



Electric and magnetic fields synergistically enhancing high power impulse magnetron sputtering deposition of vanadium coatings



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ABSTRACT

The high power impulse magnetron plasmas (HIPIMS) were employed as a novel deposition technique, which had the benefit of providing a high ionization degree. In contrast, due to the ion return effect, the deposition rate was relatively low compared to the conventional direct current. In this paper, the external electric field and external magnetic field enhanced simultaneously the HIPIMS ((E-MF)-HIPIMS) to achieve higher deposition rate of the coatings. The vanadium coatings were deposited by the (E-MF)-HIPIMS at equivalent average target powers. The substrate peak ion current density measured in the (E-MF)-HIPIMS mode was increased by a factor of 4 to the discovered current density for the HIPIMS conditions when the anode voltage was set to 70 V. The enhanced ion flux bombardment from the highly ionized (E-MF)-HIPIMS plasma led to the formation of a smoother surface and a denser structure. At the same average target power for the vanadium coatings depositions, the (E-MF)-HIPIMS exhibited higher deposition rates compared to the conventional HIPIMS by approximately 73%. The (E-MF)-HIPIMS sputtered vanadium coating exhibited significantly higher-sized film and substrate interface bonding strengths than compared to the HIPIMS by the Rockwell adhesion testing. In addition, the (E-MF)-HIPIMS sputtered vanadium coating exhibited an improved friction coefficient and improved corrosion resistance compared to the substrate and the HIPIMS sample. The improvement occurred due to the enhanced particle ionization and intense ion bombardment.

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1. Introduction

As a physical vapor deposition (PVD) technique, the magnetron sputtering has been utilized in the deposition of a high number of different coatings. In contrast, a relatively lower plasma density (10^{14} – 10^{16} m⁻³) and a degree of ion bombardment in the substrate region are the disadvantages of this technique [1]. The high power impulse magnetron sputtering (HIPIMS) is a novel ionized physical vapor deposition (I-PVD) technique, which was first proposed by Kouznetsov in 1999 [2]. A high degree of ionization fraction of the sputtered species was produced by a pulsed high peak target power density (0.5–10 kW cm⁻²) with significantly short pulse durations (20–200 μs) and low duty cycles (0.5–5%) [3]. In the PVD process,

the ion bombardment to the substrate is a critical parameter which strongly affects the structure and properties of the growing coatings [4]. Compared to the conventional direct current magnetron sputtering (DCMS), the HIPIMS sputtered coatings have the advantages of a dense texture [5], high smoothness [6], high bonding strength of the film-substrate interface [7] and good thickness uniformity on the surface of the complex shaped workpieces [8]. In spite of the corresponding superiority, one defect of the HIPIMS is that the deposition rate is lower than the DCMS deposition rate. The reason is that the ions could be attracted backwards to the target cathode and captured by the negative potential on the cathode [9–13].

Based on the lower rate in the HIPIMS, researchers utilized other PVD and HIPIMS techniques for hybrid HIPIMS formation. The hybrid HIPIMS with PVD technology included the hybrid direct current magnetron sputtering (DCMS) enhancing HIPIMS [14,15], the hybrid radio frequency magnetron sputtering (RFMS) enhancing HIPIMS [16], the hybrid medium frequency magnetron sputtering (MFMS) enhancing HIPIMS [17] and the hybrid Plasma-

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Based Ion Implantation and Deposition (PBII&D) enhancing HIPIMS [18]. Although the aforementioned hybrid HIPIMS improved the HIPIMS discharge stability or deposition rate, the ionization rate and plasma density both decreased to a certain extent. The hybrid HIPIMS with auxiliary equipment or device usually included the inductively coupled plasma (ICP) addition superimposed on the HIPIMS [19], the electron cyclotron wave resonance (ECWR) addition superimposed on the HIPIMS [20], the auxiliary anode addition superimposed on the HIPIMS [21] and the external electrical-magnetic field addition superimposed on the HIPIMS [22]. Both ICP-HIPIMS and ECWR-HIPIMS enhanced the HIPIMS deposition rate to a certain extent. In contrast, regarding the auxiliary devices, the latter have proven to be complex and expensive and associated with high energy consumption. Therefore, these devices are widely restricted in industrial processes.

In authors' previous study, the electric field and electric potential distribution in the vacuum chamber was adjusted by an auxiliary anode, which formed electric field enhancing HIPIMS (EF-HIPIMS) [21]. The magnetic field distribution in front of the magnetron cathode was optimized by a coaxial electro-magnetic coil, which formed magnetic field enhancing HIPIMS (MF-HIPIMS) [22]. In addition, an auxiliary anode was placed in the vacuum chamber and, also, an external electro-magnetic coil was installed on the HIPIMS target outside the vacuum chamber, which simultaneously enhanced the HIPIMS ((E-MF)-HIPIMS) [23]. The higher electron utilization efficiency and particle ionization rate were achieved. In the present investigation, the results on the effects of the anode voltage on the microstructure, surface micrographs, deposition rate, adhesion properties, tribological properties and corrosion behavior of the vanadium coatings deposited by (E-MF)-HIPIMS were reported.

2. Experimental procedure

2.1. Coating deposition

Fig. 1 presents the schematic diagram of the (E-MF)-HIPIMS equipment. All experiments were conducted in a stainless steel vacuum chamber with a diameter of 400 mm and a height of 400 mm. A metal planar vanadium target (50 mm in diameter, 6 mm in thickness, and 99.99% pure) was mounted on an unbalanced magnetron cathode and powered by a hybrid HIPIMS power supply developed in the authors' laboratory [24]. The working gas was argon (99.9997% pure), which was controlled by a standard mass-flow controller. The argon flow rate was retained constant at 30 ml min^{-1} . The auxiliary anode was a cubic stainless plate

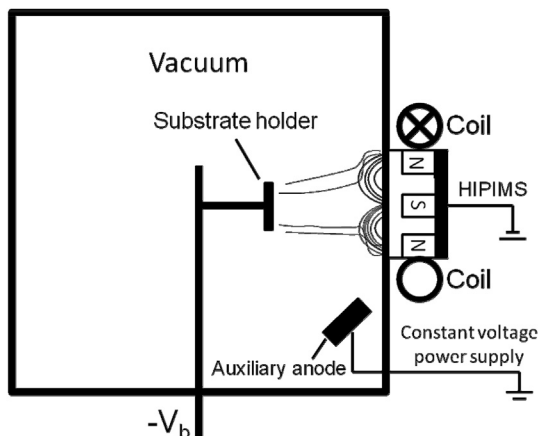


Fig. 1. Schematic diagram of (E-MF)-HIPIMS device.

(100 mm × 100 mm × 5 mm), which was located at 200 mm height in the vacuum chamber. The power of the auxiliary anode was supplied by a Zhaoxin Direct Current with a constant voltage mode, whereas the voltage could be adjusted in the range of 0–110 V. An AvaSpec-3648 optical emission spectrometer was utilized to monitor the discharges during HIPIMS and (E-MF)-HIPIMS.

The electro-magnetic coils were installed outside the chamber wall, which was supplied by a Zhaoxin direct current, whereas the current could be adjusted in the range of 0–6 A. The substrate was biased by a pulsed power supply. The chamber was evacuated to a base pressure of 5×10^{-3} Pa. The mirror-polished 1060 aluminum alloy and the (100) Si wafers were utilized as the substrates, which were installed 100 mm further from the vanadium target. The substrate was heated to approximately 150 °C, and consequently the power was turned off. Prior to loading into the vacuum chamber, the substrates were ultrasonically cleaned in ethanol and acetone for 30 min.

The experiments were performed in three stages: (1) Argon plasma etching, (2) Cu layer deposition and (3) Vanadium coatings deposition, as presented in Table 1.

2.2. Coating characterization

All signals were recorded by a digitizing oscilloscope Tektronix TDS1012B-SC. The crystal structure and preferred growth orientation of the coatings were studied by X-ray diffraction (XRD, Bruker, D8) with Cu K α ($\lambda = 0.15406 \text{ nm}$) radiation. The nano-scale surface morphology was characterized by atomic force microscopy (AFM, Bruker, AXS Dimension Icon) and scanning electron microscopy (SEM, FEI, Helios NanoLab 600i). The microstructure and thickness of the coatings were examined by scanning electron microscopy (SEM, FEI, Helios NanoLab 600i). The indents were produced by a standard Rockwell-C hardness tester with a maximum applied force of 1471 N causing the layer damage adjacent to the boundary of the indentation. The friction coefficients were evaluated on a custom-made ball-on-disc wear apparatus at the rotation speed of 50 rpm and the loading of 50 g with a 6 mm diameter GCr15 ball. The indentation morphology and wear track morphology were observed by optical microscopy (OM, KEYENCE, VHX-1000 E). The corrosion behaviors of the coatings were studied by potentiodynamic polarization tests (CHI604C). The Tafel polarization curves were obtained within a 3.5 wt% NaCl solution. The surface corrosion was achieved, whereas the surface morphology analysis was conducted by scanning electron microscopy.

3. Results and discussion

3.1. (E-MF)-HIPIMS process characteristics

Fig. 2 presents the substrate ion current waveforms of the vanadium coatings prepared by both HIPIMS and (E-MF)-HIPIMS processes. It could be observed that the substrate ion current density in the (E-MF)-HIPIMS mode were all higher compared to the HIPIMS mode at the equivalent average target powers. Regarding the (E-MF)-HIPIMS mode, the substrate peak ion current density gradually increased when the anode voltage was increased from 0 to 70 V. When the anode voltage was 70 V, the substrate peak ion current density reached 128.5 mA cm^{-2} . Through comparison, the substrate peak ion current density was lower in the HIPIMS, with a minimum current of only 33.2 mA cm^{-2} . The substrate peak ion current density measured in the (E-MF)-HIPIMS mode was increased by a factor of 4 compared to the substrate peak ion current density in the HIPIMS when the anode voltage was set to 70 V. The external electric field generated by the auxiliary anode and the magnetic field induced by the electro-magnetic coils, both

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