Investigation of gas discharge features during HiPIMS using optical emission spectroscopy

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Abstract. The paper presents the results of the investigation of the emission spectra of a gas discharge during high-power impulse magnetron sputtering (HiPIMS) of a titanium target at different power supply parameters. In such mode, the fraction of ionized atoms of the target material in the discharge increases significantly, what affects the properties of the deposited coatings. The characteristic features of the operation of the system in HiPIMS mode were singled out, and also the discharge spectra were compared at high-power impulse and conventional (sputtering on a direct current) operation modes of the magnetron. As a result of the study, it was shown that the use of optical emission spectroscopy during HiPIMS makes it possible to effectively determine the parameters of the stable operation of the system and determine the boundary at which a transition into high-power regime occurs.

1. Introduction
One of the most promising methods for applying conductive and dielectric coatings is HiPIMS (High-Power Impulse Magnetron Sputtering) [1–5], the first mention of which was made in the late 90's. During two decades, this technology has overcome the long path of development and adaptation and now occupies a significant place in industrial production (microelectronics and machine building). Using this method, the percentage of ionized atoms of the sputtered material can reach 90 % [1–3]. This and a number of other parameters significantly improve such properties of the formed coatings as their adhesion to the substrate, density and uniformity.

In this paper, a study of the gas discharge of a magnetron during HiPIMS by optical emission spectroscopy is presented. This tool of diagnosing the discharge parameters allows determining its component composition and other properties quite accurately [4–5]. The measurements were carried out at different settings of the high-power unit: frequency, peak voltage, and pulse duration. When a high-power pulsed magnetron power supply unit operates at low voltages (pulse amplitude up to ~500 V) in argon medium, necessary for the synthesis of high-density coatings discharge conditions might not occur. The reason for this is the low gas and the sputtered substance ionization (these parameter corresponds to the discharge of a classic magnetron which is operating on a direct current). Optical emission spectroscopy of the discharge is an effective way to determine the transition to the HiPIMS regime.

2. Experimental
Experiments were carried out at the upgraded vacuum deposition unit UVN-71. The residual pressure in the chamber was 0.02 mTorr, the working pressure of argon (purity 99.95 %) was 0.6 mTorr. A
planar 6 mm thick magnetron with a diameter of 115 mm with a cooled titanium target (99.9 % purity) was used as the sputtering system. To operate the magnetron in high-power pulsed mode, a specialized power unit with a peak voltage value of 1000 V and current of 1000 A was used (the average power was not more than 5 kW).

To perform the spectral analysis, the ISM3600 spectrophotometric attachment with the special software complex Aspect 2010 was used, with the help of which the data obtained during the experiments were processed.

For the study, the most intense spectral lines corresponding to the emission of titanium and argon atoms and ions were chosen. In figure 1(a), (b) discharge emission spectra corresponding to magnetron sputtering at direct current (DC mode), direct impulse current (DC-pulse (PM)) and high-power impulse mode (HiPIMS) are shown.

![Figure 1](image_url)

**Figure 1.** Spectra of the gas discharge of a magnetron during sputtering in HiPIMS and DC (a) and DC and DC-PM (b) modes.

In figure 2(a)–(c) the dependences of the intensities of the spectral lines Ti I (atom) on the peak voltage of the pulse at its various durations are shown. At pulse duration of 50 μs, the radiation intensity is small regardless of frequency, what may indicate a low deposition rate in this mode. At pulse duration of 100 and 150 microseconds, a significant increase in the intensities of the spectral lines is observed. At a voltage of 540–560 V, the increase of the growth rate of the intensities of the lines under consideration is well seen.

In figure 2(d)–(f) analogous dependences for Ti II (single-charged ion) are presented. It can be seen from the dependences that the degree of ionization at a pulse duration of 50 μs is small. With a further increase in the pulse duration, it significantly increases. In this case, the intensification of the growth of spectral lines occurs even at a voltage of 520–530 V.

Figure 3 shows the graphs for Ar I ((a)–(c)) and Ar II ((d)–(f)). The dependences for argon shown in figure 3 correlate with analogous ones for titanium - with the increase of maximum pulse voltage, the intensity of all lines increases. There is also a critical value of this voltage (inflection point) at which the growth rate of spectral lines increases.

In figure 4 the ratio of the main spectral lines as a function of the frequency and duration of the pulse for voltages of 500, 540, and 580 V is shown in the form of column diagrams.

Figure 4 clearly represents the distribution of the emission intensities of various particles in the discharge. At a frequency of 100 Hz and a pulse duration of 50 μs (figure 4(a)), the dependence is similar to sputtering in the DC mode – the lines corresponding to the emission of titanium atoms predominate. With further growth of the pulse frequency and duration, redistribution of the intensities of the atomic lines (Ti I) and ions (Ti II) of the target material occurs - the fraction of the ionized component noticeably increases. The spectral lines corresponding to the emission of titanium ions show the highest intensity, what indicates a transition to the real HiPIMS regime. At the same time,
with the increase of the frequency and duration of pulses, the intensities of emission lines for all
groups of charged particles increase, without a significant redistribution of their ratio.

Figure 2. Intensities of the spectral lines Ti I and Ti II at a pulse duration of 50 (a), 100 (b), (e) and 150 (c), (f) microseconds.

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Figure 3. Intensity of the spectral lines of Ar I and Ar II at a pulse duration of 50 (a), 100 (b), 150 (c), and 100 Hz (d), 200 Hz (e), 300 Hz (f) μs.

Figure 5 shows the emission spectra of a gas discharge measured at a voltage of 600 V and a pulse duration of 50 μs, but at different frequencies which were 100 Hz (HiPIMS (−)) and 200 Hz (HiPIMS (+)). "−" indicates the absence of the HiPIMS mode, the amplitude of the Ti I spectral lines
predominates over Ti II; "+" – the inverse mode, in which the spectrum takes the opposite form and corresponds to the HiPIMS mode. At a frequency of 100 Hz, the lines corresponding to the emission of neutral titanium atoms have the highest intensity, which is typical for operation in the classical DC mode of the magnetron, while when increasing the frequency up to 200 Hz, the line of ionized titanium acquires the highest intensity. This indicates a transition from low-power to high-power mode. For the technology of manufacturing film structures, a precise determination of the magnetron operation modes is an important task, the solution of which allows achieving efficient deposition of high-quality coatings with specified characteristics.

**Figure 4.** The ratio of the intensities of the spectral lines in the discharge for frequencies and pulse widths of 50 μs and 100 Hz (a), 50 μs and 200 Hz (b), 100 μs 100 Hz (c), respectively.

**Figure 5.** Spectra of the gas discharge of a magnetron under different operating conditions.

From the above data follows the fact that optical emission spectroscopy is an effective way of determining the modes of operation of a magnetron during the usage of HiPIMS technology. With the help of this diagnostic method it is possible not only to determine the state of the gas discharge, but also to estimate the ratio of charged particles in it.

3. Conclusion

A high-power gas discharge of a magnetron was studied using optical emission spectroscopy. To achieve the most efficient operation in the HiPIMS mode, optimum frequency and pulse width settings of a high-amplitude power supply are required. During magnetron sputtering of a titanium target at frequencies below 100 Hz and pulse duration less than 50 microseconds, at voltages no higher than 580 V, the qualitative picture of the distribution of the intensities of the spectral lines is similar to the magnetron sputtering at a direct current (DC mode). At a relatively low peak voltage, short pulse duration and their frequency, it is not possible to achieve the efficient ionization of sputtered target and
gas atoms in a vacuum chamber, what doesn’t allow using the advantages of HiPIMS technology. Optical emission spectroscopy is an effective tool for determining the transition point to a high-power pulse mode.

The possibility of qualitative analysis of the discharge was determined on the basis of measuring the spectral characteristics of the radiation. This makes it possible to quickly determine the mode in which the magnetron sputtering system operates at the given moment and, if necessary, make the appropriate adjustment, by changing the parameters of the power unit.

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