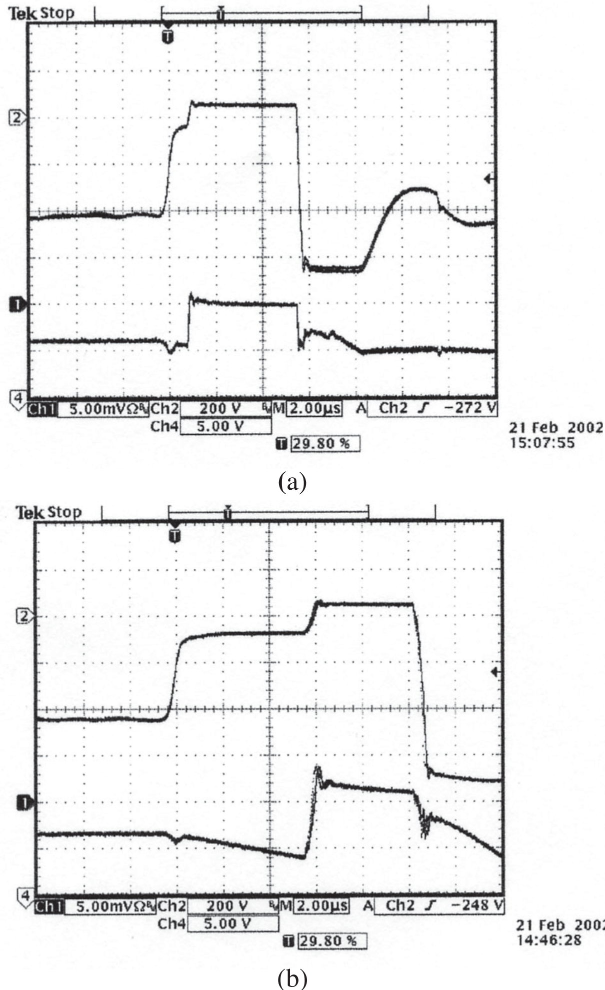


Figure 10: Arc events and response for a) 1 microsecond and b) 5 microsecond delay. Channel 1: Current; Channel 2: Voltage

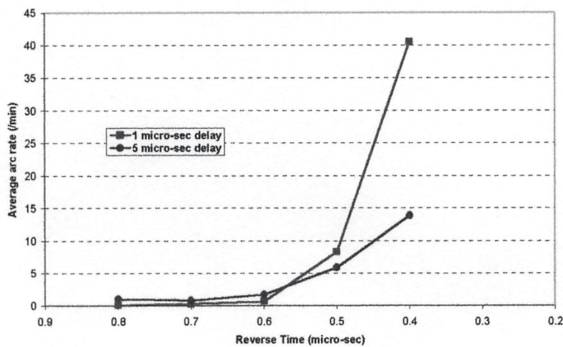


Figure 11: Arc rates for 1 and 5 microsecond arc response delay.

Extending the arc response delay from 1 microsecond to 5 microseconds is shown here to have an effect on the arc rates measured, again most significant at the shorter reverse times. This result is consistent with a greater cumulative discharge per arc event or in the case of localized oxidized regions the persisting arc being allowed to more effectively remove such "defects". Persisting arcs cxan pose the concern of particulate generation. However, in some applications, fewer high-energy events may be an advantageous tradeoff over a higher number of low energy incidents.

Figure 12 shows the cumulative effect of the higher energy arc activity on a more extended time deposition and for a more typical duty factor. Here the 50 kHz, 90% duty factor process was allowed to run for over an hour. Arcs for both the 1 microsecond and the 5 microsecond delay were logged throughout the run and plotted. The result was a rapidly worsening condition for the shorter delay case.

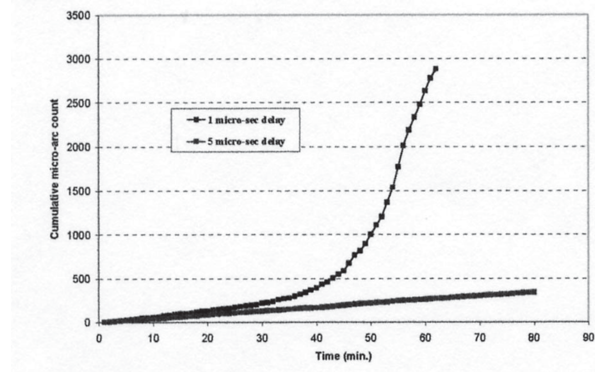


Figure 12: Cumulative arc counts with 1 and 5 microsecond arc delay for extended run at 50 kHz, 90% duty.

CONCLUSION

High rate pulsed-dc reactive aluminum oxide depositions performed in the target transition region are shown to provide deposition rates in excess of 10-times the rate demonstrated from a poisoned target. Tests run under closed loop process control at frequencies from 40 kHz to 350 kHz demonstrate critical reverse times or duty factors exist for adequate charge scrubbing to allow stable arc free operation over long-term depositions. Sustained reverse current (electron current) through the duration of the reverse pulse is presented as the basis for this behavior. Higher frequencies tend to allow for higher duty factors that achieve arc free operation. For the system and conditions tested here, a duty factor of less than 80% was required at 60 kHz to provide a process that was arc free while the 90% duty factor at 250 kHz allowed for over 5 hours of arc free reactive sputtering. Lower deposition and re-deposition rates at higher frequencies (11) are suggested, in part, as the cause for this result.

Increasing reverse voltage from 10% to 20% of the average forward value is shown to provide an increased current draw to the cathode during the off pulse. For very short off-time pulsing this increase in electron current provides improved charge scrubbing to the target surface resulting in decreased arc rates. The benefit is shown to diminish rapidly with increasing off time to a point where little if any effect is expected below 95% duty factor. Since typical applications operate with duty factors ranging from 50% to 90% this effect is not expected to be beneficial under normal circumstances.

Prolonging the arc response delay from 1 to 5 microseconds is shown to decrease arc rates as well as prevent process degradation over medium-term runs. This effect is attributed to more energy being released during individual arc events making each event more effective at removing localized charge or oxidized or included defects. More study is needed to understand the impact this has on other process attributes such as particulate generation.

REFERENCES

1. P. Kelly, P. Henderson, R. Arnell, G. Roche, D. Carter, "Reactive Pulsed Magnetron Sputtering Process for Alumina Films," *J. Vac. Sci. Technol.,* V18, 6, pp.2890-2896, (2000)

2. A. Belkind, A. Freilich, R. Scholl, *J. Vac. Sci. Technol*., Vol. A17, pp. 1934 (1999).

3. G. Roche, L. Mahoney, *Vacuum Solutions*, V12, 11 (1999)

4. S. Schiller, K. Goedicke, J. Reschke, V. Kirchhoff, S. Schneider and F. Milde, "Pulsed Magnetron Sputter Technology," *International Conference on Metallurgical Coatings and Thin Films*, April, (1993).

5. W. D. Sproul, "High Rate Reactive Sputtering Process Control," *Surface and Coatings Technology*, 33, 73-81, (1988).

6. R.A. Scholl, "Power Systems for Reactive Sputtering of Insulating Films," Advanced Energy Industries Inc.

7. M.K. Olsson, K.K Macak, U. Helmersson and B. Hjorvarsson, "High Rate Reactive dc Magnetron Sputter Deposition of Al2O3 Films*", J Vac Sci Technol*, A16 (2), Mar/Apr, (1998).

8. F. Milde, D. Schulze, G. Teschner, P. Wiedemuth and T. Rettich, "Experience with High Power DC Supplies with Fast Arc Suppression in Large Area Coating," 44th Annual Technical Conference Proceedings of the Society of Vacuum Coaters, April, 2001.

9. L. Lou, G. McDonough, H. Walde, D. Carter, G. Roche and R. Scholl, "Closed Loop Controlled Reactive Dual Magnetron Sputtering*," American Vacuum Society Conference*, Fall 1998.

10. P.J. Kelly and R.D. Arnell, "The Control of the Structure and Properties of Oxide Coatings Deposited by Pulsed Magnetron Sputtering," *Advanced Coatings and Surface Engineering Laboratory*, Fall Meeting (1998)

11. D. Carter, G. McDonough, L. Mahoney, G. Roche and H. Walde, "Deposition Behavior and Film Characteristics of Aluminum Oxide Deposited using High Frequency Pulsed-DC Magnetron Reactive Sputtering," *46th International Symposium of the American Vacuum Society,* October 2000.