Current–voltage characteristics of an impulse magnetron discharge in target material vapor

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Abstract. The magnetron discharge with hot (uncooled) target in an impulse mode has been experimentally investigated. The I–V characteristics have been measured depending on the magnetic field strength for three target materials: copper, chromium, and silicon. For melted copper and hot chromium targets, stable gasless (no argon) operation of the magnetron has been demonstrated with maximum impulse power densities about 2.5 kW/cm² (averaged over the racetrack area). For silicon target, maximum impulse power density was 1.5 kW/cm² at low argon pressure (0.1 Pa). The magnetic field dependences of discharge parameters have shown the associated changes in differential plasma impedance.

1. Introduction
Magnetron sputtering is among the main tools for deposition of thin-film coatings. Despite the wide application of magnetron sputtering systems, in this area there is a high demand in improving both the quality of films and the efficiency of their preparation process. In the past 10 years, special attention of researchers and innovative industries dealing with thin-film technologies has been focused on the pulsed regimes of magnetron operation, mostly on the high-power impulse magnetron sputtering (HiPIMS) with long (> 1 ms) [1, 2] and short (< 1 ms) [3–5] pulses. They provide an increase in the density and adhesion of films, and also allow tailoring the film structure within a wide range due to outstandingly high degree of plasma ionization [6].

Another modification of magnetron sputtering that is becoming more and more popular is hot target sputtering [7–10], and in the extreme cases, gasless hot (or melted) target sputtering [11–14] (MHT, or MLT). These devices demonstrate extraordinary deposition rates, e. g. liquid copper target magnetron [8] provides film growth rate as high as 2.5 μm/min/kW at a distance of three target diameters above the target surface due to additional mechanism of deposition—evaporation. For certain materials, in these magnetrons the working (argon) gas inlet can be completely closed, and the discharge would...
operate exclusively in the target material vapor. In such deposition processes, the number of coatings defects associated with the impact of inert gas species can be minimized.

We have recently demonstrated the combination of two approaches (long HiPIMS and MHT) with a copper target [15] to achieve high plasma ionization levels, large deposition rates, and to increase the energy efficiency of the process due to optimized utilization of power that heats up the magnetron target. A distinctive feature of the discharge form we have been studied is its quasi-stationary behavior, that is, maintaining high constant values of discharge current and voltage, as well as ion density, throughout the discharge pulse (up to tens of ms) [15]. We propose a quite evident term of impulse magnetron with liquid (or hot) target (IMLT or IMHT) to name these magnetron regimes.

In present contribution, we make a deeper insight into the topic and report on overall observations and voltage/current relations measurements of an impulse magnetron discharge in target material vapor operated with Cu, Cr, and Si targets with and without the working gas (Ar). These materials are extensively utilized in thin-film industries, e.g. for creation of protective wear and corrosion-resistant Cr-based coatings in automotive field; Cu metallization of VLSI in microelectronics, as well as preparation of corrosion-resistant Si-based coatings.

2. Experimental setup
The experimental setup consists of a specially designed planar magnetron where a target is thermally insulated from a water-cooled magnet system by a set of tungsten spacers [15]. In the experiments, 76.2 mm diameter targets with different thicknesses were used: Cu (99.99%), Cr (99.95%), and Si (99.999%) (Beijing Goodwill Metal Technology Co., Ltd.). Cu target was placed in a Mo crucible. Cr target was operated in a sublimation regime, and no crucible was needed. For the reported experiments, we used graphite crucible for Si melting since the interaction between Si and C runs relatively slow. The main thermal parameters of the target materials used are presented in Table 1.

| Material | Type          | Melting point (°C) | Thermal conductivity (W/m/°C) | Temperature corresponding to 1 Pa vapor pressure (°C) [16] |
|----------|---------------|--------------------|-------------------------------|----------------------------------------------------------|
| Cu       | Conductor     | 1083               | 401                           | 1273                                                     |
| Cr       | Conductor     | 1857               | 93.9                          | 1205                                                     |
| Si       | Semiconductor | 1415               | 130                           | 1500                                                     |

Table 1 shows that unlike copper and silicon, chromium target enables producing high vapor pressure (1 Pa) at temperatures well below the melting point. It means that for Cr, gasless operation of the discharge does not require actual melting of the target.

Magnetic field at the target surface was varied by moving the magnets assembly along the magnetron’s symmetry axis. In order to specify the magnetic field configuration, in the figures and throughout the text we indicate $B$-field as $B_r$ value in the racetrack center measured by a Hall probe 2 mm above the target surface. Determined in this manner, magnetic field could be tuned from 260 to 530 G during the experiments. The setup scheme is presented in Figure 1.

The experiments with each target were carried out in the following order. The vacuum chamber was evacuated to a base pressure of $1 \times 10^{-4}$ Pa. Then argon gas (99.998% pure) was introduced, and DC magnetron discharge was switched on at working pressure of 1 Pa. DC discharge parameters for copper, silicon, and chromium targets are shown in Table 2. Under ion bombardment, the target was heated to high temperatures, and eventually the material started to evaporate intensively either after melting (Cu, Si) or while remaining solid (Cr). When the pressure of target material vapor was high enough, argon inlet was closed, and DC magnetron discharge continued to glow exclusively in this vapor. The moment for argon inlet closing was determined by stabilization of DC discharge voltage
and current readings together with the emission color change [11]. Then the high-power pulses were applied to the cathode with different voltage loads and at various magnetic field values. The pulse duration in all cases was set at $\tau_{\text{pulse}} = 20$ ms.

![Figure 1. Experimental setup.](image)

The discharge voltage was measured with Pintek DP-100 differential probe. The discharge current was measured by Honeywell CSNR 161 current sensor. The electrical signals were recorded by digital storage oscilloscope SIGLENT AKIP-4126/3A-X.

| Table 2. DC magnetron discharge parameters for Cu, Cr, and Si targets. |
|-----------------|-----|------|-----|
| Target material | $P_{\text{DC}}$ (kW) | $U_{\text{DC}}$ (V) | $I_{\text{DC}}$ (A) |
| Cu              | 1   | 560–690 | 1.4–1.8 |
| Cr              | 2   | 440–730 | 2.5–4.5 |
| Si              | 2.3–2.7 | 660–930 | 2.5–4.0 |

3. Results
Typical waveforms of voltage and current pulses for all three target materials are presented in Figure 2. Long quasi-stationary HiPIMS (L-HiPIMS) mode is characterized by longer pulses (1–200 ms), as compared to conventional HiPIMS concepts [1]. The discharge parameters in L-HiPIMS are almost constant during a single pulse provided the pulse-on time is lower than 5 ms. Generally, the current decreases with time linearly—for copper target the discharge current decrease rate is 0.4 A/ms, for chromium it is 0.1 A/ms, while for silicon the current remains constant even at 20 ms pulse.

The Si target is peculiar since it is semiconductive. Moreover, the specific volume of liquid Si is lower than that of the solid, hence once the racetrack region is melted, it rapidly becomes depleted of the material, and large amounts of Si are needed to prepare the convenient liquid Si target.
The performance of power supply was tested by measuring the discharge current waveform for different pulse durations. Discharge current recorded for the IMLT with copper target is shown in Figure 3 for different pulse-on times: 10, 20, and 40 ms. It has been shown that the pulse shape remains the same despite varying the pulse-on time.

Figure 3. Cu IMLT discharge current waveforms for different pulse-on times $\tau_{\text{pulse}}$.

The $I$–$V$ curves of the magnetron discharge with Cu (Figure 4a) and Cr (Figure 4b) targets have been achieved in gasless (self-sputtering) regime. The $I$–$V$ characteristics of IMLT with Si target (Figure 4c) has been achieved at argon pressures of 1 Pa and 0.1 Pa, however no true gasless operation has been demonstrated.

Figure 4. $I$–$V$ curves for copper (a), chromium (b), and silicon (c) targets.
When the magnetic field is low, the discharge voltage is higher for the same current values. The lower the magnetic field, the higher is the voltage, however the maximum discharge power is lower. Attempting to exceed the maximum power in our experimental configuration in each case led to formation of an arc instead of a confined magnetron discharge. For comparison, the $I–V$ curves of Cu IMLT with and without Ar are shown in Figure 5.

In conventional magnetron sputtering devices, the relationship between the discharge current density and the discharge voltage is $j_d \propto U^\delta$, where $2 < \delta < 20$ [4]. IMLT with copper target is characterized by two distinct $I–V$ regions that are linear in the logarithmic scale. These regions are characterized with different $\delta$ factors. At low magnetic field values (below 36 mT), the $I–V$ curve shows two regions with positive differential impedance. At 36 mT B-field, in the low-current region the voltage is independent of current meaning that plasma differential impedance is virtually zero. When $B$-field is higher than 36 mT, the low-current part of the curve demonstrates negative differential impedance. For IMHT with Cr and Si targets, the $I–V$ curves in log scale demonstrate only one region with a fixed $\delta$ factor.

The impact of the gasless operation on the $I–V$ curves is illustrated in Figure 5 for Cu target.

![Figure 5. $I–V$ curves for Cu IMLT with 1 Pa argon (solid symbols) and for gasless Cu IMLT (open symbols).](image)

For each discharge voltage, gasless operation results in higher discharge current densities that indicates both the higher ionization efficiency of metal atoms and the vapor pressure increase.

4. Conclusion
Current–voltage characteristics of an impulse magnetron discharge with hot copper, chromium, and silicon targets have been studied for different magnetic field strength of the magnetron.

It has been shown that maximum impulse power densities about 2.5 kW/cm$^2$ can be achieved in gasless self-sputtering impulse discharge with copper and chromium targets. For silicon target, maximum impulse power density was 1.5 kW/cm$^2$ at low argon pressure (0.1 Pa).

The $B$-field dependence of $I–V$ curves has revealed changes in plasma differential impedance for all materials used.

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