

Reactive High Power Impulse Magnetron Sputtering (HiPIMS)

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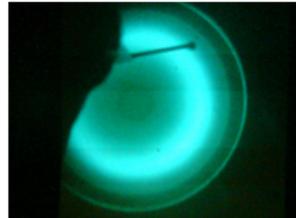
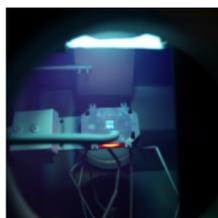
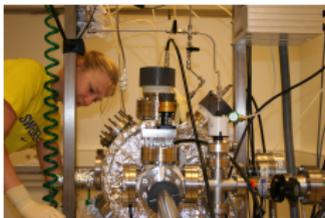
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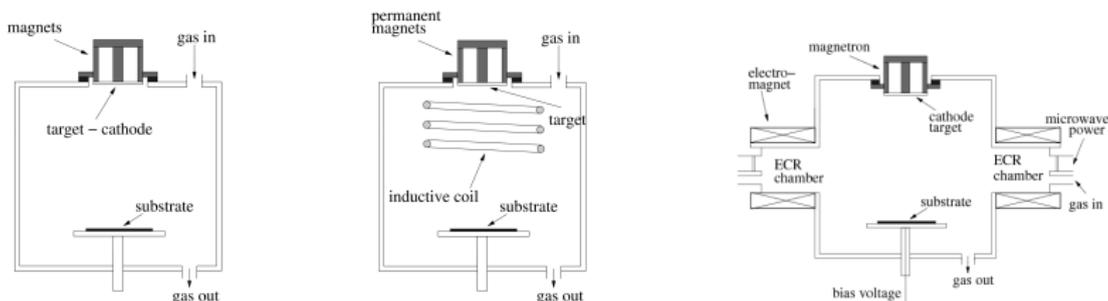


Introduction



- Magnetron sputtering has been the workhorse of plasma based sputtering methods for over three decades
- For many applications a high degree of ionization of the sputtered vapor is desired
 - controlled ion bombardment of the growing film – controlled by a negative bias applied to the substrate
 - collimation – enhanced step coverage
- Ionized flux of sputtered vapor introduces an additional control parameter into the deposition process

Introduction



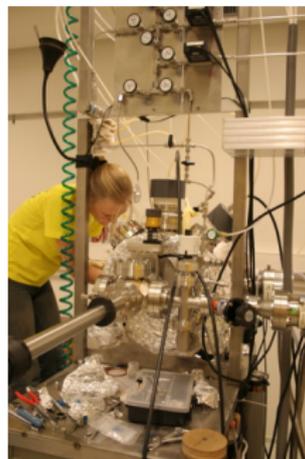
From Gudmundsson (2008b), J. Phys.: Conf. Ser. **100** 082002

- In magnetron sputtering discharges increased ionized flux fraction is achieved by
 - a secondary discharge between the target and the substrate (rf coil or microwaves)
 - reshaping the geometry of the cathode to get more focused plasma (hollow cathode)
 - increasing the power to the cathode (high power pulse)
- Common to all highly ionized magnetron sputtering techniques is a very high density plasma

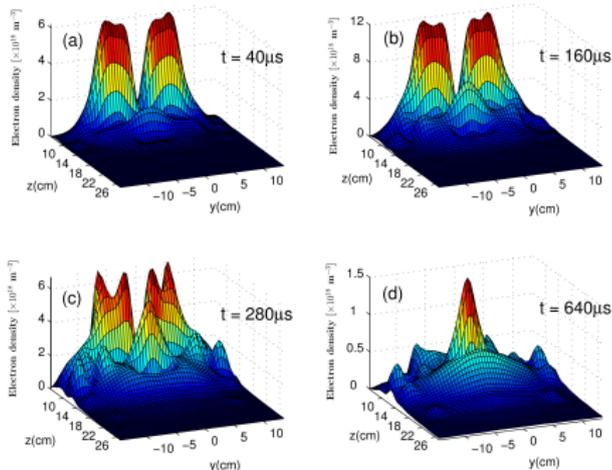


Introduction

- In a conventional dc magnetron discharge the power density is limited by the thermal load on the target
- In a HiPIMS discharge a high power pulse is supplied for a short period
 - low frequency
 - low duty cycle
 - low average power
- The high power pulsed magnetron sputtering discharge uses the same sputtering apparatus except the power supply



Introduction

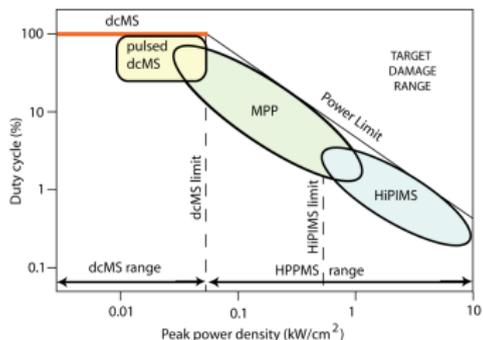


(After Bohlmark et al. (2005), IEEE Trans. Plasma Sci. **33** 346)

- Temporal and spatial variation of the electron density
- Ar discharge at 20 mTorr, Ti target, pulse length 100 μs
- The electron density in the substrate vicinity is of the order of $10^{18} - 10^{19} \text{ m}^{-3}$

Introduction

- High power pulsed magnetron sputtering (HPPMS)
- HiPIMS
 - a pulse of very high amplitude, an impulse, is applied to the cathode and a long pause exists between the pulses
- Modulated pulse power (MPP)
 - the initial stages of the pulse (few hundred μs) the power level is moderate (typical for a dcMS) followed by a high power pulse (few hundred μs up to a ms)



From Gudmundsson et al. (2012), JVSTA **30** 030801

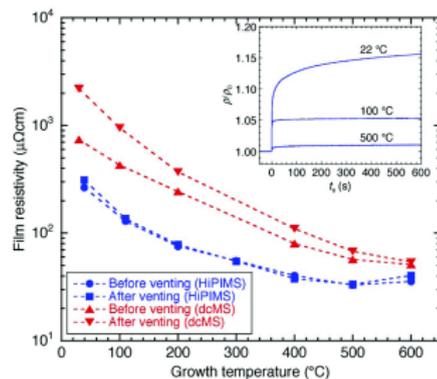
- Power density limits
 - $\rho_t = 0.05 \text{ kW/cm}^2$ dcMS limit
 - $\rho_t = 0.5 \text{ kW/cm}^2$ HiPIMS limit

Reactive HiPIMS - Applications



Application – Film Resistivity

- TiN as diffusion barriers for interconnects
- HiPIMS deposited films have significantly lower resistivity than dcMS deposited films on SiO₂ at all growth temperatures due to reduced grain boundary scattering
- Thus, ultrathin continuous TiN films with superior electrical characteristics and high resistance towards oxidation can be obtained with HiPIMS at reduced temperatures



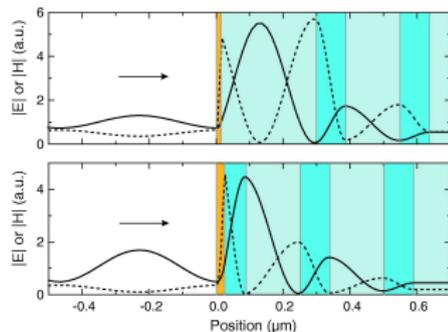
From Magnus et al. (2012) IEEE EDL **33** 1045



Application – Bragg mirror

- Multilayer structures containing a high-contrast ($\text{TiO}_2/\text{SiO}_2$) Bragg mirror fabricated on fused-silica substrates
 - reactive HiPIMS TiO_2 (88 nm)
 - reactive dcMS SiO_2 (163 nm)
 - capped with semitransparent gold
- Rutile TiO_2 ($n = 2.59$) and SiO_2 ($n = 1.45$) provide a large index contrast
- Smooth rutile TiO_2 films can be obtained by HiPIMS at relatively low growth temperatures, without post-annealing

Agnarsson et al. (2013) TSF 545 445



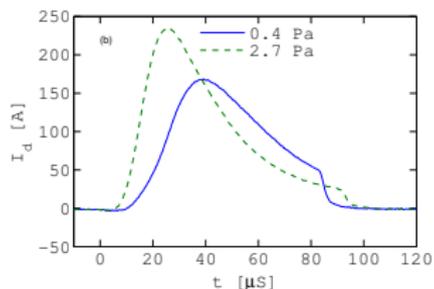
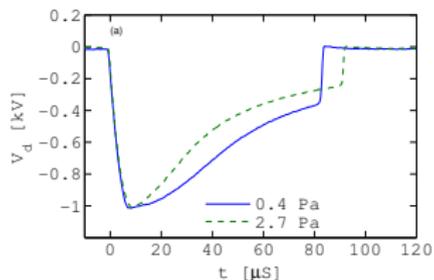
From Leosson et al. (2012) Opt. Lett. 37 4026

Reactive HiPIMS - Voltage - Current - Time characteristics



HiPIMS - Voltage - Current - time

- To describe the discharge current-voltage characteristics the current-voltage-time space is required
- The early work on HiPIMS used 50 – 100 μs pulses and a pulse repetition frequency in the range 50–1000 Hz
- The cathode voltage and the discharge current depend on the discharge gas pressure

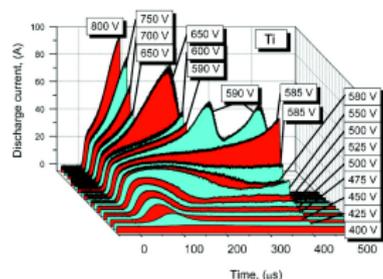
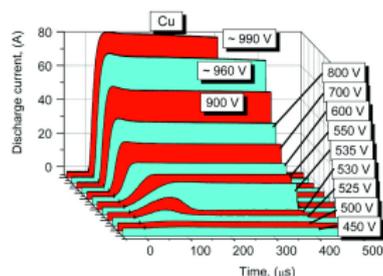


From Gudmundsson et al. (2012), JVSTA 30 03080



HiPIMS - Voltage - Current - time

- For longer pulses the initial pressure dependent current peak is followed by a second phase that is power and material dependent
- The initial phase is dominated by gas ions, whereas the later phase has a strong contribution from self-sputtering
- For some materials, the discharge switches into a mode of **sustained self-sputtering**



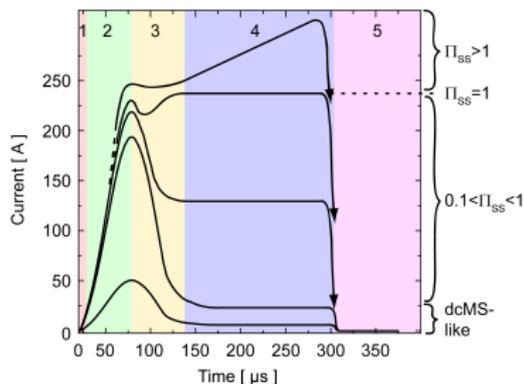
From Anders et al. (2007),

JAP 102 113303 and JAP 103 039901



HiPIMS - Voltage - Current - time

- A schematic illustration of the discharge current assuming square shaped voltage pulses
- The current is generally characterized by an initial peak followed by a more or less stable current plateau (bottom current curves)
- In other cases it shows an initial peak followed by a second increase of the discharge current (top current curves)



From Gudmundsson et al. (2012), JVSTA **30** 030801

HiPIMS - Voltage - Current - time

- The self-sputtering can operate in a self-sustained mode, when the ions of the sputtered vapor are created at high enough rate that the ions of the working gas are not needed
- The condition for sustained self-sputtering is expressed as

$$\Pi_{ss} = \alpha\beta_t Y_{ss} = 1$$

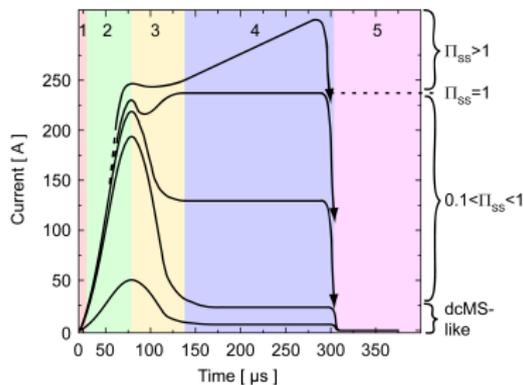
where

- α is the probability of ionization of the sputtered atom
- β_t is the probability that the newly formed ion of the sputtered vapor returns to the target
- Y_{ss} is the self-sputter yield of the ion
- This is a steady state situation and the current remains constant



HiPIMS - Voltage - Current - time

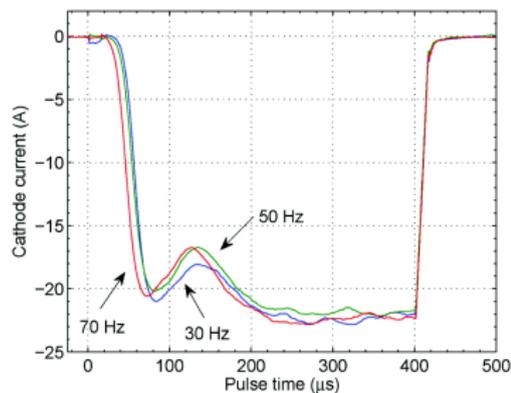
- The bottom curve represents a range of low self-sputtering, $\Pi_{ss} < 0.1$ and the discharge physics in the plateau/runaway phase is dcMS-like
- The middle range of power densities, with $0.1 < \Pi_{ss} < 1$, represents partially self-sputtering discharge
- The top curve represents self-sputtering runaway which requires $\Pi_{ss} > 1$ and a self-sputter yield $Y_{ss} > 1/(\alpha\beta_t) > 1$



From Gudmundsson et al. (2012), JVSTA **30** 030801

HiPIMS - Voltage - Current - time

- Ar discharge with Ti target
- The initial peak in current results large flux of atoms from the target
- Collisions of the sputtered atoms with the working gas result in heating and expansion of the working gas – **rarefaction**
- A significant fraction of the sputtered atoms experience electron impact ionization (the ionization mean free path ~ 1 cm) and are attracted back to the target to participate in the sputtering process – **self-sputtering**

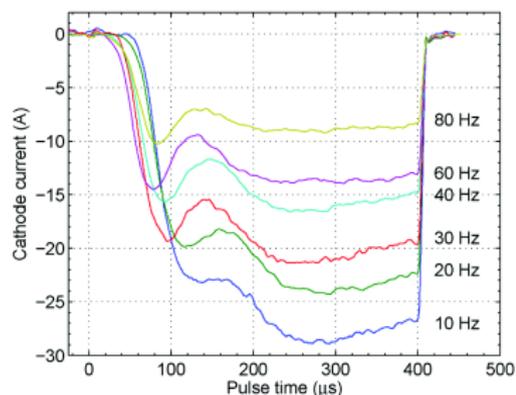


From Magnus et al. (2011) JAP **110** 083306



HiPIMS - Voltage - Current - time

- During reactive sputtering, a reactive gas is added to the inert working gas
- The current waveform in the reactive Ar/N₂ HiPIMS discharge is highly dependent on the pulse repetition frequency, unlike for pure Ar
- N₂ addition changes the plasma composition and the target condition can also change due to the formation of a compound on its surface

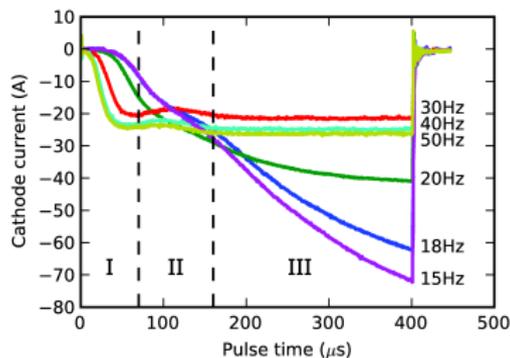


From Magnus et al. (2011) JAP **110** 083306



HiPIMS - Voltage - Current - time

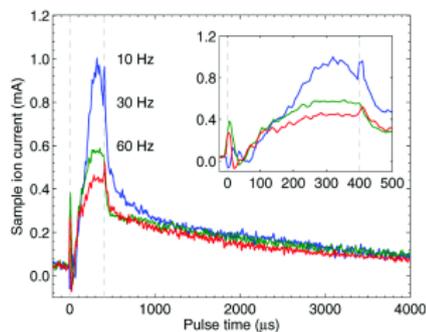
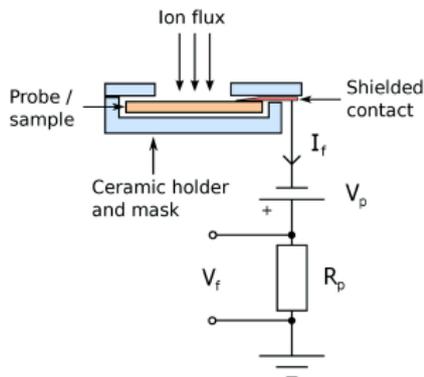
- Similarly for the Ar/O₂ discharge, the current waveform is highly dependent on the repetition frequency and applied voltage which is linked to oxide formation on the target
- The current is found to increase significantly as the frequency is lowered



From Magnus et al. (2012), JVSTA **30** 050601

HiPIMS - Voltage - Current - time

- The observed changes in the discharge current are reflected in the flux of ions impinging on the substrate



From Magnus et al. (2011), JAP **110** 083306

HiPIMS - Voltage - Current - time

- The discharge current I_d is the sum of the ion current I_i and the secondary electron current $I_i\gamma_{SE}$ or

$$I_d = I_i(1 + \gamma_{SE})$$

where γ_{SE} is the secondary electron emission coefficient of the target material

- Also

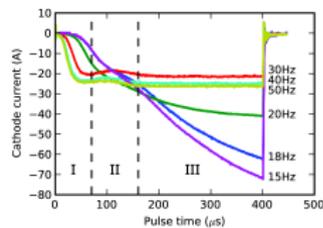
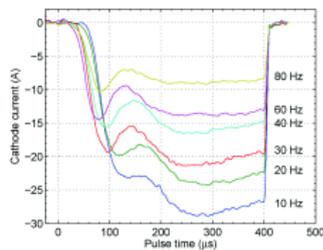
$$I_i \propto n_i \propto \frac{1}{\mathcal{E}_T}$$

- The total energy loss per electron-ion pair lost from the system \mathcal{E}_T is expected to increase with the addition of nitrogen
- We must turn to the secondary electron emission yield to explain the self-sputtering runaway and observed frequency dependence of the current in the reactive discharge



HiPIMS - Voltage - Current - time

- HiPIMS differs significantly from dcMS, due to the fact that self-sputtering quickly becomes dominant and the working gas ions (mostly Ar^+ and N_2^+ or O_2^+) are depleted from the area in front of the target, due to rarefaction
- The secondary electron emission yield is governed by the composition of the target (Ti or TiN or TiO_2) and the type of ions that are bombarding it



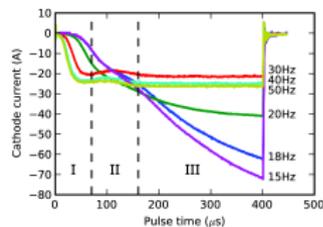
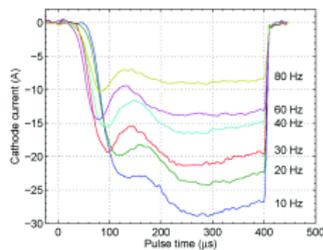
From Magnus et al. (2011), JAP **110** 083306

and Magnus et al. (2012), JVSTA **30** 050601



HiPIMS - Voltage - Current - time

- γ_{SE} is practically zero for singly charged metal ions impacting a target of the same metal
- γ_{SE} will be higher for self sputtering from a TiN or TiO₂ target, where N⁺-ions or O⁺-ions are also present, than for self-sputtering from a Ti target, where multiply charged Ti ions are needed to create secondary electrons



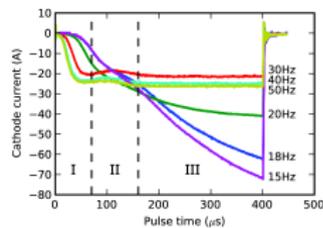
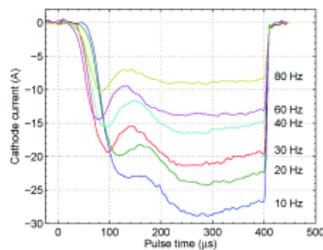
From Magnus et al. (2011), JAP **110** 083306

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HiPIMS - Voltage - Current - time

- At high frequencies, nitride or oxide is not able to form between pulses, and self-sputtering by Ti^+ -ions (singly and multiply charged) from a Ti target is the dominant process
- At low frequency, the long off-time results in a nitride or oxide layer being formed on the target surface and self-sputtering by Ti^+ - and N^+ -ions or O^+ -ions from TiN or TiO_2 takes place



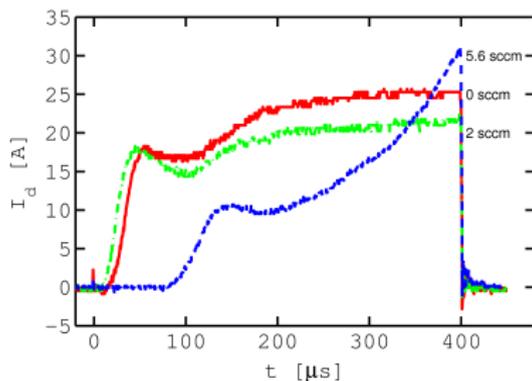
From Magnus et al. (2011), JAP **110** 083306

and Magnus et al. (2012), JVSTA **30** 050601



HiPIMS - Voltage - Current - time

- As the oxygen flow is increased a transition to oxide mode is observed – The delay in the onset of the current increases, the initial current increases, the initial current peak is lowered and a transition to a self-sputtering runaway occurs
- It has been confirmed that in the oxide mode, the discharge is dominated by O^+ -ions, due to oxygen atoms sputtered off the target surface



The current waveforms for an Ar/O₂ discharge with a Ti target where the oxygen flow rate is varied – 600 V, 50 Hz and 0.6 Pa

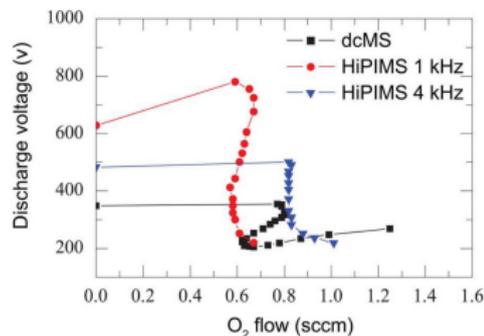
From Gudmundsson et al. (2013), ISSP 2013, p. 192

Hysteresis



HiPIMS - Hysteresis

- Typically, a hysteresis effect is seen in reactive sputtering
- The hysteresis effect originates from the changing target conditions due to the reaction of the target surface with the reactive gas
- Sputtering at low reactive gas flows is referred to as metal mode sputtering
- Sputtering at high flows of reactive gas is referred to as compound mode or the poisoned mode sputtering



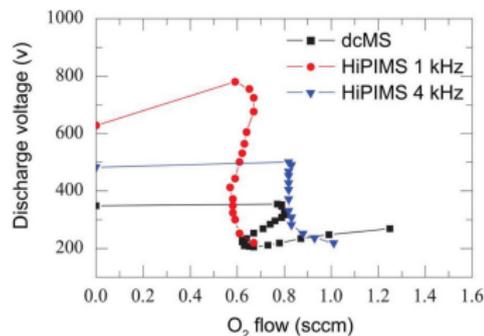
The discharge voltage V_d as a function of the O₂ flow rate during reactive dcMS and HiPIMS of a cerium target.

Aiempanakit et al. (2011), TFSF 519 7779



HiPIMS - Hysteresis

- The hysteresis occurs if the effective sputter rate of the compound is lower than for the pure metal
- Also, there is a change in the secondary electron emission yield as compound is formed
- For dcMS – oxygen flow rate in the range 0.6 – 0.8 sccm the discharge can be operated at three different target voltages
 - The upper curve refers to metal mode sputtering
 - The lower curve to compound mode sputtering



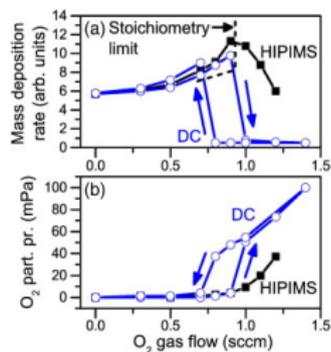
The discharge voltage V_d as a function of the O₂ flow rate during reactive dcMS and HiPIMS of a cerium target.

Aiempanakit et al. (2011), TFS 519 7779



HiPIMS - Hysteresis

- There have been somewhat conflicting reports on the hysteresis effect in HiPIMS discharges
 - Some reports emphasize the need for feedback control (Audronis and Bellido-Gonzalez, 2010; Audronis et al., 2010; Vlček et al., 2013)
 - others report a significant reduction of the hysteresis effect (Sarakinis et al., 2008; Kubart et al., 2011; Aiempanakit et al., 2011)
 - and even elimination (Wallin and Helmersson, 2008; Hála et al., 2012).

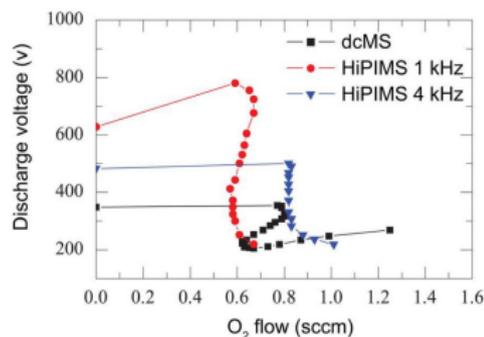


Rate of deposited mass (a) and O₂ partial pressure (b) as a function of O₂ gas flow for HiPIMS and DC sputtering in Ar/O₂ discharge with Al target.

Wallin and Helmersson (2008), TSF 516 6396

HiPIMS - Hysteresis

- Aiempanakit et al. (2011) demonstrate suppression or elimination of the hysteresis
- For a dcMS a relatively wide unstable region is observed while for an HiPIMS the width of the unstable region is substantially smaller
- There is no consensus on the hysteresis effect in the HiPIMS discharge



The discharge voltage V_d as a function of the O₂ flow rate during reactive dcMS and HiPIMS of a cerium target.

Aiempanakit et al. (2011), TSF 519 7779

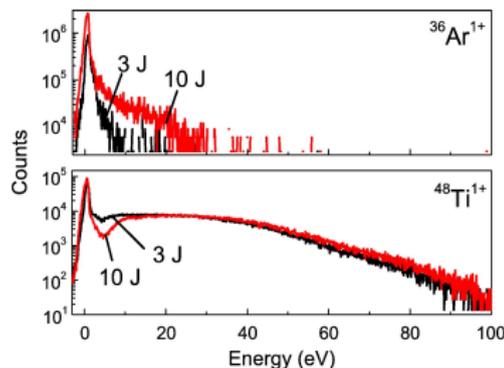


Ion energy and composition



HiPIMS - Ion energy and composition

- For non-reactive HiPIMS the IEDs show
 - the metal ions – an intense high energy tail extending to the limit of the measurement equipment
 - the ions of the working gas exhibit much lower energy
- For comparison the IEDs from dcMS show a peak at an ion energy of about 2 eV and a high-energy tail that extends to around 20 and 40 eV for Ar⁺ and Ti⁺, respectively

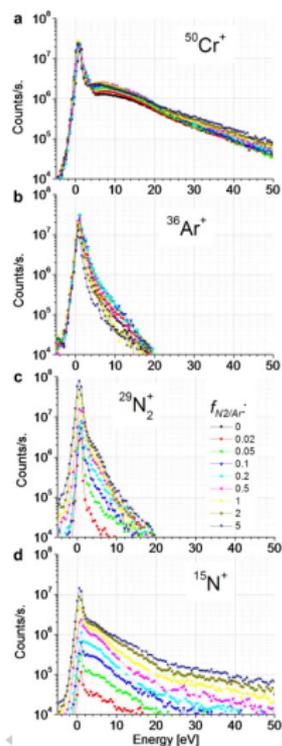


Bohlmark et al. (2006), TSF 515 1522

HiPIMS - Ion energy and composition

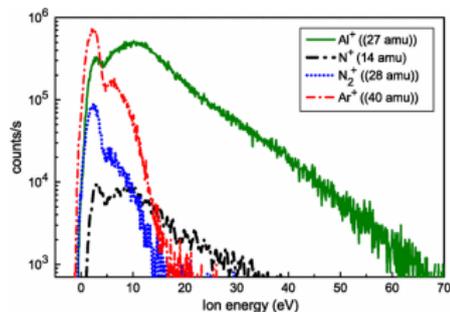
- Reactive Ar/N₂ sputtering of a Cr target
- The Cr⁺ ions comprise an intense low energy peak and pronounced high energy tails
- For reactive sputtering in Ar/N₂ discharges, low energy N₂⁺-ions and energetic N⁺-ions are present in the reactive mode
- The IED for the N⁺-ion possesses a high energy tail just like the Cr⁺-ions (or the Cr²⁺ ions), which is not observed for Ar⁺ or N₂⁺

Greczynski and Hultman (2010), Vacuum **84** 1159



HiPIMS - Ion energy and composition

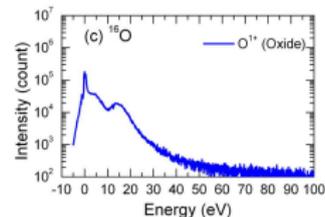
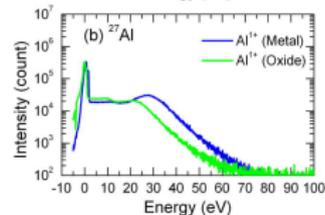
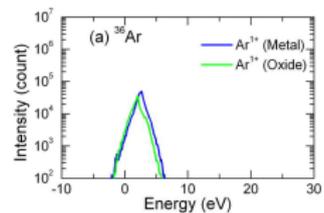
- Similar findings have been reported
 - Ar/N₂ discharge with Ti target – N⁺ behaves like Ti⁺
(Lattemann et al., 2010; Ehiasarian et al., 2007)
 - Ar/N₂ discharge with Al target – N⁺ behaves like Al⁺
(Jouan et al., 2010)
- The working gas ions and the molecular ion of the reactive gas present a similar IED
- The metal ion and the atomic ion of the reactive gas present similar IED and extend to very high energy



Jouan et al. (2010), IEEE TPS **38** 3089

HiPIMS - Ion energy and composition

- The time-averaged ion energy distributions (IED) of the ions in the metal and oxide modes of and Ar/O₂ discharge with Al target
- There is no difference for the distributions of Ar⁺-ions between the metal and oxide modes
- There is a considerable amount of O⁺-ions

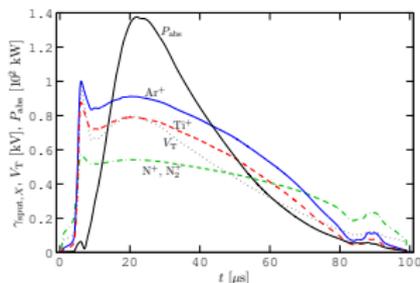


Global model studies of reactive HiPIMS



Global model studies of reactive HiPIMS

- A global model is applied to study a N_2/Ar discharge with Ti target
 - Electrons
 - metastable nitrogen molecule $N_2(A^3\Sigma_u^+)$ (6.17 eV)
 - nitrogen atoms $N(4S)$, $N(2D)$ (2.38 eV) and $N(2P)$ (3.58 eV)
 - nitrogen ions N_2^+ (15.6 eV) and N^+ (14.5 eV)
 - argon atoms $Ar(3s^23p^6)$, Ar^m ($1s_5$ and $1s_3$) (11.6 eV), Ar^r ($1s_4$ and $1s_2$) (11.7 eV), excited argon atoms 4p states $Ar(4p)$ (13.2 eV)
 - argon ions Ar^+ (15.8 eV)
 - titanium atom $Ti(a^3F)$ and titanium ion Ti^+ (6.83 eV)

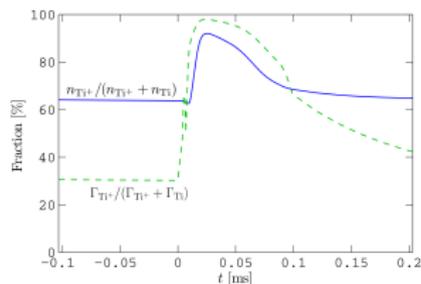


The temporal evolution of the sputtering yields $\gamma_{sput,X}$, the absorbed power P_{abs} and the target voltage V_T .



Global model studies of reactive HiPIMS

- The discharge pressure is 10 mTorr and the total gas flow is $Q = 42$ sccm which is 95% argon ($Q_{\text{Ar}} \simeq 40$ sccm, $Q_{\text{N}_2} \simeq 2$ sccm) and the gas temperature is assumed to be $T_g = 430$ K
- The pulse length is roughly $100 \mu\text{s}$ (FWHM of about $32 \mu\text{s}$) and the repetition frequency is 500 Hz (i.e. a period of $T = 2$ ms)
- The fraction of ionized metal flux at the substrate is significantly larger than the ionized metal fraction when the power is on but significantly smaller when it is off

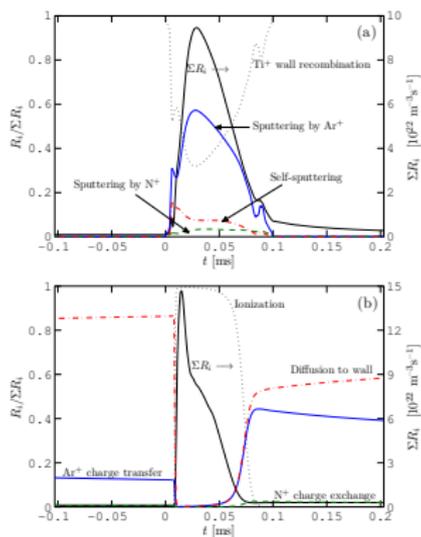


The temporal evolution of the ionized metal fraction $n_{\text{Ti}^+} / (n_{\text{Ti}^+} + n_{\text{Ti}})$ and the fraction of ionized metal flux at the substrate $\Gamma_{\text{Ti}^+} / (\Gamma_{\text{Ti}^+} + \Gamma_{\text{Ti}})$ at and around the tenth pulse period.



Global model studies of reactive HiPIMS

- The most important reactions for creation of Ti atoms, are wall recombination of Ti^+ and sputtering by Ar^+ , Ti^+ and N^+
- The most important reactions for the loss of Ti atoms are electron impact ionization, diffusion to the wall, and Ar^+ and N^+ charge transfer

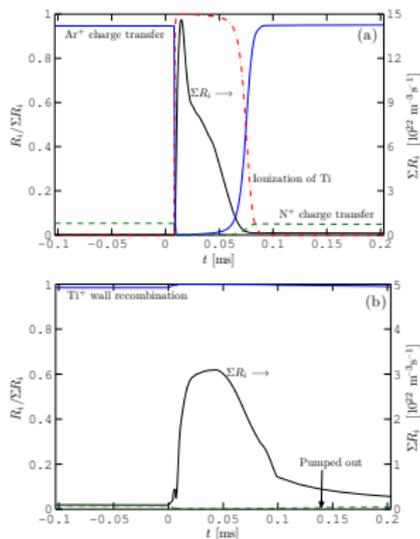


The temporal evolution of (a) the creation of Ti and (b) the loss of Ti atoms over 300 μs at and around the tenth pulse.



Global model studies of reactive HiPIMS

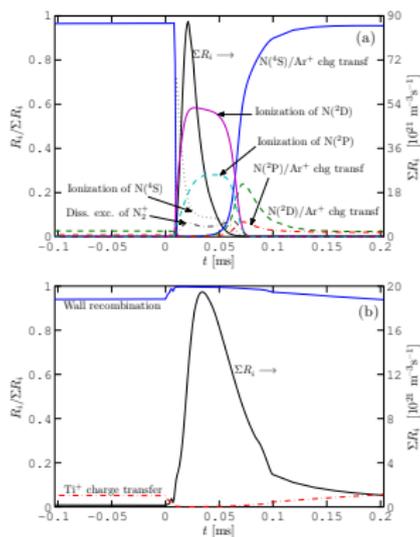
- Electron impact ionization is the dominating reaction in the creation of Ti^+ ions while the power is on but $Ar^+ - Ti$ charge transfer is the dominating reaction after the power is turned off
- Ti^+ ions are almost entirely lost to wall recombination



The temporal evolution of (a) the creation of Ti^+ and (b) the loss of Ti^+ ions over 300 μs at and around the tenth pulse.

Global model studies of reactive HiPIMS

- Electron impact ionization is most important in N^+ production when the power is on, and $N - Ar^+$ charge transfer when the power is off
- Electron impact ionization of $N(4S)$ is only most important for the first few μs after the power has been turned on
- The excited atoms are much less important during the off period when essentially all N^+ ions are created by $Ar^+ - N$ charge transfer



The temporal evolution of (a) the creation of N^+ and (b) the loss of N^+ ions over 300 μs at and around the tenth pulse.



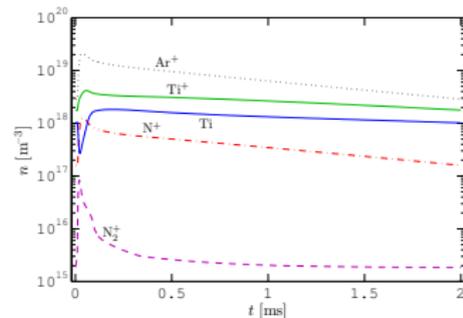
Global model studies of reactive HiPIMS

- A global (volume averaged) model of an N_2/Ar discharge was applied to study the reaction mechanism in a HiPIMS discharge with a titanium target
- It is based on a global model of a HiPIMS discharge in argon

Gudmundsson (2008a) J. Phys.: Conf. Ser. **100** 082013
- and a global model of the N_2 discharge

Thorsteinsson and Gudmundsson (2009a,b)

PSST **18** 045001 and 045002



The temporal evolution of the densities of titanium atoms and positive ions over the tenth pulse period.



Summary



Summary

- The current-voltage-time waveforms in a reactive discharge exhibit similar general characteristics as the non-reactive case
 - the current rises to a peak, then decays because of rarefaction before rising to a self-sputtering dominated phase
- At low repetition frequency, the long off-time results in a nitride or oxide layer being formed on the target surface and self-sputtering by Ti^+ and N^+ or O^+ -ions from TiN or TiO_2 takes place with an increase in secondary electron emission yield and a corresponding increase in discharge current



Summary

- There is no consensus on the hysteresis effect in the HiPIMS discharge
- The working gas ions and the molecular ion of the reactive gas present a similar IED
- The metal ion and the atomic ion of the reactive gas present similar IED and extend to very high energy



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The slides can be downloaded at

<http://langmuir.raunvis.hi.is/~tumi/hipims.html>



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