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Samsung Electronics Co., Ltd. v. Demaray LLC
Samsung Electronic's Exhibit 1059
Exhibit 1059, Page 1

Reactive pulsed magnetron sputtering process for alumina films

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The pulsed magnetron sputtering (PMS) process is now among the leading techniques for the deposition of oxide films. In particular, the use of pulsed dc power has transformed the deposition of dielectric materials, such as alumina. The periodic target voltage reversals during the PMS process effectively discharge poisoned regions on the target. This significantly reduces the occurrence of arc events at the target and stabilizes the deposition process. Many researchers have now shown that pulsed dc reactive magnetron sputtering can be routinely used to produce fully dense, defect-free oxide films. Despite the success of the PMS process, few detailed studies have been carried out on the role played by parameters such as pulse frequency, duty cycle, and reverse voltage in the deposition process. In this study, therefore, alumina films were deposited by reactive pulsed dc magnetron sputtering. Operating conditions were systematically varied and the deposition process monitored throughout. The aim was to investigate the influence of the pulse parameters on the deposition process, and the interrelationships between the occurrence of arc events and the parameters chosen. As a result of this investigation, optimum conditions for the production of high-quality alumina films under hard arc-free conditions were also identified. © *2000 American Vacuum Society.* [S0734-2101(00)04806-5]

I. INTRODUCTION

Since the initial development work in the early 1990s,[1–4] the pulsed magnetron sputtering (PMS) process has become established as one of the leading techniques for the deposition of oxide films. In particular, the use of pulsed dc power has transformed the deposition of dielectric materials, such as alumina.[1–3,5–9] The process itself has been well described in various review articles,[3,6,8–14] and no repetition is required here. It is sufficient to state that pulsed dc reactive magnetron sputtering offers significant advantages over conventional, continuous dc processing.[14] If the magnetron discharge is pulsed in the bipolar mode (see Fig. 1) at frequencies, usually, in the range 10–200 kHz, the periodic target voltage reversals effectively discharge poisoned regions on the target. This significantly reduces the occurrence of arc events at the target and stabilizes the deposition process. Many researchers have now shown that pulsed dc reactive magnetron sputtering can be routinely used to produce fully dense, defect-free oxide films. All stoichiometries are available,[5,6,8] arc events are suppressed,[1–3,6–9,15–17] deposition rates can approach those obtained for metallic films,[2,3,7,15,16] and in dual-cathode systems, very long-term (>300 h) process stability is attainable.[18,19] As a consequence, very significant improvements have been observed in the structure,[5,7,8] hardness,[7,8] and optical properties[6,13] of PMS alumina films, compared to dc sputtered films.

The target voltage wave form during asymmetric bipolar pulsed dc sputtering is shown schematically in Fig. 1. Referring to Fig. 1, the critical parameters which make up the wave form are the pulse frequency, duty factor, and reverse voltage. Duty factor is the relative proportion of the pulse cycle made up of the ‘‘pulse-on’’ period, when the target voltage is negative and sputtering is occurring. The reverse voltage is the nominal positive target voltage achieved during the ‘‘pulse-off’’ period, often expressed as a percentage of the mean-negative voltage during the pulse-on period. The schematic wave form in Fig. 1 shows a pulse frequency of 100 kHz, with a duty factor of 80%, and the reverse voltage set at 20% of the pulse-on voltage. In practice, this ‘‘square’’ wave form is not achieved due to the inherent characteristics of the plasma and the power delivery system, with both positive and negative voltage overshoots being observed.[20] These artifacts can be clearly seen in Fig. 2, an oscilloscope trace of the target voltage wave form obtained when actually operating under the conditions defined previously.

Reference has already been made to the many examples in the literature of the success of the PMS process. However, as yet, few detailed studies have been published on the role played by the pulse parameters in the deposition process. Belkind, Freilich, and Scholl,[9,10] derived an expression showing that the critical pulse frequency for arc-free operation depends on the discharge current and the pulse-off time. Although not explicitly stated, their study indicates that, for a given discharge current, the duty factor is actually the most critical parameter in establishing arc-free conditions. Also, these studies did not consider time-dependent effects, since arc counting was only carried out for 3 min per run. In situations where, during each pulse-off cycle, the parameters selected only partially discharge the poisoned regions on the target, a residual charge will accumulate until, eventually,

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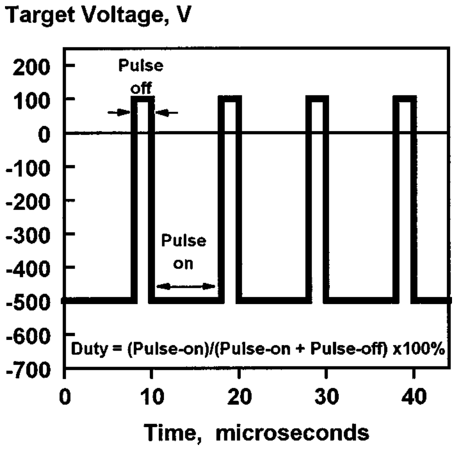

FIG. 1. Schematic representation of the target voltage wave form during asymmetric bipolar pulsed sputtering (pulse frequency=100 kHz, reverse time=2 μs, duty=80%, and reverse voltage=20%).

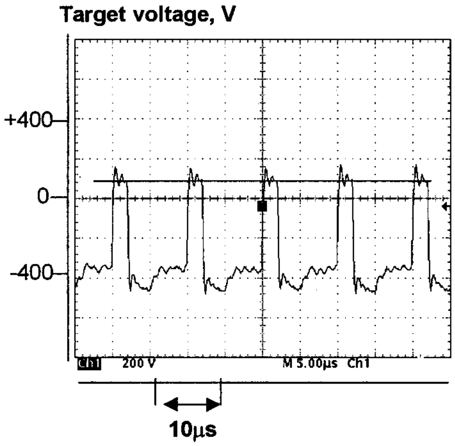

FIG. 2. Oscilloscope trace of the target voltage wave form when operating in asymmetric bipolar pulsed mode at 100 kHz (80% duty and 20% reverse voltage).

arcing occurs. Thus, conditions which appear to prevent arcing at the beginning of a deposition run can prove ineffective as the run progresses.

In this study, alumina films were deposited by reactive pulsed dc magnetron sputtering. Operating conditions were systematically varied and the deposition process monitored throughout. The aim was to investigate the influence of the pulse parameters, such as pulse frequency, duty factor, and reverse voltage, and the interrelationships between the occurrence of arc events and the parameters chosen. As a result of this investigation, optimum conditions for the production of high-quality alumina films under hard arc-free conditions were also identified.

II. EXPERIMENT

The commercial interest generated by the PMS process has led to the development of new power delivery systems. These include ac supplies, single- and dual-channel pulsed dc supplies, and pulse units which can be connected in series with the output from standard dc magnetron drivers. This article concentrates on the use of this latter type of system, in which the magnetron discharge could be pulsed over the frequency range 1–100 kHz. Parallel studies are also being made of the latest generation of pulsed dc supply which extends the maximum pulse frequency up to 350 kHz.[21,22]

The dc power supplies used in this study were the Advanced Energy MDX and Pinnacle magnetron drivers. These power supplies were used in conjunction with the Advanced Energy Sparc-le V pulse unit. The Sparc-le V unit allows the pulse parameters to be varied over the following ranges; frequency: 1–100 kHz, reverse time: 1–10 μs, and reverse voltage: 10%–20%. The dc supplies were operated in current regulation mode.

The Sparc-le V unit allows both hard arc and microarc events to be monitored. Hard arcs are generally considered to be a discharge which takes place between a region on the cathode and an earthed surface, whereas microarcs are discharges between different sites on the cathode. While microarcs can normally be tolerated, hard arc events are extremely detrimental to the deposition process.[3,8] Thus, in this study only the incidence of hard arcs was monitored.

The work performed here was carried out in a Teer Coatings Ltd. UDP 450 closed-field unbalanced magnetron sputtering rig, which has been described in detail elsewhere.[7,8] Alumina films were deposited by reactive unbalanced magnetron sputtering from a 99.5% pure Al target. In all cases the base pressure was $<2\times10^{-5}$ mbar, the argon flow rate was adjusted to give a chamber pressure of 2×10^{-3} mbar prior to deposition, and the target current was set to 6 A. The target was precleaned with the substrates shuttered, but no sputter cleaning of the substrates themselves was carried out. In fact, the substrate holder was allowed to float electrically throughout. The flow of reactive gas was controlled by an optical emissions monitoring (OEM) system tuned to the 396 nm line in the Al emission spectrum. An OEM turn-down signal of 25% was used for all depositions, i.e., reactive gas was allowed into the chamber until the OEM signal had fallen to 25% of the initial 100% metal signal. A feedback loop then maintained the OEM signal at this value for the duration of the deposition run, which was typically 90 min. Previous experience had shown that such conditions would produce stoichiometric Al_2O_3 films.[8]

Figure 3 shows the characteristic hysteresis behavior of this system as the oxygen flow rate is varied. As the oxygen flow is increased initially, the target voltage rises slightly. Operating in this ''metallic'' regime could result in the formation of a substoichiometric aluminum oxide film. At a flow rate of approximately 13 sccm of oxygen, the target poisons rapidly and the negative target voltage falls from 395 to 250 V. The target then remains poisoned until the O_2 flow rate is reduced to <4 sccm. Operating in the ''poisoned'' regime would produce stoichiometric films, but at very much reduced deposition rates. The OEM system allows control to be maintained at any point on the hysteresis curve. Figure 4 shows the relationship between target voltage and the OEM setting, expressed as a percentage of the 100% metal signal. As can be seen, operating at a turn-down signal of 25%

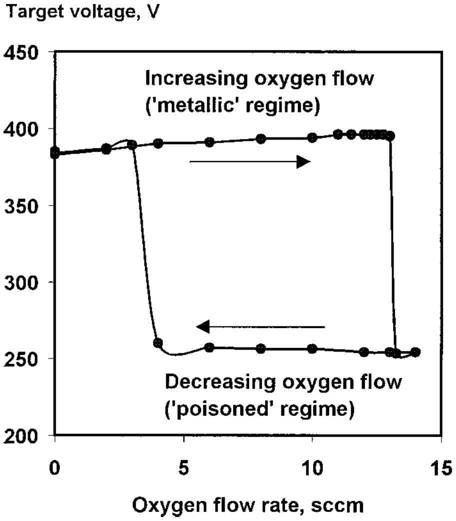

FIG. 3. Hysteresis behavior displayed during reactive sputtering of alumina.

maintains the target between the metallic and poisoned regimes in a ''partially poisoned'' mode. This allows stoichiometric Al_2O_3 films to be deposited at acceptable rates.

The first stage of this investigation was to deposit a series of alumina films under systematically varied conditions using the Pinnacle/Sparc-le V combination referred to above. For each run, the total number of hard arcs detected by the Sparc-le V was recorded. The film properties were then investigated, and the effectiveness of the deposition conditions at arc suppression was considered. The Taguchi method[23] was used to design this experiment. This method utilizes fractional factorial arrays which are designed to optimize the amount of information obtained from a limited number of experiments, and, as such, it is a very efficient experimental technique. The Taguchi L9 array was selected, which allows up to four factors to be varied at three levels, although only three factors were actually used. The factors chosen were pulse frequency (at levels 20, 35, and 50 kHz), reverse time (1, 5, and 10 μs), and reverse voltage (10%, 15%, and 20% of the nominal sputtering voltage). This range of frequencies was chosen because the Sparc-le V limits the maximum reverse time which can be selected at frequencies greater than 50 kHz. Higher frequencies were explored in a second array. The initial experimental array is summarized in Table I.

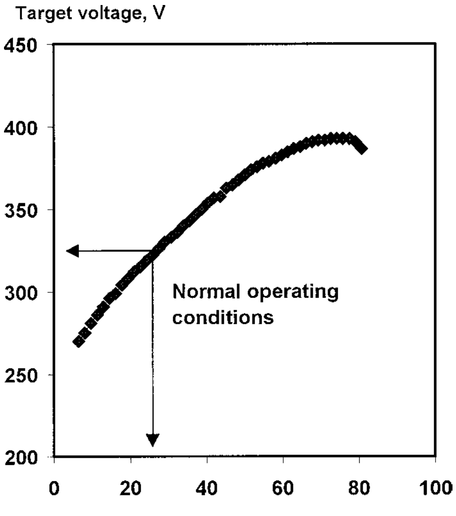

FIG. 4. Relationship between optical emission (OEM) signal and target voltage during reactive sputtering of alumina.

TABLE I. Experimental Taguchi L9 array for the investigation of alumina films.

Run No.	Pulse frequency (kHz)	Reverse time (μs)	Reverse voltage (%)
1	20	1	10
2	20	5	15
3	20	10	20
4	35	1	15
5	35	5	20
6	35	10	10
7	50	1	20
8	50	5	10
9	50	10	15

The alumina films were deposited onto precleaned glass substrates which were subsequently sectioned for analytical purposes. The coating structures were examined by scanning electron microscopy (SEM), with the thickness of each coating being measured from fracture section micrographs. Deposition rates were then calculated from these measurements. The composition of the coatings was determined using a JEOL JXA-50A microanalyzer equipped with WDAX. A high-purity aluminum standard was used in the analysis, with oxygen content being determined by difference. X-ray analyses were carried out using a Philips system, operating in θ–2θ mode (Cu$K\alpha$ radiation), and the resistivity of the coatings was measured using a four-point probe.

Following this, a second array of experiments was carried out. In this case, coatings were deposited over an extended range of pulse frequencies, up to 100 kHz. Also, the MDX magnetron driver was used as the dc supply to allow comparison with the Pinnacle unit. Deposition runs were repeated under, otherwise, identical conditions, but at different levels of duty factor. Care was taken between runs to sputter clean the target, such that all runs started with the target in a similar condition. Run times were varied to ensure that the total pulse-on time was consistent, i.e., the total sputtering time was constant. The reverse voltage was fixed at 20% of the nominal sputtering voltage. The number of hard arcs displayed by the Sparc-le V was recorded at regular intervals, both to monitor the onset of arcing, and to give the total cumulative number of arc events for each set of conditions. The coating structures and properties were investigated as for the preceding array.

III. RESULTS

The deposition rates and total number of hard arcs recorded during each of the Taguchi array runs are listed in Table II. The deposition rates have been normalized to target current to give the rate per minute, per A. The maximum

TABLE II. Taguchi L9 array data table.

Run No.	Duty factor, %	No. of hard arcs recorded	Coating thickness (μm)	Normalized dep'n rate (nm/min/A)
1	98	>10 000	4.5	10.0
2	90	1823	3.0	9.3
3	80	5	1.4	4.6
4	96.5	>10 000	2.0	6.5
5	82.5	492	4.0	8.8
6	65	0	0.75	2.3
7	95	>10 000	3.75	11.6
8	75	2754	1.4	4.6
9	50	0	1.8	4.0

number of arcs which can be displayed by the counter on the Sparc-le V is 10 000. Where this value was reached before the end of a run, a value of >10 000 has been inserted in Table II. Also listed in Table II are the duty factors for each run, arising from the array settings of pulse frequency and reverse time. Statistical analyses were carried out on these data using a software package from the American Supplier Institute, entitled ANOVA-TM. This package was used to compute the level averages using deposition rate and number of hard arcs as response variables, i.e., to compute the average response of each variable at each level of each factor. The results of these analyses are shown graphically in Figs. 5 and 7, respectively. It appears from Fig. 5 that reverse time and reverse voltage both have significant, but opposite influences, on deposition rate. In the case of reverse time, this is simply because, as this factor is increased, so the pulse-off time becomes a greater proportion of the total pulse cycle, i.e., the duty factor is reduced and sputtering takes place for a lesser proportion of each cycle. This is illustrated in Fig. 6, which shows the positive correlation between the duty factor and normalized deposition rate (correlation coefficient, $r = 0.77$). Thus, it could be argued that, of the variables investigated, reverse voltage actually has the most significant influence on deposition rate. As reverse voltage is increased from 10% to 20%, the level average for the normalized deposition rate increases from 5.6 to 8.3 nm/min/A, a factor of approximately 1.5 times.

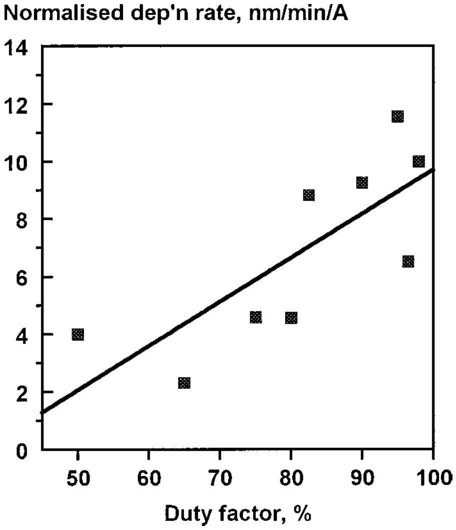

FIG. 6. Relationship between the duty factor and normalized deposition rate for reactive pulsed sputtered alumina films.

The Taguchi analysis using the total number of hard arcs detected as the response variable is shown in Fig. 7. Rather surprisingly, pulse frequency and reverse voltage do not appear to influence the response variable, whereas the level average for reverse time varies from 10 000 to virtually zero as this parameter is increased from 1 to 10 μs. Clearly, varying the reverse, or pulse-off time can have a very significant

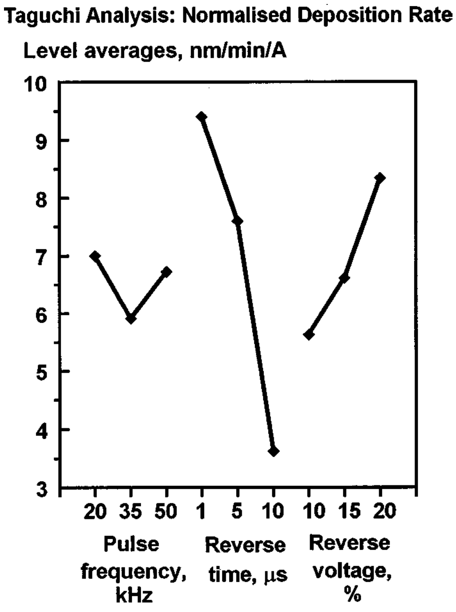

FIG. 5. Taguchi analysis of alumina films, using the normalized deposition rate as the response variable.

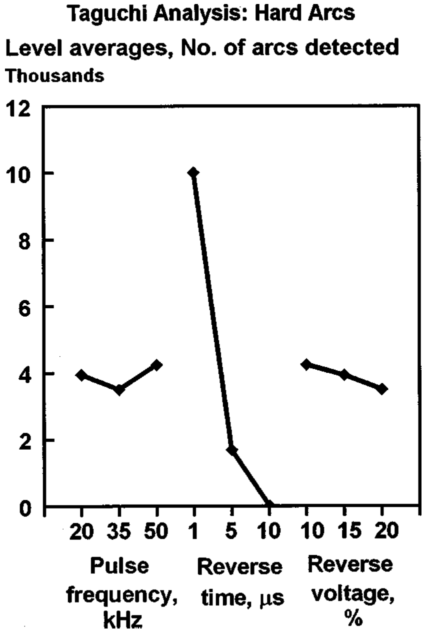

FIG. 7. Taguchi analysis of alumina films, using the total number of hard arcs detected as the response variable.

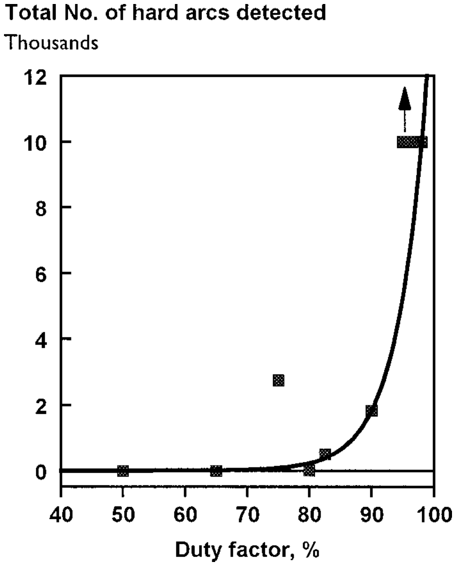

FIG. 8. Relationship between the duty factor and the number of hard arcs detected during the deposition of the Taguchi array alumina films.

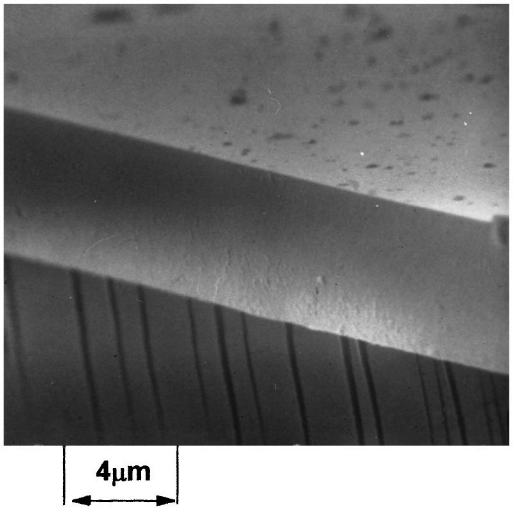

FIG. 9. SEM micrograph of the fracture section of an alumina film deposited on a glass substrate: Taguchi array run 1.

effect on the occurrence of arc events. Again, though, varying the reverse time has the effect of varying the duty factor. Figure 8, therefore, shows the relationship between the duty factor and the number of arc events recorded for the Taguchi array runs. At duty factors of 95% and higher, greater than 10 000 hard arc events were recorded, independent of the pulse frequency and reverse voltage selected. At lower duty factors the number of arc events decreases exponentially until at 65% and below zero arcs were recorded. At intermediate duty factors, arc events were reduced substantially, but not eliminated. There is some scatter in these data; at a duty factor of 75% the arc count was unexpectedly high compared to the counts at 80% and 82.5% duty. Reference to Table I reveals that in the former case the reverse voltage was set at 10% of the nominal sputtering voltage, whereas in the latter case it was set to 20%. It may, perhaps, be the case that reverse voltage exerts a second-order influence on the occurrence of arcs. This suggestion is merely speculative at this stage. Finally, in these analyses the anticipated interaction between pulse frequency and reverse time was not observed. This may well have been due to the limited range of pulse frequencies investigated.

When the films themselves were examined, very little run-to-run variation was observed. By way of example, Fig. 9 is a SEM micrograph of the fracture section of array coating run 1. In this case, as in all other cases, the coatings were fully dense and defect free, with glass-like featureless structures. Compositional analysis, x-ray diffraction, and four-point probe measurements also showed a consistent pattern. In all cases, within the accuracy of the equipment, the compositions were found to be stoichiometric Al_2O_3. X-ray analysis indicated that these coatings were amorphous. This would be expected, as their deposition temperatures did not exceed 250 °C. Finally, four-point probe measurements confirmed that the coatings were highly insulating. All resistivity readings exceeded 20 MΩ cm, which is the maximum value that could be measured by the probe.

To investigate further the parameters influencing arcing, the second array, described earlier, was carried out. Table III lists the pulse frequencies, reverse times, and duty factors investigated (the reverse voltage was set to 20% throughout). Also included in Table III is the total number of hard arcs displayed by the Sparc-le V at the conclusion of the deposition run. The arc count was also monitored at regular intervals during each run. Figure 10 shows the incidence of arc events during a series of runs carried out at 60 kHz pulse frequency. In these runs, the reverse times were varied from 2 to 6 μs, giving duty factors ranging from 88% to 64%. It is again clear from Fig. 10 that there is a strong relationship between the duty factor and the occurrence of hard arcs. As the duty factor is lowered, the incidence of arcing is significantly reduced. Indeed, at 64% duty, hard arc events were completely suppressed for the duration of the deposition run. At other duty factors there was still an initial arc-free period lasting for several minutes. However, in these cases, charge accumulation eventually reached the point where breakdown occurred. Beyond this point the incidence of arcing increased at an exponential rate.

TABLE III. Run conditions and hard arc counts for second alumina array.

Run No.	Pulse frequency (kHz)	Reverse time (μs)	Duty factor (%)	Total hard arcs detected
1	60	6	64	0
2	60	4	76	37
3	60	3	82	385
4	60	2	87.5	5784
5	20	5	90	1545
6	35	5	82.5	492
7	70	4	72	497
8	80	2	84	3998
9	100	2	80	1041

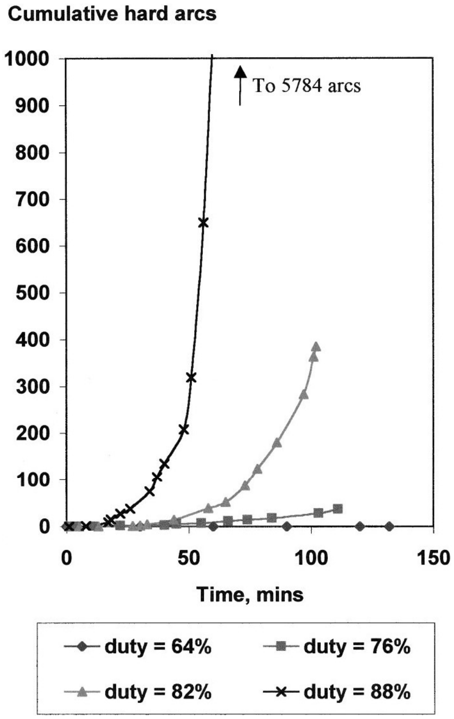

FIG. 10. Influence of the duty factor on the incidence of hard arc events during reactive pulsed sputtering of alumina films (pulse frequency=60 kHz).

Overall, the arc counts for the second array were generally lower than those for the Taguchi array. While there may be a number of reasons for this, the second array runs were all carried out at a reverse voltage of 20%. This may, again, be weak evidence that reverse voltage can influence arc suppression.

To confirm that the events recorded by the Sparc-le V unit were indeed arcs, and not merely artifacts of the arc-counting circuitry, the target voltage wave forms were investigated using an oscilloscope. By triggering the oscilloscope on target current, it was possible to capture actual arc events. Figure 11 shows a typical example. At the onset of the arc event, the discharge voltage collapses and the current rises significantly. In this example, it is at least two pulse cycles before the discharge is reestablished.

Coating structures and properties were investigated for the second array coatings, as for the initial array. Again, all coatings were x-ray amorphous with stoichiometric alumina compositions. An example of the structures of these coatings is given in Fig. 12, which shows a SEM micrograph of the fracture section of the coating deposited at 80 kHz (duty =84%). Interestingly, the high number of arcs recorded during the deposition of each of these coatings does not seem to have had a detrimental effect on the structures, which still appear fully dense and defect free. Once again though, further analysis of these films is planned.

IV. DISCUSSION

A number of interesting points have emerged from this investigation. The first Taguchi array demonstrated that over the range tested pulse frequency alone does not significantly influence deposition rate, or the incidence of hard arcs during the deposition of alumina films. The point has already been made that a greater interaction with other parameters might, perhaps, be expected if the range of frequencies was extended. In the case of deposition rate, reverse voltage is the critical factor at any given duty factor. It has been suggested[12] that this may be a result of preferential target cleaning arising from the bipolar nature of the target voltage. At the end of each pulse-off period the target voltage is reversed. At that instant, ions in the vicinity of the target will be accelerated by the normal negative sputtering voltage, plus the positive pulse-off voltage. Thus, at the beginning of each pulse-on period there will be a flux of ions incident at the target with a higher than average energy. Such a flux would preferentially sputter clean poisoned regions of the

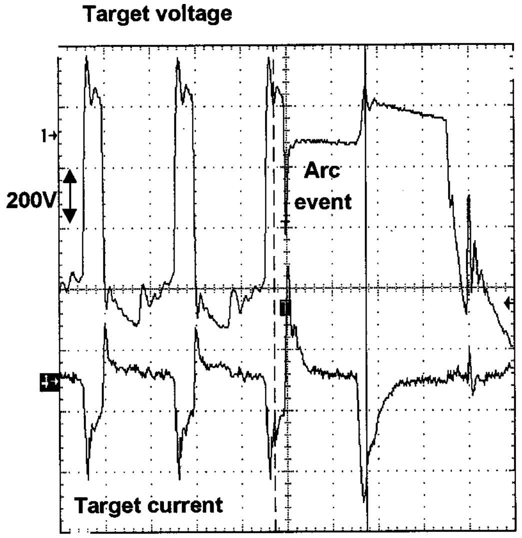

FIG. 11. Oscilloscope trace of the target voltage wave form capturing an arc event during pulsed reactive sputtering of alumina films (pulse frequency =100 kHz and duty=80%).

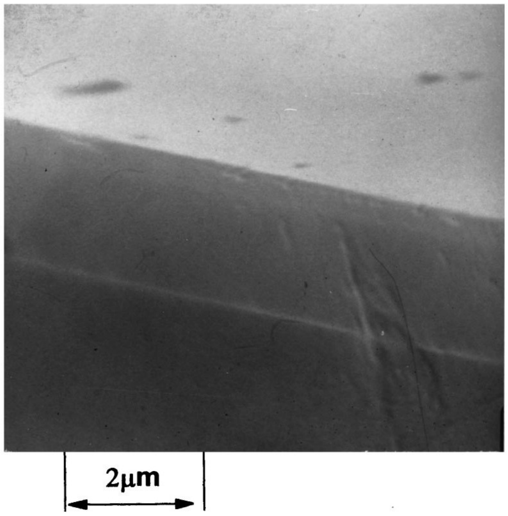

FIG. 12. SEM micrograph of an alumina film deposited onto a glass substrate at 80 kHz pulse frequency and 84% duty.

target. Since the sputtering rate from a metallic target is higher than the rate from a poisoned target, this would have the effect of raising the deposition rate. Clearly, the effectiveness of the target cleaning would be increased as the magnitude of the reverse voltage is increased, giving rise to the trend observed here. Further studies, including the use of a time-resolved Langmuir probe, are planned to investigate this in more detail.

Both arrays have demonstrated the very strong dependence of hard arc events on the duty factor selected. It appears, therefore, that it would be more appropriate to consider a critical duty factor for arc-free operation, rather than a critical frequency (accepting, again, the limited range of frequencies tested). From these experiments, a duty factor of 70% or lower is necessary, independent of pulse frequency, if arc suppression throughout the duration of a deposition run is the prime concern. The second array did show that limited periods of arc-free operation can be achieved at higher duty factors. This finding is in agreement with Belkind, Freilich, and Scholl,[9,10] who also obtained arc-free reactive sputtering of alumina for short time periods at duties greater than 90%. However, this study indicated that such conditions do not remain arc free and that breakdown soon occurs. Once this has happened, the incidence of arcing then increases at an exponential rate. The scatter observed in the data presented here probably reflects the difficulty in replicating target conditions at the beginning of each run. The target condition is certainly an important, but currently unquantified, factor. Finally, on this subject, and underlining the comments made about the target condition, there may also be some evidence to suggest that increasing the reverse voltage can be beneficial in reducing arcing. In a manner analogous to the influence of reverse voltage on deposition rate, the mechanism for this may again be preferential cleaning of the poisoned regions of the target at the beginning of each pulse-on period. This is somewhat speculative at this stage, and any actual effect is very much second order, compared to the duty factor.

The other surprising point to come out of this work is the apparent insensitivity of the coating structures and properties to the incidence of arcing. The alumina films showed a great deal of similarity at the relatively superficial level of examination used here. All coatings were x-ray amorphous with stoichiometric Al_2O_3 compositions. All structures were fully dense and defect free. More sophisticated analysis of these films is planned for the future, including nanohardness measurements and surface roughness measurements.

To summarize the findings of this work, high-quality alumina films can be deposited by pulsed reactive magnetron sputtering over a broad range of conditions. No significant differences in performance were observed between the two dc magnetron drivers used. The optimum conditions to achieve hard arc-free operation throughout the course of a deposition run, using the power delivery systems and deposition conditions employed here, and for pulse frequencies in the range 20–100 kHz, are to select a duty factor of 70%, with the reverse voltage set to 20%.

V. CONCLUSIONS

High-quality defect-free alumina films have been deposited by pulsed reactive magnetron sputtering over a broad range of conditions. A systematic study of the deposition conditions demonstrated that the incidence of hard arcs is largely controlled by the duty factor selected, and is independent of pulse frequency (over the range tested). It is more appropriate, therefore, to consider the concept of a critical duty factor for arc-free operation, rather than a critical frequency. This study indicates that for the deposition of alumina films a duty factor of 70% or lower is necessary for medium-term (i.e., several hours) arc-free operation. The deposition rate also appeared to be independent of pulse frequency, but to increase with reverse voltage at any given duty factor.

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