Substrate Heating and Deposition Rate Measurement in a HIPIMS Discharge

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ABSTRACT

The high degree of ionisation known to be present in the deposition flux generated via HIPIMS magnetron discharge provides significant potential to modify the properties of deposited films, whilst avoiding the excessive stresses that can be induced via substrate biasing in conventional DC or pulsed-DC deposition. This could be particularly advantageous when coating polymeric web substrates, where applying such a bias can be problematic. The thermally sensitive nature of these materials also precludes the use of post deposition annealing as a means of structural modification of the coating, and so the HIPIMS technique could potentially provide an appropriate means of improving the growth and subsequent properties of coatings on web. The thermal power density was measured at the substrate position at a variety of operating parameters in HIPIMS mode and compared with continuous DC and pulsed DC discharges at the same average power. Of these, the HIPIMS discharges delivered significantly less thermal power to the substrate (5 to 10 times less), and it was found that a HIPIMS discharge at 2500W average power was equivalent in substrate thermal power density to a 500W DC discharge. Deposition rates were measured at these conditions to enable the estimation of thermal flux per depositing atom.

INTRODUCTION

Magnetron sputtered films are commonly used to coat large-area and heat sensitive substrates such as polymer web, but are restrictive in terms of the structures and properties of these coatings, which cannot be annealed post-deposition due to the nature of the substrate. The ability to generate film structures that normally require high energy input, without the need for excessive deposition, or post-deposition treatment temperature would be advantageous – perhaps enabling such technologies as high quality transparent conductors on flexible polymer substrates. One means of enhancing the film structure without such heating is to increase the level of ionisation of the coating flux arriving at the substrate. DC magnetron sputtered coating flux tends to have ionisation rates of less than 1%; this can be improved upon significantly via delivering the power to the target cathode in the high power impulse magnetron sputtering (HIPIMS) mode. Ionisation rates of up to 70% have been reported for the sputtering of titanium in this mode [1,2], which would suggest the possibility of significant improvement of film properties over conventional sputtering techniques. This degree of ionisation is created by the intense pulse of power delivered via this technique, with the degree of ionisation being a function of the target material and the peak power delivered during the pulse. Such levels of ionisation are comparable to those achieved via cathodic arc deposition, but without the issue of formation of macro-particles that can lead to defects within the film. The HIPIMS technique has previously been employed to deposit a range of metallic [1-3], metal oxide[4-6] and metal nitride[7,8] coatings onto rigid substrates (including tool steel, silicon and glass).

In addition to the high levels of ionisation of the depositing flux, HIPIMS has typically low duty cycles and long pulse-off periods that lead to a substantially lower total heat load to the substrate when compared with dc and pulsed-dc magnetron sputtering. Preliminary measurements by Kelly and Bradley [9] indicated a reduction in thermal power density at the substrate of 5 to 10 times when using HIPIMS rather than continuous or pulsed-dc over the range 500W to 2500W and temperatures of 20 to 40 ^{O}C.

A further observation of the HIPIMS deposition flux is a reduced deposition rate when compared to continuous and pulsed-dc sputtering. As throughput is a significant factor in the commercial viability of coating processes, reduced deposition rate could be a disadvantage of this mode of sputtering. Also, as deposition rate and heat delivery to the substrate strongly interact in conventional magnetron sputtering, any reduction in substrate temperature in HIPIMS mode could only be an advantage if it is not offset by excessive loss of deposition rate.

The data presented here expand upon the initial thermal power flux measurements in HIPIMS mode and also include deposition rate measurements so as to allow direct comparison to continuous and pulsed-dc sputtering by calculating the level of thermal power delivered to the substrate for the same average process power and the same deposition rate.

EXPERIMENTAL

A Teer Coatings UDP 450 magnetron sputtering rig was fitted with a single unbalanced magnetron with a titanium target (99.5% purity, 300 x 100mm). The target was sputtered in argon at 0.2 Pa for all measurements, and power was delivered

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in continuous and pulsed-dc modes via an Advanced Energy Pinnacle plus supply, and in HIPIMS mode via a Huettinger HMP1/1_P2 supply. Target current and voltage waveforms, and hence power levels, were monitored directly via a digital oscilloscope. Deposition rates were measured at a distance of 150mm from the target surface via a Maxtek crystal monitor, and substrate temperature measured in a similar position over time via a thermal probe, described below. A shutter was placed such that the probes could be isolated from the plasma between readings.

The thermal probe consists of an electrically floating copper disc, 24mm in diameter and 2mm thick, mounted in a ceramic (Macor®) block, such that only the front face was exposed to the plasma, and conductive thermal losses were reduced by minimising the contact area between the copper probe and any other surface. K-type thermocouples attached directly to the back of the disc allowed the temperature to be monitored during deposition and later used to calculate the net thermal energy input, and hence power density of the deposition flux at this position. A description of its operation and the subsequent derivation of thermal power flux is given below.

Magnetron glow discharges were generated in continuous dc, and at 350 kHz asymmetric bipolar pulsed-dc (50% duty), at three time-averaged discharge power levels (500W, 750W, 1000W). HIPIMS glow discharges were generated at the same three time-averaged power levels (500W, 750W, 1000W) and at five pulse frequencies (100Hz, 250 Hz, 500 Hz, 750 Hz, 1000Hz) at each of these power levels. The pulse width was 100µs in each case. The deposition rate and heating and cooling rates were measured for each discharge, and used to determine the thermal energy flux to the substrate for a normalised deposition rate.

THERMAL ENERGY FLUX MEASUREMENTS

The thermal flux to the substrate position was measured by a method proposed by Thornton [11], based on the temporal evolution of a small mass of material located in the substrate position. The method has been extensively employed for the study of energy flux to a substrate in a magnetron discharge, and a technique for calibration of such a probe given by Wendt, et.al. [12]. This approach was later employed by Cada, et.al.[10], for the measurement of thermal energy flux in a pulsed-dc magnetron discharge; the probe used here being identical to that used by Cada and described in detail in that reference.

The technique is based on a model that accounts for the energy balance at the substrate (or in this case, the probe), including energy delivered from the argon plasma and the deposition flux to the probe, and energy lost via secondary electron emission, re-sputtering of deposited material, radiation, etc. By consideration of this energy balance, the thermal capacity and dimensions of the probe, and the thermal gradient from the front face (exposed to plasma and deposition flux) and the thermocouple junction at the rear, a simple equation is derived for the net thermal energy flux, Q (mWcm^{-2}), based on the rate of temperature change at a given temperature value, dT/dt (Ks^{-1}):

$$Q = (C_p/A).dT/dt \qquad (1)$$

Where, A is the exposed area of the probe (4.52 cm^2), and C_p (3.1 JK^{-1}), the thermal capacity of the probe. (Error determined to be ~5%.)

Therefore, by measuring the rate of temperature rise of the probe the energy flux for that deposition condition could be determined. The value of Q is calculated here in mWcm^{-2} rather than Wm^{-2}, in order to provide more relevant values for the process.

RESULTS

Figure 1 shows the target current and voltage waveforms for 350 kHz pulsed-dc at 1kW, as measured via oscilloscope, and the resultant power waveform. The waveforms for the 1kW HIPIMS discharge at 1kHz are given in Figure 2 – although the whole cycle is not shown here in order to show the pulse itself in full detail. Figure 3 shows the measured deposition rates for HIPIMS discharges. The rate increases both with average discharge power, as may be expected, but also with frequency. It should be noted that the pulse width was 100µs in each case, so the duty increases with frequency. The deposition rates for continuous and pulsed-dc are given in Table 1. It can be seen that the deposition rate in continuous dc mode is significantly higher than for pulsed-dc (at 350 kHz and 50% duty), although rates for pulsed-dc and HIPIMS (Figure 3) are of the same order.

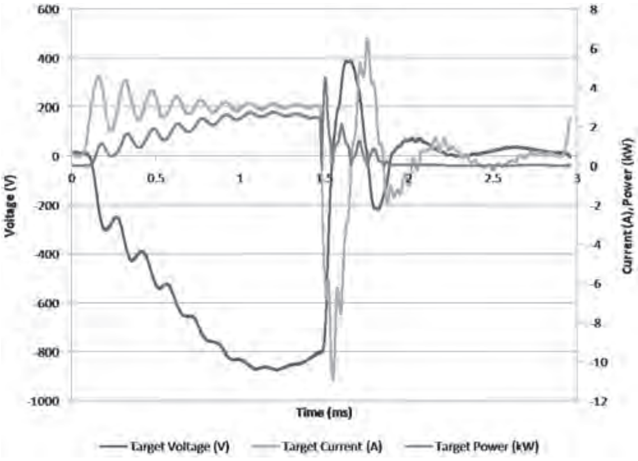

Figure 1: Measured target current and voltage waveforms, and calculated power for 350 kHz pulsed dc discharge at 1000W.

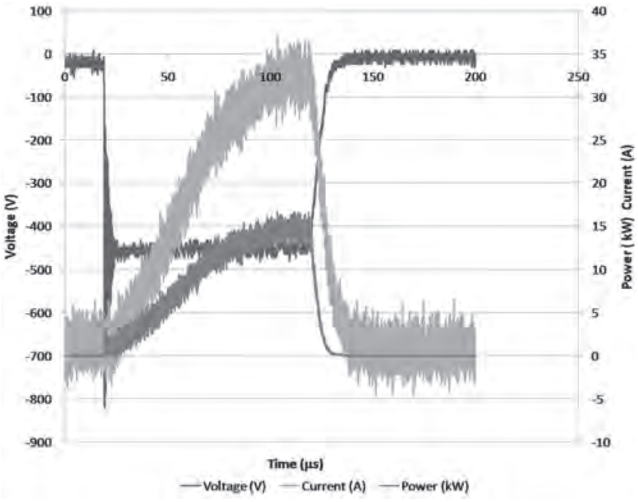

Figure 2: Measured target current and voltage waveforms, and calculated power for 1kHz HIPIMS discharge at 1000W.

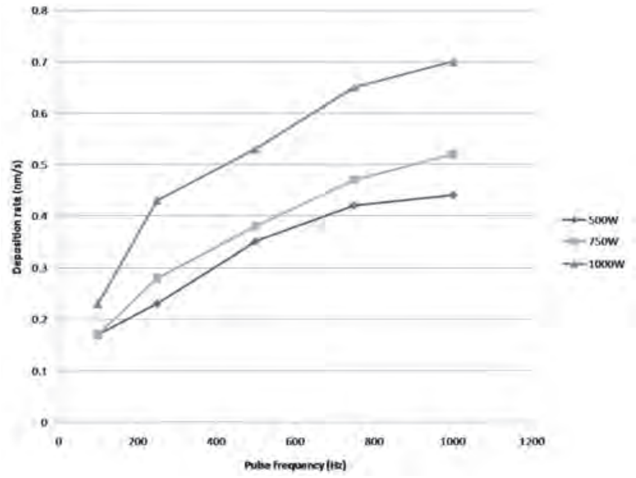

Figure 3: Deposition rated for HIPIMS discharges.

Table 1: Deposition rates for continuous and pulsed-dc discharges.

	Deposition Rate (nm min^{-1})	
Power (W)	Continuous DC	Pulsed at 350 kHz
500	0.53	0.19
750	1.08	0.31
1000	1.47	0.41

The thermal energy flux delivered to the substrate (probe) is given for each case in Table 2. Continuous and pulsed dc discharges are shown to generate significantly increased thermal load on the substrate, as compared with all HIPIMS discharges at all time-averaged powers.

Table 2: Thermal energy flux values for all discharges.

	Thermal Energy Flux (mWcm^{-2})						
Discharge Power (W)	DC frequency (kHz)		HIPIMS frequency (Hz)				
	0	350	100	250	500	750	1000
500	15.64	8.62	0.32	0.47	0.21	0.30	0.31
750	26.66	16.59	0.22	0.19	0.76	0.19	0.17
1000	49.43	27.87	1.39	4.02	4.37	3.87	1.81

The values for the thermal energy flux were divided by the deposition rate in each case to arrive at a thermal energy flux value per nm per minute of deposition. This provides a more meaningful measure of heat load that may be applied to a given process. These values are given in Figure 4, which shows two distinct groups of values, those for HIPIMS discharges having very low values compared with continuous and pulsed dc. It should be noted that the use of broken lines connecting the latter values are for illustration only, and are there to distinguish the groups more clearly, although the relationships between pulse frequency, deposition rate and heat load to the substrate in mid-frequency pulsed-dc magnetron sputtering are well known [10,13], and would approximate this general trend.

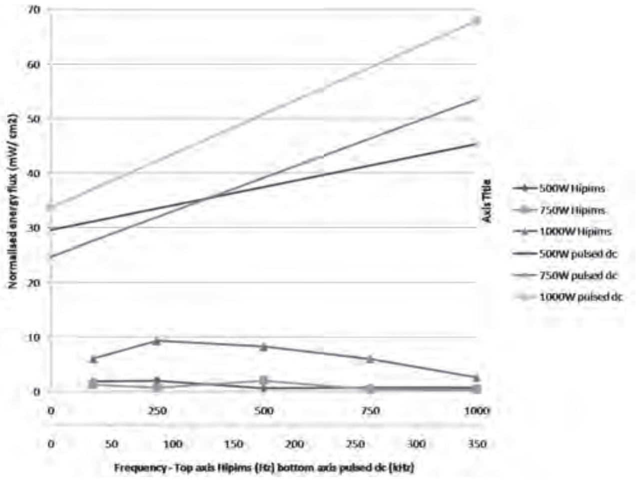

Figure 4: Normalised thermal energy flux for equivalent deposition rates with varying power and frequency in HIPIMS and pulsed-dc modes.

DISCUSSION

The data presented here confirm the significant difference in both substrate heating and deposition rate between dc (continuous and pulsed) and HIPIMS deposition systems. What is most striking is the contrast in the rate of thermal energy flux when the difference in deposition rate is accounted for. In normalising the data to compare values for 1nm min^{-1} deposition rate (measured as a function of deposited mass via crystal monitor), we are effectively deriving a figure for the thermal energy input per deposited atom. Figure 4 illustrates convincingly that using the HIPIMS method of power delivery results in significantly lower heat load on the substrate *for the same thickness of film* than conventional dc and pulsed-dc magnetron sputtering. This may allow deposition of films onto substrates for which the temperature of deposition had previously proved to be prohibitive to the exploitation of the process.

The suggested benefit of increased ionisation of the deposition flux to the structure and properties of the deposited film have not however been addressed here. It is yet to be seen whether it is possible to grow higher quality films at lower deposition temperatures, or to eliminate the need for post-deposition annealing in order to achieve the desired film properties. A comparison of films grown under various HIPIMS parameters, along with a measure of the thermal energy flux at that substrate would be required to establish its usefulness in this respect. Additional characterisation of the degree of ionisation of the deposition flux, and the contribution of this factor on film growth would be necessary to understand, and make full use of this process.

The effect of pulse frequency in mid-frequency pulsed-dc magnetron sputtering has been shown to be significant both to the substrate thermal energy flux [10] and the deposition rate [13]. However, the pulse frequency in HIPIMS mode has shown less significance here on either process parameter. The pulse width was set to 100s in each case in order to reduce the significant number of variables exhibited by the HIPIMS power supply. This has lead to varying duty values at each frequency, with the duty increasing proportionally with frequency. Characterisation of a wider process envelope, although beyond this present work, may identify trends in deposition rate and thermal energy flux that aid in the optimisation of the HIPIMS process.

Due to the physical constraints of the coating system and probes used for this work, the separation between sputter target and the point of measurement was 150mm. This is outside the more usual range of 60-120mm separation of substrate from target cathode used in commercial systems. As both the deposition rate and thermal flux to the substrate are likely to be affected by the degree of separation of the target and substrate (this is well known in continuous and pulsed-dc processes), more realistic values need to be obtained within this range of separation. Such measurements are currently taking place in a dedicated rig that also allows full 2-dimensional (perpendicular and parallel to target) mapping of the discharge. A purpose-built thermal probe that is more sensitive to the lower thermal energy flux of the HIPIMS discharge will also be employed to investigate further the thermal effects of varying the full range of HIPIMS pulse parameters.

CONCLUSION

Significant reductions in the thermal energy flux delivered to the substrate can be achieved for the deposition of titanium films by using the HIPIMS mode of target power delivery, as compared with continuous dc or mid-frequency pulsed-dc techniques. When also taking into account the relative deposition rates of these technologies, the thermal energy flux generated per unit deposition rate is significantly lower (more than10 times lower in some cases) for HIPIMS discharges. This could be of significant benefit in the deposition of thin films onto temperature sensitive substrates.

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