High Power Pulsed Reactive Sputtering of Zirconium Oxide and Tantalum Oxide

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ABSTRACT

Zirconium oxide and tantalum oxide films were deposited in an inverted cylindrical magnetron cathode using high power pulsed sputtering. Average power densities were between 5 and 7 W/cm^2 and peak power densities were in the range of 0.5 kW/cm^2. In all of the experiments the oxygen was introduced using flow control, and the current and voltage waveforms of the HPPMS supply changed in a reproducible manner as the targets went from the metallic to the poisoned mode. Coatings were done with the targets in the poisoned mode and for both materials high index films were deposited with very little evidence of arcing. The maximum deposition rate achieved for zirconium oxide was 1.9 nm/min, while tantalum oxide was deposited at 18 nm/min. Comparisons are made with coatings deposited using mid frequency AC sputtering in the same system. The specific deposition rate for zirconium oxide made using the HPPMS power supply was 25% of that when using the AC power supply. In contrast, the specific deposition rates for tantalum oxide were very similar for the two power supplies. X-ray diffraction revealed that the zirconium oxide grew in the monoclinic phase.

INTRODUCTION

High power pulsed magnetron sputtering (HPPMS), which uses peak power densities on the order of kW/cm^2 with duty cycles of 1-2%, has emerged as a promising new technique for producing high levels of ionization in sputtering plasmas [1-5]. With the development of HPPMS power supplies that are capable of arc handling [6,7], titanium oxide and aluminum oxide have recently been deposited using this method [8,9].

There were two purposes for the work reported here. The first was to study the use of HPPMS in an inverted cylindrical magnetron cathode. Inverted cylindrical magnetrons produce unique geometrical confinement of the sputtering plasma. This provides an interesting comparison with previous HPPMS work, which has been done on planar and rotating magnetrons. The second purpose was to study the reactive deposition of zirconium oxide and tantalum oxide using HPPMS with this geometry. Both are high index materials that are important in applications ranging from optical devices to thermal barriers on turbine blades.

EXPERIMENT

The inverted cylindrical magnetron used in these experiments is shown in Figure 1 and has been described elsewhere [10].

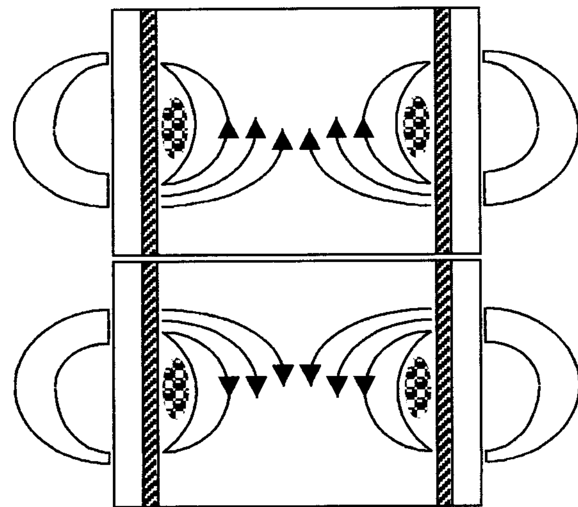

Figure 1: Geometry of the inverted cylindrical magnetron cathode used in these experiments.

Briefly, it consists of two independently driven coaxial cylindrical magnetrons, both of which can use either balanced or unbalanced magnets. Each magnetron is 19 cm in diameter by 10 cm high. The racetracks typically cover approximately one half of the target area, so the power densities we report are calculated assuming an eroded area of 300 cm^2 for each target.

When the HPPMS power supply was used, it was connected to only the top cathode. The peak powers were typically on the order of 0.5 kW/cm^2. The reported average powers were calculated using $1/2\ CV^2f$, where $1/2\ CV^2$ is the energy stored in the power supply capacitor bank on each pulse and f is the pulse frequency.

The coatings deposited using HPPMS were compared to those made by driving both cathodes simultaneously using 40 kHz AC power [11]. Optical emission spectroscopy is normally used to control the reactive gas flow in this system. However,

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fluctuations in the plasma intensity prevented the use of optical emission control in the case of HPPMS. Therefore, all of the coatings reported here (both AC and HPPMS) were deposited with the oxygen introduced in flow control and the targets fully poisoned. This should result in a worst-case situation with respect to arcing.

Commercially pure (99.5%) zirconium and tantalum targets were used. The argon flow was 75 sccm for all of the experiments. The system is pumped with a turbomolecular pump and the pressure is maintained through downstream control using a pendulum gate valve.

In all of the experiments the substrates were located in the center of the upper racetrack. The reported deposition rates were calculated from thicknesses measured with an Alpha Step 250 profilometer on silicon wafers. A Gaertner L116B ellipsometer was used to determine the optical constants for coatings on silicon at a wavelength of 632.8 nm.

RESULTS AND DISCUSSION

Zirconium Oxide

Using the HPPMS power supply, the transition from the metallic to the poisoned mode of the target was easy to determine from the output voltage and current waveforms. Figure 2 shows the voltage, current, and power as functions of time for a single pulse with the zirconium target sputtering in the metallic mode. Figure 3 shows the same parameters with the zirconium target sputtering in the poisoned mode.

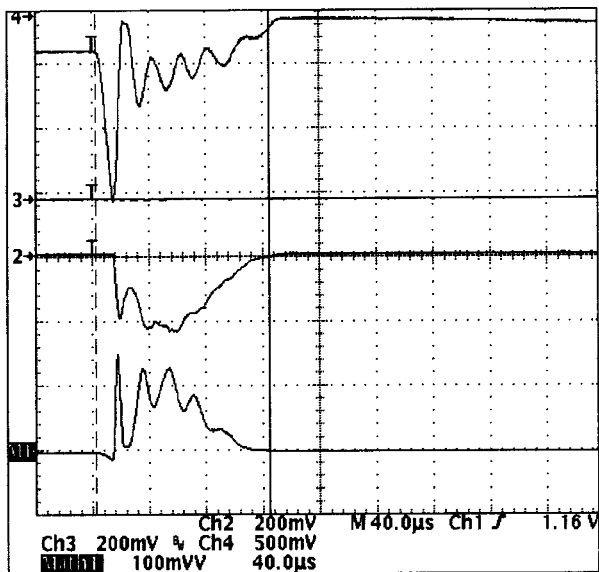

Figure 2: The voltage (channel 4, 500 V/div), current (channel 2, 200 A/div) and power (channel M1, 100 kW/div) as functions of time for a single pulse from the HPPMS power supply with the zirconium target in the metallic mode.

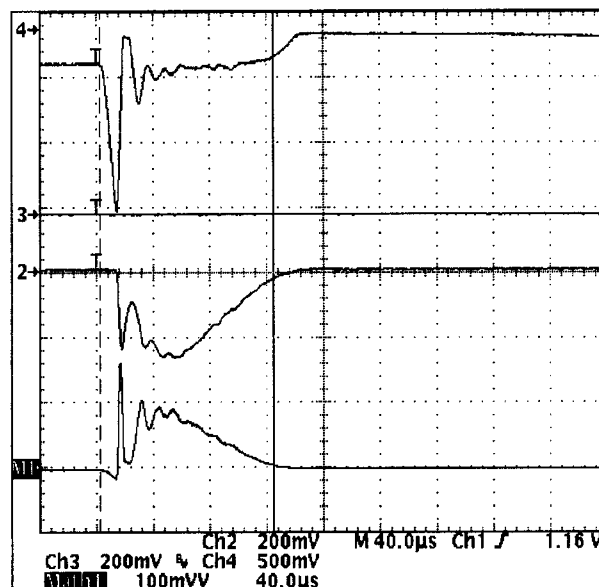

Figure 3: The voltage (channel 4, 500 V/div), current (channel 2, 200 A/div) and power (channel M1, 100 kW/div) as functions of time for a single pulse from the HPPMS power supply with the zirconium target in the poisoned mode.

These qualitative differences in the waveforms were very reproducible and proved to be a reliable indicator of the target condition. We also noticed that the magnitude of the initial large voltage spike (approximately 1400 V) did not change as the target poisoned, but the magnitude of the associated initial current spike did. We were able to use the value of this initial current to map out a hysteresis curve for the process and the results are shown in Figure 4.

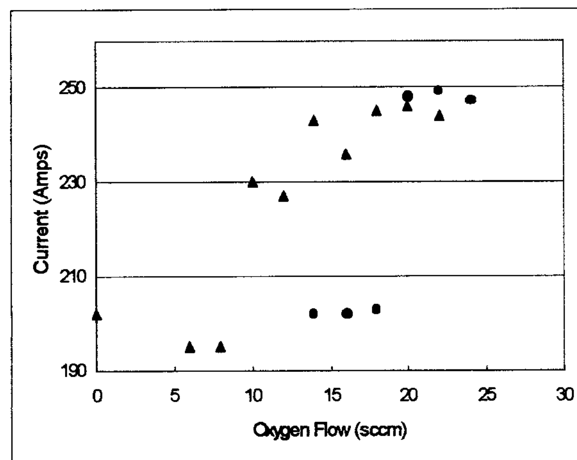

Figure 4: The magnitude of the initial current spike as a function of oxygen flow for increasing (circles) and decreasing (triangles) flows while sputtering zirconium oxide.

The data shown in Figure 4 were taken at an average power density of 8 W/cm^2, a peak power of 0.5 kW/cm^2, a pulse frequency of 400 Hz and a pressure of 5 mTorr. It is possible that these changes in current could provide a signal with which to control the reactive gas flow in an HPPMS process.

As the waveforms show, there was virtually no indication of arcing at any oxygen flow during the deposition of zirconium oxide. Furthermore, the racetrack was open with very few arc tracks after the deposition of high index films.

Table 1 summarizes the power density, deposition rate and optical constants for zirconium oxide coatings deposited using both HPPMS and AC sputtering. The total pressure during the HPPMS process was 3.1 mTorr and during the AC process it was 4.0 mTorr. In both cases the coating time was 100 minutes.

Table 1: Power density, deposition rate, and optical constants for zirconium oxide sputtered in the poisoned mode using both HPPMS and 40 kHz AC power.

Mode	Power Density (W/cm^2)	Rate (nm/min)	n	k
HPPMS	7.0	1.9	2.03	-.0002
40 kHz	4.3	4.7	2.26	-.0005

Even though the average power density for the HPPMS process was 60% greater than for the AC process, the deposition rate was only 40% that of the AC process. Normalizing the two processes to the same power density, the specific HPPMS rate is approximately 25% of the AC rate. This agrees very well with the relative rates for aluminum oxide and titanium oxide reported by others at this conference [8,9].

X-ray diffraction measurements determined that the zirconium oxide deposited using HPPMS grew in the monoclinic phase. This is typical of what is found in single layer sputtered zirconia films [12].

Tantalum Oxide

The waveforms for tantalum oxide sputtered in the metallic and poisoned modes by HPPMS also went through a transition very much like the one shown in Figures 2 and 3. There was also very little arcing or evidence of arc tracks on the target in the case of tantalum oxide sputtered over a range of conditions using HPPMS.

Table 2 summarizes the power density, deposition rate and optical constants for tantalum oxide coatings deposited using both HPPMS and AC sputtering. The total pressure during both processes was 5 mTorr. The HPPMS coating time was 20 minutes and the AC coating time was five minutes.

Table 2: Power density, deposition rate and optical constants for tantalum oxide sputtered in the poisoned mode using both HPPMS and 40 kHz AC power.

Mode	Power Density (W/cm^2)	Rate (nm/min)	n	k
HPPMS	4.8	17.9	2.11	-.002
40 kHz	4.8	16.8	2.17	-.0008

The striking result here is that with comparable power densities the deposition rates are also comparable for the two processes, unlike the case for other materials.

For the coatings reported in Table 2, the average power and oxygen flow for the HPPMS process were 1440 W and 50 sccm respectively and for the AC process they were 2875 W and 100 sccm. Measurements made on a number of films using both processes showed that the targets were fully poisoned under these conditions. Moreover, two cathodes were running at the indicated power density in proximity to the substrate in the AC case and only one in the HPPMS case. If anything, the additional material from the bottom cathode should have resulted in a thicker coating in the AC case than in the HPPMS case. An explanation for the similarity in rates for HPPMS and AC deposition of tantalum oxide is still open.

CONCLUSION

High power pulsed magnetron sputtering has been used to deposit zirconium oxide and tantalum oxide in an inverted cylindrical magnetron cathode. In both cases high index films were made with the targets in the poisoned mode with very little evidence of arcing. The specific deposition rate for zirconium oxide using the HPPMS power supply was approximately 25% of the rate when using a 40 kHz AC supply. In contrast, the specific deposition rate for tantalum oxide was very similar for the two supplies.

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