Pulse length selection for optimizing the accelerated ion flux fraction of a bipolar HiPIMS discharge

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Abstract
The effect on the energy distributions of metal and gas ions in a bipolar high-power impulse magnetron sputtering (HiPIMS) discharge as the negative and positive pulse lengths are altered are reported. The results presented demonstrate that the selection of the pulse lengths in a HiPIMS discharge is important in optimizing the amount of accelerated ions. A short enough negative pulse is needed so that ions do not escape to the substrate before being accelerated by the positive pulse that follows the main negative HiPIMS pulse. The length of the positive pulse should also be long enough to accelerate the majority of the ions, but a too long positive pulse depletes the process chamber of electrons so much that it makes it difficult to initiate the next HiPIMS pulse. When pulse lengths of negative and positive pulses are properly selected, the fraction of ions, both metal and gas, accelerated by the positive pulse voltage is close to 100%.

Keywords: HiPIMS, bipolar HiPIMS, mass spectroscopy, pulse length

(Some figures may appear in colour only in the online journal)

1. Introduction
Bipolar high-power impulse magnetron sputtering (HiPIMS) refers to the application of positive voltage pulses following the conventional negative HiPIMS pulses and has recently attracted significant attention due to its capability of accelerating plasma ions toward the surface of the growing film [1–5], potentially overcoming the deposition rate problem in HiPIMS [6] and as a possible solution for ion bombardment during sputter deposition of insulating materials [7]. It is only recently that a HiPIMS discharge operated in bipolar mode has been realized [1, 2, 6, 8] but the idea of introducing a positive voltage in a HiPIMS discharge can be traced back to the suggestion by Konstantinidis et al [9] for increasing the deposition rate in HiPIMS discharges. The mechanism behind the observed ion acceleration during the positive pulse in bipolar HiPIMS is the generation of an electric field by increasing the plasma potential ($V_p$) in at least part of, the plasma region in-between the cathode and substrate as suggested by Wu et al [6] and measured by Velicu et al [1]. Keraudy et al [2] proposed that the plasma potential is uniformly increased with the applied positive potential within the magnetic trap adjacent to the target (the region where magnetic field lines intersect only the target surface) and that a drop in $V_p$ occurs in a transition region between the magnetic trap and the grounded region (the region where magnetic field lines intersect only grounded surfaces, i.e. either the...
chamber walls, a grounded substrate or the anode at the magnetron (which is held at ground potential). Such a $V_p$ distribution is also what was measured by Velicu et al [1], at least during the first few μs of the application of the positive pulse. On the other hand measurements by Hippler et al [10] indicate a significant potential fall only at the grounded substrate. In this case, the expected ion acceleration toward an insulating (electrically floating) substrate would only correspond to $V_f$ and the floating potential ($V_f$). Independent of whether the ions are accelerated in a potential fall in either a transition region or at a grounded substrate, the temporal and spatial distribution of the ions is important and dependent on the pulsing procedure applied.

For ions to be accelerated they need to reach the area of the potential fall while the accelerating positive pulse is applied. It follows that the factors influencing the fraction of accelerated ions include the time of ion creation (i.e. length and shape of the ion-generating HiPIMS pulse), the length and timing of the positive pulse, all in relation to the location of the accelerating field in the plasma volume. In the literature, different trends in the accelerated ion fraction have been reported which can be attributed to different experimental configurations employed: different magnetic field configurations, different lengths of both the negative HiPIMS pulse and positive pulse, etc. As a result of these differences, different trends in the accelerated ion fraction have been observed. In some studies, where a relatively long HiPIMS pulse is used (100 μs), only a very small portion of the detected ions are accelerated to energies close to the positive pulse potential [10–12], while in another study, also using long HiPIMS pulses, the majority of ions are accelerated [13]. On the other hand, studies in which shorter negative pulses are applied report consistently higher fractions of accelerated ions in the total ion flux [2, 5, 14], but also in this case there is an exception [1].

The studies cited above employed a fixed length of the HiPIMS pulse in combination with a positive pulse of different parameters (length, amplitude and delay between the negative and positive pulses). Here, we show that the length of the negative HiPIMS pulse is important in tuning the amount of accelerated ions in a bipolar HiPIMS discharge. By systematically investigating the effect of the length of both the negative and the positive pulses we show that the fraction of ions accelerated can be maximized. From the results generated, we can also draw a conclusion on where in the plasma volume the majority of ions are accelerated in our particular case.

2. Experimental details

All measurements are done in a cylindrical high vacuum chamber 30 cm in height and 40 cm in diameter pumped to a base pressure of ~10⁻⁷ Torr. A strongly unbalanced type II magnetron (TORUS circular, Kurt J. Lesker company) is fitted with a 5 cm diameter Ti target (99.9% purity) with a thickness of 0.6 cm. More details about magnetron magnetic field configurations and the effects on the electron and ion fluxes can be found in reference [15]. The radial magnetic field, at the target racetrack, is 75 mT and the magnetic null point occurs 10 mm above the center of target surface. Sputtering is carried out by introducing Ar (99.997%) at a flow rate of 30 sccm and the pressure is regulated at 0.66 Pa (5 mTorr) using a throttle valve. A bipolar pulsing unit (Ionautics AB) is used for the delivery of a negative pulse ($U_{rev}$) immediately followed by a positive pulse ($U_{rev} = 70$ V).

In the experiments the effects from the length of the negative pulse ($\tau_{neg}$) and the length of the positive pulse ($\tau_{pos}$) are studied while the peak current density ($J_{pk}$) (averaged over the whole target surface) and the total average power ($P_{avg}$) are maintained at ~0.5 A cm⁻² and ~60 W, by adjusting $U_d$ and the pulse repetition frequency ($f_p$). Consequently, the average discharge current was not held constant, however, varied typically only within 5% around a value of 100 mA.

Ion energy distributions functions (IEDFs) are measured by a quadrupole mass spectrometer with an energy filter (PSM 003, Hiden analytical Ltd.) using an orifice diameter of ~50 μm. The orifice is aligned approximately to the center of the target surface at a distance of ~8.5 cm. The electrode potentials of the mass spectrometer optics are tuned using $^{36}$Ar⁺. The same tuning values are used for all experiments to facilitate comparison between the investigated parameters. The ion flux intensity is measured in counts per second (cps) and no attempts have been made to adjust the intensity measurements for spectrometer performance at different ion energies. Less abundant isotopes ($^{36}$Ar⁺ and $^{46}$Ti⁺) are used in order to avoid detector saturation during time-averaged ion flux measurements. The ion energy distributions are measured from 0 to 100 eV with energy step of 0.1 eV using an acquisition time per data point of 200 ms. The total flux intensity is obtained by integrating the intensity with respect to the ion energy.

3. Results and discussion

3.1. Effect of the length of the negative pulse

Selected target voltage and current waveforms for $\tau_{neg} = 10, 50, 100, 150$ and 200 μs followed by $\tau_{pos} = 200$ μs at $U_{rev} = 70$ V, are shown in figure 1. The peak current ($I_{pk}$) is maintained at ~10 A (corresponding to $J_{pk} ~ 0.5$ A cm⁻²) and, by altering $f_p$ within 110–2000 Hz, $P_{avg}$ is maintained at 60 W. There is a delay in the onset of the target current which increases with $\tau_{neg}$. That means, the delay increases with decreasing $f_p$. For the shortest HiPIMS pulse, $\tau_{neg} = 10$ μs, a higher $U_d$ of ~700 V is needed to reduce this delay and to increase the current rise to become high enough to reach $I_{pk} = 20$ A during the short pulse.

The behavior of the onset of the current in a HiPIMS discharge has previously been described by Yushkov and Anders [16] to depend on $U_d$ and on the electron density remaining from the preceding pulse. With higher $U_d$, there is a faster development of the current, characterized by a shorter time lag in the onset and a more rapid increase. The electron density decays during the time between the negative discharge pulses with a typical characteristic decay time in the order of a few ms after the termination of the negative HiPIMS pulse [17]. Unsurprisingly, a low remaining plasma density results in a
The application of a slightly higher discharge voltage.

The shortest HiPIMS-pulse (10 μs) is more abrupt due to the application of a slightly higher discharge voltage.

Figure 1. Recorded voltage and current waveforms are shown for different length of the negative HiPIMS pulse. The current rise for the shortest HiPIMS-pulse (10 μs) is more abrupt due to the slow onset of the subsequent pulse [16]. As will be shown below (section 3.2), the positive pulse contributes in increasing the time lag in the onset of the target current.

Time-averaged IEDF measurements, corresponding to the pulsing conditions shown in figure 1, are presented in figure 2 (τ_{neg} = 10–200 μs and τ_{pos} = 200 μs). The overall shape of the distributions of the gas and metal ions are distinct from each other. Ar ions exhibit sharp and narrow peaks, while Ti ions show sharp peaks with a broad tail to higher energies. There are clearly two energy populations of both gas and metal ions. The higher energy ions have their peak positioned slightly above 70 eV, matching the applied U_{rev}, and the low-energy ions have a peak at 2–4 eV. The intensity ratio between low-energy ions and high-energy ions vary with τ_{neg}. This is especially pronounced for the metal ion population. For the shortest pulse (τ_{neg} = 10 μs) only a small amount of low-energy Ar ions are detected (<10² cps) and no low-energy Ti ions are reliably detected. Increasing τ_{neg} increases the population of low-energy ions and at the same time decreases the population of accelerated ions of both the gas and the metal.

We note that the characteristic shapes of the IEDFs observed for the gas and metal ions are a result of the origin of these ions, as described by Čada et al [18]. Ar ions originate from the ionization of gas atoms with a thermal energy distribution of less than 1 eV while the Ti ions originate from Ti atoms sputtered from the target and thus have an initial energy distribution related to the sputtering process [19] with a high-energy tail. The energies observed for the low-energy ions, 2–4 eV instead of <1 eV typical for thermalized ions, is due to the potential difference between the plasma and the grounded spectrometer [19, 20]. Likewise, the population of accelerated ions near 70 eV is a consequence of the difference between the increased plasma potential during the positive pulse and the ground potential.

The observed decreasing trend in ion acceleration with longer HiPIMS-pulses is likely due to the fact that longer discharge pulses (i.e. increasing τ_{neg}) will allow a larger fraction of ions to pass the region of ion acceleration before the ion-accelerating positive pulse is applied. Assuming that ion acceleration during the positive pulse (discussed in section 3.2) occurs only within the sheath of the grounded orifice (8.5 cm from the target), and neglecting collisions, the TOF for Ti ions with kinetic energies in the range 1–20 eV is in the range of 10–40 μs. It then follows that Ti ions which are generated earlier than 40 μs before the start of the positive pulse and leaving the ionization region close to the target with kinetic energies larger than 1 eV will not be accelerated, as they will reach the mass-spectrometer orifice before the start of the positive pulse. Similarly, all Ti ions with energies lower than 20 eV generated later than 10 μs before the start of the positive pulse will be accelerated. The discussed energy range covers well the expected peak of ∼2.5 eV for the Thompson distribution of sputtered Ti (approximated using ε_{ab}/2, where ε_{ab} is the surface binding energy) [21] as well as the observed peak of the energetic tail of Ti ions at ∼14 eV in the IEDF of a conventional HiPIMS discharge (τ_{pos} = 0 and τ_{neg} = 20 μs) as in figure 5. Calculation of the TOFs for the Ti ions with energies of 2.5 eV and 14 eV yields ∼26 μs and ∼11 μs, respectively. Ar ions, on the other hand, yields a TOF of 37–117 μs for an energy range of 0.1–1 eV. Using such a low ion energy range for the sake of calculating the TOFs of Ar ions is motivated by the fact that Ar ions in HiPIMS discharges are pre-dominantly low-energetic [22–25]. We conclude that decreasing τ_{neg} leads to larger fraction of ions being generated closer to the start of the positive pulse and therefore a larger fraction of the generated ions will be accelerated by the positive pulse potential.

To visualize the fraction of ions accelerated, the complete set of recorded IEDFs are integrated over a low-energy range, 0–60 eV, and over a high-energy range, 60.1–100 eV. The intensity of the high-energy ions is compared with the total ion intensity (i.e. integration of the ion intensity over the full energy range 0–100 eV) and are presented in figure 3. For long negative pulse lengths, the low-energy ions dominate, especially for Ti where only about 12% of the intensity comes from accelerated ions, while for the shortest pulse close to 100% of the intensity is from accelerated ions. The Ar ions are affected in a similar way, with 26% of the intensity from accelerated ions for long HiPIMS pulses and 90%–94% for short pulses. As the pulses are shortened, there is a saturation in the integrated intensity from accelerated ions at about 20 μs for Ti ions and at about 30 μs for Ar ions. For Ar ions there appears to be a reduction in accelerated ions for the shortest pulse (τ_{neg} = 10 μs), however, the low signal-to-noise ratio in the low energy region makes this observation uncertain.
Figure 2. Ion energy distributions for Ar⁺ and Ti⁺ using $\tau_{\text{neg}} = 10\text{--}200\mu s$ and $\tau_{\text{pos}} = 200\mu s$. 
The slightly higher fraction of accelerated Ar ions compared to Ti ions for longer HiPIMS pulses (figure 3) is likely due to variations in the ion composition during the HiPIMS pulse, as shown in previous studies [26, 27]. The variations in the ion composition could be due to several factors, such as the different phases of a HiPIMS discharge [26, 28] as well as gas rarefaction [29, 30]. For longer HiPIMS pulses, the plasma drifts out to larger axial distances [31]. This leads to preferential ionization of additional working gas atoms, since they are more homogenously distributed in the volume compared to the neutral sputtered species [18].

3.2. Effect of the length of the positive pulse

Similar measurements were done keeping $\tau_{neg}$ constant at 20 $\mu$s but altering $\tau_{pos}$ from 0 to 400 $\mu$s and keeping $U_{rev}$ constant at 70 V. Corresponding voltage and current waveforms are shown in figure 4. A delay in the onset of the target current is generally observed when introducing a positive pulse after the HiPIMS pulse due to a more effective depletion of electrons to the target in-between the HiPIMS pulses [3, 5, 32]. Due to this delay, and a limited $\tau_{neg}$, the power per pulse decreases. It was therefore necessary to increase the pulse repetition frequency from $f_p = 1070$ to 1330 Hz and 1430 Hz for the two longest positive pulses ($\tau_{pos} = 200$ and 400 $\mu$s) in order to keep $P_{avg} = 60$ W. Small adjustments of $U_d$ were also needed for the two longest positive pulses to maintain $I_{pk} \sim 10$ A.

Time-averaged IEDFs for the different $\tau_{pos}$ are shown in figure 5. As in figure 2, the gas and metal ions are distinct from each other in that the Ar$^+$ IEDFs are composed of sharp peaks while the Ti$^+$ IEDFs are composed of sharp peaks and a broad energetic tail. In all cases, except for $\tau_{pos} = 0$ $\mu$s, the IEDFs exhibit two peaks corresponding to low-energy ions centered at energies $<2$ eV and a population of accelerated ions centered slightly above 70 eV. Increasing the length of the positive pulse decreases the intensity of the low-energy ions of both gas and metal.

The accelerated ion fractions of the integrated intensities are shown in figure 6. For both Ti$^+$ and Ar$^+$ the fraction of accelerated ions increases with increasing $\tau_{pos}$. However, already for the shortest pulse investigated, $\tau_{pos} = 10$ $\mu$s, a significant portion of ions is affected by the applied positive potential as evaluated to $\sim$91% for Ti$^+$ and $\sim$75% for Ar$^+$. Increasing $\tau_{pos}$ increases the intensity of accelerated ions up to $\tau_{pos} = 200$ $\mu$s, after which only a negligible increase is observed. Possibly, there is a reduction in Ar$^+$ ion intensity for $\tau_{pos} = 400$ $\mu$s, which, similar to the case of $\tau_{neg} = 10$ $\mu$s, is uncertain due to the low signal-to-noise ratio in the low energy region.

It has been shown before that the fraction of ions being accelerated increases as the length of the positive pulse increases, see Kozák et al [11] and Tiron and Velicu [14]. As discussed in the introduction, there are different ideas [2, 6, 10, 14] presented on where the ion acceleration occurs. Our preliminary measurements of the floating potential at the substrate position (not presented here) indicate that, for the experimental conditions presented in this paper, the potential of a floating substrate follows very closely the positive target potential, as similarly observed by Hippler et al [10], which is a strong indication that the acceleration during the positive pulse occurs in the sheath at the grounded mass-spectrometer orifice. This observation is in line with the expected TOF of the ions discussed earlier. The TOF of the Ti ions with energy of 14 eV is 11 $\mu$s, which, even for the short positive pulse of $\tau_{pos} = 10$ $\mu$s, allows the majority of fast Ti ions to reach the acceleration region at the orifice before the positive pulse is turned off. On the other hand, the TOF for $\sim$2.5 eV Ti ions is 26 $\mu$s, which
Figure 5. Measured IEDFs for different $\tau_{pos}$ applied after a 20 $\mu$s main HiPIMS pulse.
explains why the low-energy peak of Ti ions is not accelerated for the short positive pulse $\tau_{\text{pos}} = 10 \mu s$ while for $\tau_{\text{pos}} = 50 \mu s$ the majority of them are.

4. Conclusions

The effects of negative and positive pulse lengths on the high-energy fraction of Ti$^+$ and Ar$^+$ ions in a bipolar HiPIMS discharge has been systematically investigated by comparing integrated ion flux intensities between high- and low-energy ranges from measured IEDFs. With the given distance between the sputtering target and the mass spectrometer orifice ($\sim 8.5$ cm) it can be observed that if the negative HiPIMS pulse exceeds 20 $\mu s$ in length, the fraction of accelerated metal ions starts to decrease rapidly from $\sim 100\%$ to a saturation level of $\sim 10\%$ for $\tau_{\text{neg}} = 200 \mu s$. The reason for this decrease in the amount of accelerated metal ions is that $\tau_{\text{neg}}$ exceeds the TOF of Ti$^+$ ions and therefore a larger fraction of metal ions arrive at the orifice before the positive pulse is applied. For the longest $\tau_{\text{neg}}$ investigated, the percentage of accelerated ions is $\sim 11\%$ for Ti$^+$ and $\sim 26\%$ for Ar$^+$. Using a relatively short HiPIMS-pulse ($\tau_{\text{neg}} = 20 \mu s$), the fraction of high-energy ions in the flux is as high as $\sim 91\%$ for Ti$^+$ and $75\%$ for Ar$^+$, even when using a positive accelerating pulse as short as 10 $\mu s$. The fraction of accelerated ions increases and settles at a level of $\sim 100\%$ for Ti$^+$ and $\sim 98\%$ for Ar$^+$ as the positive pulse is increased to 200 $\mu s$. Summarizing, a too short positive pulse will allow ions to arrive at the mass spectrometer without being accelerated and a too long pulse will not bring additional benefits but only contribute in depleting the process chamber of electrons, increasing the time needed to initiate the next HiPIMS pulse.

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Figure 6. The fraction of counts from high energy ions (>60 eV) as a function of the length of the positive pulse ($U_{\text{rev}} = 70$ V).
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