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Hot-target HiPIMS deposition of W-fuzz layers

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Abstract. Porous tungsten coatings were prepared by magnetron deposition in He environment. The depositions were carried out in HiPIMS mode with a frequency of 1 kHz and a pulse duration of 300 μs. The tungsten target was thermally insulated from the water-cooled cathode. The samples were mounted on three different distances from the target. Temperature of each sample was measured with a dedicated thermocouple and recorded throughout the deposition process. The structure of resulting W-fuzz layers is analyzed and discussed. The deposition rate of tungsten fuzz in a hot-target magnetron system operated in HiPIMS mode is shown to be ~100 nm/min.

Keywords: reactive magnetron sputtering, evaporation, HiPIMS, target poisoning, hot-target magnetron, copper, chromium, silicon.

1. Introduction

Nanostructuring of the tungsten (W) surface under the influence of helium (He) ion flows has been studied by many scientific groups since 2006 [1]. Under the influence of helium flows, the formation of fiberform nanostructures (FNs), or so-called "fuzz", can be observed. For a long time, this effect was studied from the point of view of helium accumulation in the near-surface layer [2–5]. Since 2014, studies have begun to appear on the formation of fuzz in magnetron sputtering systems [6]. However, the growth rate of the fuzz layer formation is so low that it takes quite a long time to grow a reasonably thick structure (≤1 μm), typically 5–10 hours, with a characteristic He ion flux ~10^{20} $m^{-2}s^{-1}$.

In our work, we show that much higher deposition rate of a structured W layer can be achieved in magnetron sputtering systems than was previously demonstrated in [6, 7].

2. Experimental procedure

To deposit a layer of tungsten fuzz, a weakly unbalanced circular magnetron (Magneto series by Pinch, LLC) with a 75-mm-diameter and 2-mm-thick thermally insulated tungsten target (99.93%) was used. Thermal insulation was implemented by mounting the target on three U-shaped tantalum supports (1 mm high and 15 mm long), made of 0.1 mm Ta foil. To reduce thermal load on the elements of the installation, a system of screens was used. Tungsten samples, 10×7×0.05 mm^3 in size, were mounted on a holder made of tantalum wire at different distances from the target (see Fig.1). All samples were facing the target racetrack. The vacuum chamber was pumped out to $7 \cdot 10^{-6}$ Torr. After that the pumping speed was reduced, while the residual pressure did not exceed $3.5 \cdot 10^{-5}$ Torr, and the helium working gas (99.9999%) was introduced until the working pressure was set to $3.2 \cdot 10^{-2}$ Torr. For the first 3 minutes of deposition, the magnetron was operated in the DC mode with a power stabilization of 1.5 kW, and then it was switched to the HiPIMS mode with an average power of 1.5 kW, also with a power stabilization, at 1 kHz repetition frequency, and a pulse-on time of 300 μs. Between the pulses, a standby discharge was ignited in the DC mode with a current stabilization of 10 mA and a power of 4 W. After 2.5 minutes of operation in the HiPIMS mode, a bias voltage of –150 V was switched on in the frequency mode of 100 kHz, with a duty cycle *D* of 50%. The total processing time was 30 min. The temperature of the samples, the waveforms of the discharge current and voltage were measured continuously during the deposition. The temperature of each sample was measured by a dedicated W-Re thermocouple.

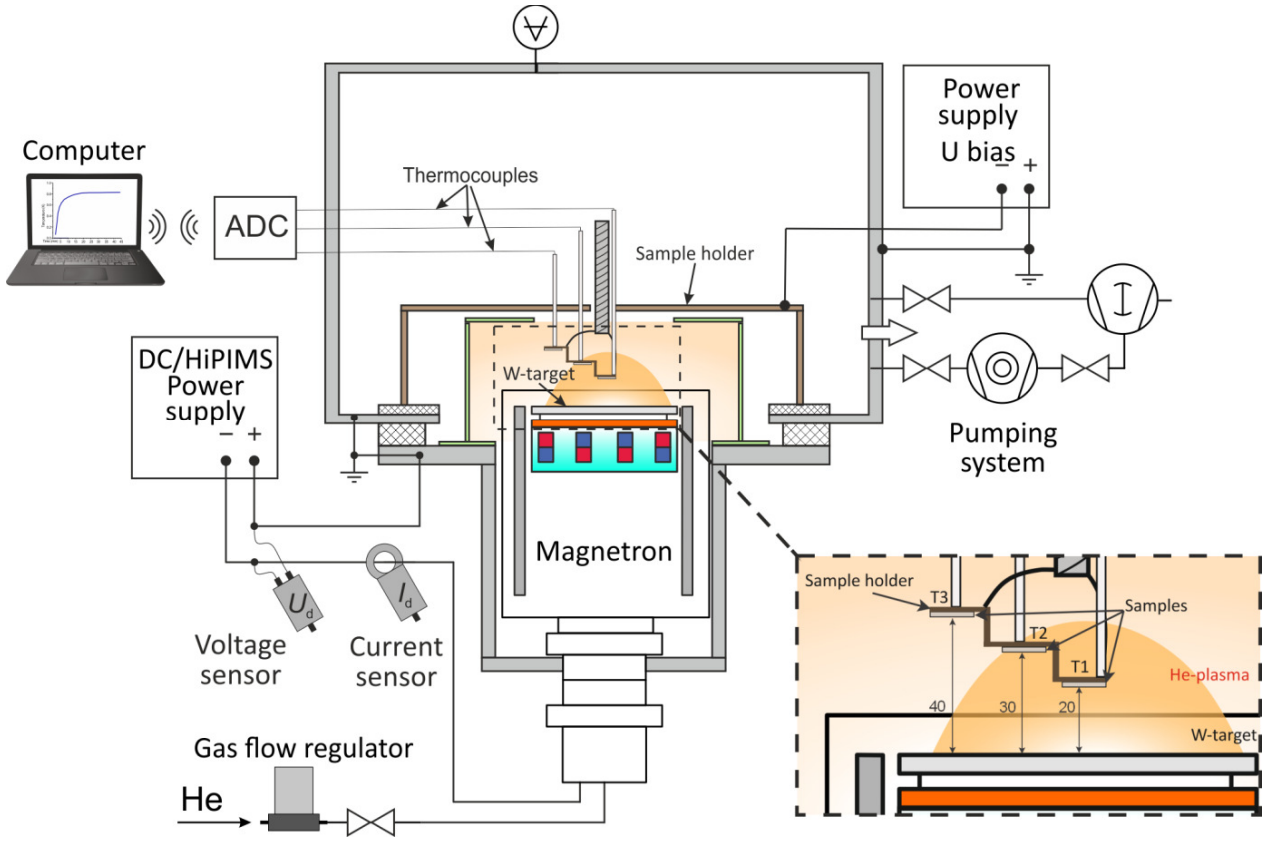

Fig.1. Experimental setup.

After deposition, the samples were cooled in helium flow for 10 minutes. Then the pumping was stopped, helium was introduced into the chamber up to 1 Torr pressure, and the samples were left for cooling for another 50 minutes.

SEM images were taken to determine the coating thickness and surface structure. The coating thickness was estimated on the fracture zone of the sample.

3. Results and discussion

The temporal evolution of temperature for each sample is shown in Fig.2. Discharge current and voltage waveforms taken at the beginning and at the end of the deposition process with and without the bias voltage are presented in Fig.3.

It can be seen from the temperature measurement graphs (Fig.2) that the temperature of the sample after switching on the discharge in the DC mode reaches the steady state in 2 minutes. During this time, all thermodynamic processes associated with heating of the target and the system of screens are balanced. Despite the fact that the distance between the furthermost samples is nearly 2 cm, their temperature differs by no more than 60 K and reaches a maximum value of 1200 K. When the magnetron is switched to the HiPIMS mode, the temperature of the samples slightly decreases by no more than 7 K. This effect can be due to an error of the algorithm for calculating the average power in the pulsed mode, since the current signal has a complex shape (Fig.3). On the other hand, it can also be explained by changing ion flux values on the samples, which might introduce deviations in thermocouple readings. The effect of ion fluxes on thermocouple readings becomes particularly noticeable after applying a bias potential. The temperature value returns to its initial level (before the bias was turned on) when the bias voltage is turned off.

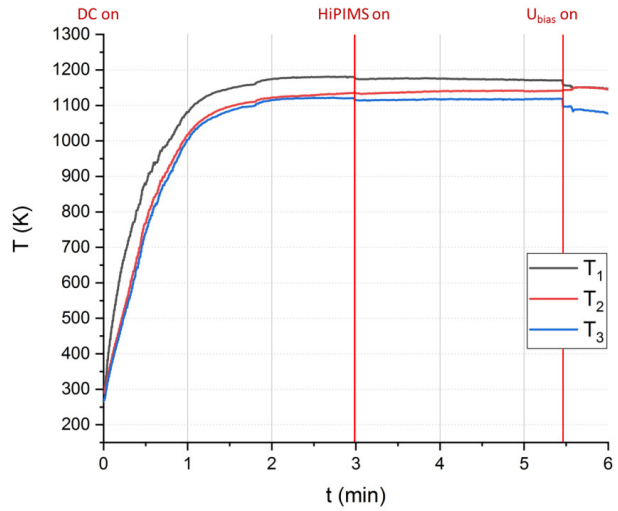

Fig.2. Sample temperature evolution during the deposition process.

Immediately after switching from DC to HiPIMS mode, the discharge pulsed current signal has a smooth shape, unless the bias voltage is applied. In this case, the peak current reaches 40 A and decreases to 10 A by the end of the pulse (see Fig.3a @ 5 min). However, by the end of the experiment, the pulse shape changes—the peak current reaches 100 A and an additional peak of ~40 A appears (see Fig.3a @ 27 min). When bias voltage is applied, the signal shape also changes – an additional peak appears immediately (see Fig.3b @ 6 min). By the end of the experiment, we observe additional current fluctuations with characteristic frequency ~10 kHz (see Fig.3b @ 27 min), while the peak current increases to 120 A. The reason for the occurrence of such fluctuations is the subject of further research. However, it should be noted that the change in the peak current during the experiment occurs gradually, i.e. it is possible that the increase will continue, within a longer deposition process.

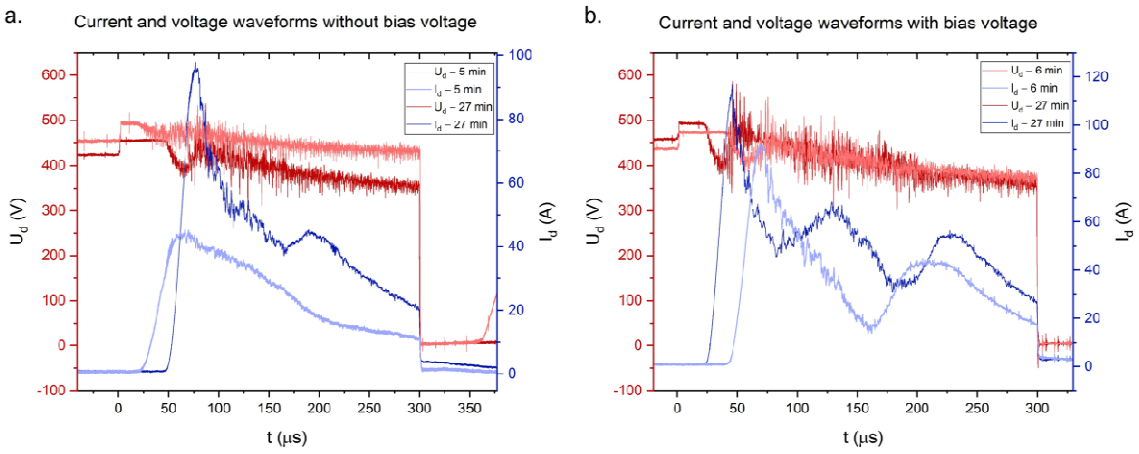

Fig.3. Current and voltage waveforms taken at the beginning and at the end of the deposition process: a) without bias voltage, b) with bias voltage switched on.

The images of deposited layers taken by SEM are shown in Fig.4. Since the temperature of the samples was nearly the same, the resulting surface structure is almost identical. However, a significant difference is observed in the coating thickness – almost 3 times between the furthermost samples (2.9 μm vs. 1.1 μm). Hence, the deposition rate of tungsten fuzz in a magnetron system with a hot target and in the HiPIMS mode can be as high as ~100 nm/min. As the distance increases

by a factor of 2, the deposition rate drops to 35 nm/min. These values differ from those given in articles [6, 7] by one and a half orders of magnitude.

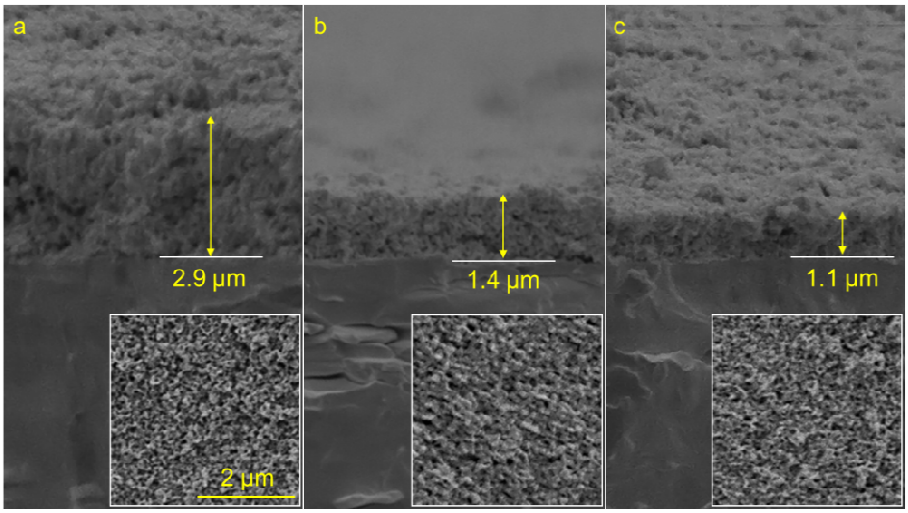

Fig.4. Thickness of a structured tungsten coating deposited in the HiPIMS mode with a bias voltage at a distance from the target, a) 20 mm, b) 30 mm, c) 40 mm.

4. Conclusion

In this work, a structured tungsten coating was deposited in a magnetron system with a hot target in the HiPIMS mode with a frequency of 1 kHz and a pulse time of 300 μs. The rate of coating formation was 35–100 nm/min, depending on the distance from the sample to the target. The maximum temperature difference between the samples at distances of 20 and 40 mm from the target was no more than 60 K. During deposition in HiPIMS mode, the pulse shape changed with time – additional oscillations appeared on the waveforms and the peak current increased. The minimum value of the peak current was observed at the beginning of the experiment (40 A), while the maximum value reached 120 A near the end of the experiment.

Acknowledgements

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5. References

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