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# Surface erosion of hot Cr target and deposition rates of Cr coatings in high power pulsed magnetron sputtering



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### ARTICLE INFO

#### ABSTRACT

Keywords: High power pulsed magnetron sputtering (HPPMS) Cr coatings Hot target Evaporation and sublimation High power pulsed magnetron sputtering (HPPMS) of a hot chromium target by using a combined power source is the aim the present research. The combination of HPPMS technique and hot target can be effective way to produce Cr coatings. The investigations were performed by using a power supply with pulse frequency of 500 Hz, duty cycle of 4% in the range of power density averaged over pulse period from 15 to  $65 \text{ W/cm}^2$ . The specific feature of the investigation is that sputtering and sublimation of the target occur simultaneously. The experimental results and the calculations revealed the important role of sublimated Cr atoms concentration near the target in increasing the pulsed and average discharge current and in setting up the self-sputtering mode. The erosion yield of the hot Cr target is determined, and in the investigated power range, it increases from 1.5 to 30 atoms per ion due to sublimation. As a result, the coating deposition rate by HPPMS from hot solid target can be increased 10–30 times in comparison to cooled target sputtering.

# 1. Introduction

High power pulsed magnetron sputtering (HPPMS) is widely used both in industry and scientific research as a tool to produce high-quality functional coatings for several purposes. A distinguishing feature of HPPMS is the high power generation during pulses. The peak of power during a pulse is approximately 2-3 orders of magnitude higher than the magnetron power averaged over the pulse period. The pulse repetition frequency is usually in the range from 0.1 to 10 kHz [1]. However, the averaged power of HPPMS power supplies is in the same range of those for direct current (DC) or middle-frequency (MF) magnetron sputtering systems. Due to the high pulse power, much higher instantaneous fluxes of both deposited particles and ions are produced. These circumstances contribute to a significant improvement in the properties of the coatings and thin films. The several possibilities in varying the pulse duration, magnitude of pulse current, and also in the use of additional means capable to adjust the composition and energy of erosion flow, have provided this method with the ability to control the structural and functional properties of coatings within a wide range [1-5].

One of the disadvantages of powerful pulsed magnetrons is the instability of their operation caused by the uncontrolled transition of a glow discharge into a high-current state and the possibility of disruption into an arc discharge [6-8]. To overcome this problem, it has been proposed to use combined electric power supplies for magnetrons. For example, modulated pulsed power sputtering shows good results [9–11]. In this case, a power is applied to sputtering target by synchronizing the low-current (DC or MF) with the high-power supplies [12]. As a result, the work period of such sputtering system consists of two stages: firstly, a pre-ionized stage with low power and background plasma densities of about  $10^9 \text{ cm}^{-3}$  is produced by DC or MF power supply, and at this stage a weak gas ionization is maintained in plasma discharge; then, a high-current pulse is generated by HPPMS power supply. Such procedure of power pulsed sputtering is quite widely adopted in practice.

However, in technologies based on HPPMS there is still a significant disadvantage, that is a low coating deposition rate, lower in comparison to conventional DC or MF magnetron sputtering [13–15]. This is because the main mechanism for creating an erosive flow of atoms on the target surface is sputtering. For DC sputtering, deposition rates of metal coatings does not exceed 10 nm/s at target power density within  $5-50 \text{ W/cm}^2$ , substrate target distance within 0.05–0.15 m and working gas pressure below 1 Pa [16,17].

The issue of increasing the coating deposition rate by using technologies based on HPPMS is in the focus of many researchers. For example, some new modifications of power supplies that are able to stabilize deposition and control the flow of sputtered ionized particles are proposed [14,18]. This makes it possible to increase the deposition rate

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by several tens of percentage points. However, it is expected that evaporation of target surface in addition to their sputtering should lead to more substantial improvement of productivity of HPPMS systems.

At present, there are research findings indicating that the most significant increase in the deposition rate of metal coatings (1 or 2 orders of magnitude) occurs by using magnetrons with liquid-phase targets when their surface intensively evaporates [19-22]. There are some technological limitations in the work of these magnetrons. The target must be placed in the crucible and kept in the working chamber in horizontal position. However, to obtain coatings from some metals (for instance, Cr, Ti, Mg) that have a high sublimation rate one can operate without crucible. Then the deposition process can be more convenient and flexible, as in the case of chromium. The industrial demand for chromium coatings is very high as they provide good protective and decorative characteristics. In our previous articles, it was shown that hot Cr target sputtering with MF power supply has high deposition rates. Due to target sublimation, additional to sputtering, deposition rates of Cr coatings can be increased approximately 10 times in comparison to cooled target sputtering at power density about 50 W/  $cm^2$ , target substrate distance 0.08 m and working pressure not more than 0.2 Pa [23,24].

In this regard, there is an issue about the possibility of a significant increase in the deposition rate of high-quality Cr coatings from hot Cr target sputtering with HPPMS power supply in the combined mode. In our case, the combined power supply has a synchronized electrical signal from a low-current DC source and a high-power pulse of microsecond duration. At the first stage, a weak ionization of the discharge gap occurs, which promotes stability of the current pulse form and total energy within each pulse. The low duration of a high power pulse avoids the forming of arc discharges. We will call such method as HPPMS with DC pre-ionization.

The combination of the advantages of both approaches should substantially widen the technological capabilities of HPPMS systems. However, the implementation of this method into the practice of scientific research and industry requires a systematic study of processes occurring in plasma discharge and methods to control them, mechanisms of target erosion and boundaries of increasing the deposition rate. Such issues are addressed in this article.

#### 2. Description of experiments and diagnostic methods

The experiments on magnetron deposition of chromium coatings were carried out in an argon atmosphere at a pressure of 0.2 Pa. The base pressure was 0.003 Pa. For sake of comparison, a cooled and a hot Cr (99.95%) targets were used, both in the form of disks with the same diameter (0.09 m) and thickness (0.008 m). The structural features of the hot target, which significantly reduce the heat-conducting exchange with the cooled body of the magnetron and thus ensure high-temperature heating of the target, are described in [24]. The magnetron body with cooled target was made of copper, and in the case of a hot target it was made of AISI 321. The magnetic system had an unbalanced configuration similar to the case described in [23]. The spatial distribution of the longitudinal component of the magnetic induction vector  $B_r$  is shown in Fig. 1.

The HPPMS technique was developed with the help of a specially developed combined power source (Applied Electronics, Tomsk, Russia), which consists of a HPPMS power supply (APEL-M-5HiPIMS) and a DC source with low power (APEL-M-5PDC). The pulse frequency of the combined power supply is the same as for HPPMS power source. A DC pulse precedes a high power (HPP) pulse. The current and voltage diagrams within a period are shown in Fig. 2. The operation parameters of the combined power supply are reported in Table 1.

The power of the HPPMS source averaged over a period ( $Q_{HPP,per}$ ) varied in the range from 1000 to 4000 W. The value of the upper limit was conditioned by the need to prevent the hot target degradation because of cracks formation during its heating and melting. The power



**Fig. 1.** Spatial distribution of the longitudinal component of the magnetic induction vector  $B_r$ . Here r = 0 corresponds to the position of the target center.



Fig. 2. (Color online only) Current and voltage diagrams for pulse period of the combined power supply.

## Table 1

Pulse paramet	ers of the	combined	power	source
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Parameter	Notation	Value
HPP frequency	V	100 Hz
HPP period	t HPPsper	2000 μs
HPP sputtering duration	t HPPson	80 μs
DC sputtering duration	t DCson	1895 μs
Pause time	t pause	25 μs

density during HPP sputtering was between 388 and  $1552 \text{ W/cm}^2$ . The DC source power averaged over a period ( $Q_{DC,per}$ ) was about 100 W in all experiments.

The current and discharge voltage were measured by a Tektronix TDS 2022D digital oscilloscope with a LT 200-S/SP48 current gauge.

The optical spectral characteristics of the plasma in a gas discharge region were analyzed using a two-channel (225–920 nm) AvaSpec spectrometer. The integration time was 1.05 ms, the number of measurements for averaging was 20. The focal distance was 0.3 m. To identify spectral lines, the NIST atoms spectra database (version 5.3) was used. The intensities of the optical signals for each plasma component were summed to obtain their integral values. For this, Ar II (270, 527, 541 and 810 nm), Cr I (312, 426 and 530 nm) and Cr II (268, 284,

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