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The Effect of Pulse DC and DC Substrate Bias during In Situ Cleaning PVD Process on Surface Roughness

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Abstract

Surface morphology modification during in situ cleaning of physical vapor deposition (PVD) process is essential to strengthen and prevent unexpected adhesion failure during machining. Applying pulse direct current (PDC) on substrate bias is still uncommon compared to a conventional direct current (DC). This paper is to compare the effects of DC and PDC applied at substrate bias, to the surface roughness and homogeneity. Tungsten carbide (WC) cutting tool insert and argon were used as substrate and inert gas, respectively. The runs were conducted to compare the bias at DC (-500V) and PDC (-200V, -500V, -800V). The surface roughness and homogeneity were inspected using atomic forced microscopy (AFM) and Minitab version 16 exploited to analyze the data. The wettability based on water drop static contact angle on the substrate surface was measured by a digital 800k USB 2.0 CCD DCAM and VIS ver7 (Professional Edition) software. PDC on the substrate bias produced finer grain structures compared to DC. Further increase in PDC voltage resulted in homogenous surface with finer and more globular microstructure with higher surface energy. Hence PDC at optimum level provides better surface readiness prior to coating compared to DC and proven to be a critical factor for further enhancement of coating adhesion.

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Keywords: *Physical Vapor Deposition, Tungsten Carbide, Pulse Direct Current, Substrate Bias, Surface Roughness.*

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1. Introduction

The introduction of negative bias potential to a conductive substrate creates plasma sheath's environment which caused the positive inert gas ions to accelerate and hit the substrate surface. Ions with enough energy will sputter and expel out surface atoms. The application of pulse DC technique was first conducted in the nineties for depositing process [1]. Since then, focus on having PDC substrate bias during depositing process is ongoing study. Application of PDC improves on adhesion as well as hardness, surface structure, coefficient of friction and wear rate of coating materials [2-6]. In addition, PDC during in situ cleaning has significant advantages in eliminating arcing problems which then leads to a better process flexibility and stability [7-8]. Furthermore, it enhances plasma density and mobility which is important to create more bombardments to the surface [9-10] and improves coating quality [11]. Moreover, PDC bias allows better control on surface roughness depth compared to only DC biasing [12]. Finally, process temperature can be reduced and become more stable [13]. In situ PDC technique using medium frequency range of 10 to 500 kHz was commonly preferred [14-16].

During in situ process, the positive inert gas ions acceleration will increase with the raise of negative bias potential of the substrate. However, at higher potential, the tendency of arcing to occur is very high and causing much unexpected

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drawbacks to coating adhesion [17-18] later on. Moreover, it will also cause problem to the subsequent substrates because arcing will contaminate the chamber of the machine. The adhesion quality will be degraded further until proper cleaning and maintenance is carried out [19]. Based on the available results and findings from previous works, the arcing issue can be solved by having negative PDC bias instead of only DC bias applied to the substrate. However, there were still less researches and papers that have been published focused on PDC at in situ compared to the conventional method of DC bias [20-23].

Focused of this research was to compare the effects on the substrate surface roughness and homogeneity at different levels of substrate biasing during the PVD in situ cleaning.

2. Experimental Procedures

2.1. In Situ Cleaning Techniques

Experiments were carried out using VTC PVD 1000 unbalanced magnetron sputtering system from VACTEC Korea. During in situ cleaning process, the chamber was vacuum pumped to 5.0 x 10^{-5}mbar and process temperature set at 400ºC. Then, pure Argon (99.999%) of 50sccm was pumped into the chamber through ion gun outlet until the process pressure dropped to 4.0 x 10^{-3} mbar. Substrate of tungsten carbide (WC) cutting tool insert (SPGN120308 / SPG422 / TAP04615 / S120 G10E) by Sumitomo was mounted onto a planetary holder which rotated at 2rpm. Prior to in situ cleaning, the substrate was soaked into ultrasonic bath of ethanol (purum ~96% based on denaturant – free substance) using JAC 1505 ultrasonic machine at 42º for 30 minutes. Then it was dried off using a hand dryer. The substrate was immediately inserted into PVD chamber for in situ pretreatment to prevent contamination or oxidation. The in situ parameter settings of sputtering system tabulated in Table 1.

Table 1. Substrate Bias Parameters During In Situ Cleaning

Experiment	Substrate Bias
1	untreated
2	PDC -200V
3	PDC -500V
4	PDC -800V
5	DC -500V

2.2. Surface Roughness Characterizations

Post in situ cleaning substrate's surfaces were inspected using non-contact mode atomic force microscopy (AFM) of XE-100 model from Park System. Topography's scanning area was set to 5 μm^2 at a scan rate and selected frequency of 0.5 μm/s and 261.86 kHz, respectively. Data interactions were analyzed using one-way ANOVA [24] with the help of Minitab version (16) software. Static contact angle of water drop was measured using a digital 800k USB 2.0 CCD DCAM and VIS ver7 (Professional Edition) software which allows auto calculation of the angles.

3. Results and Discussion

3.1. Surface Roughness difference PDC Biases using AFM

Substrate's surface topographies in three-dimension at different substrate biases are shown in Figure 1. Evidence of sputtering process did take place during in situ cleaning is clearly illustrated in the comparison between Figure 1(a) for untreated sample with Figure 1(b-d) for treated samples. Negative potential on substrate bias helps to elevate the speed of positive argon ions, Ar^+ towards it and enhance the sputtering process. In addition, WC surface grain structures gradually improved from coarse spikes with peaks and valleys to finer globular structures as the PDC bias voltage increased (Figure 1). Furthermore, the substrate's surfaces of higher PDC bias became more homogenous compared to at the substrate of lower PDC bias voltage. Moreover, this phenomenon indicates an important characteristic of plasma sputtering effect on substrate surface [25].

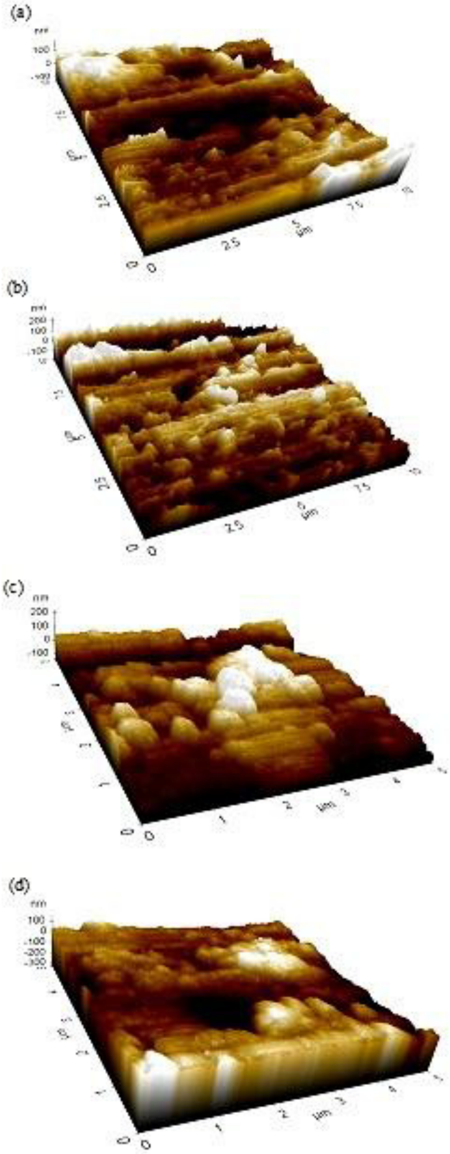

Fig. 1. 3-dimensional surface roughness images taken by AFM, (a) 0V, (b) PDC -200V, (c) PDC -500V and (d) PDC -800V.

Finer globular and highly homogenous grain structures lead to a wider surface area and as consequences provides a higher surface energy. The characteristics allow interactions via formation of Van der Waals bonding between two materials. Hence, it is an important criterion for better adhesion between substrate and coating materials [26]. It is evident from water drop contact angles at different levels of in situ cleaning depicted in Fig. 2.

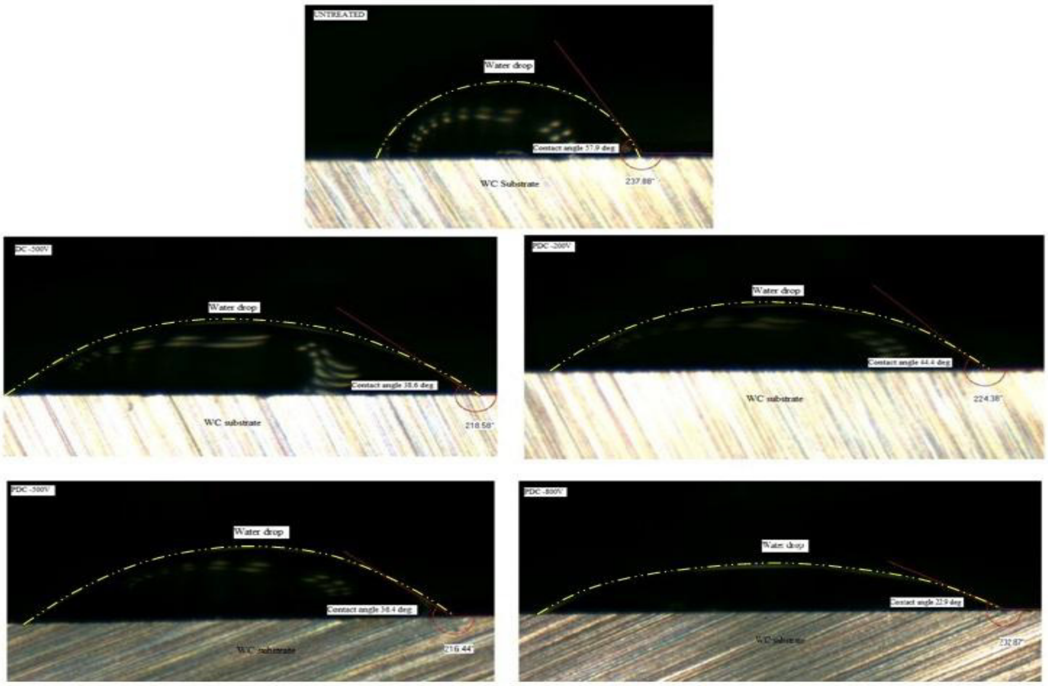

Fig. 2. Water drop wetting ability on WC substrate at different substrate biasing voltage levels of in situ cleaning (Untreated substrate surface, substrate bias of PDC -200V, PDC -500V, PDC -800V and DC -500V).

Table 2: Contact Angles between Water Drop and WC Substrate Surface

In situ substrate bias	Contact angle
Untreated	57.9 º
DC -500V	38.6 º
PDC -200V	44.4 º
PDC -500V	36.4 º
PDC -800V	22.9 º

The contact angles are summarized in Table 2. Surface energy trends show similar pattern as the surface topology result. Thus, at higher PDC biasing the effectiveness of plasma sputtering increased. It is a critical indicator in order to determine optimum parameter of in situ cleaning process.

3.2. Surface Roughness Comparisons Between PDC and DC -500V Biases

Figure 3, illustrates surface topography after in situ cleaning process at DC of -500V. Surface roughness of DC sample was slightly coarser compared to PDC as in Figure 1(c) indicates pulse voltage improved surface structure. For instance, observed arcing during in situ cleaning of DC might generate more irregular structures which put materials prone to severe adhesion defect. This argument is in good agreement with conclusion made by Mitterer et al.1997 [27] and Sarakinos et al. 2010 [18].

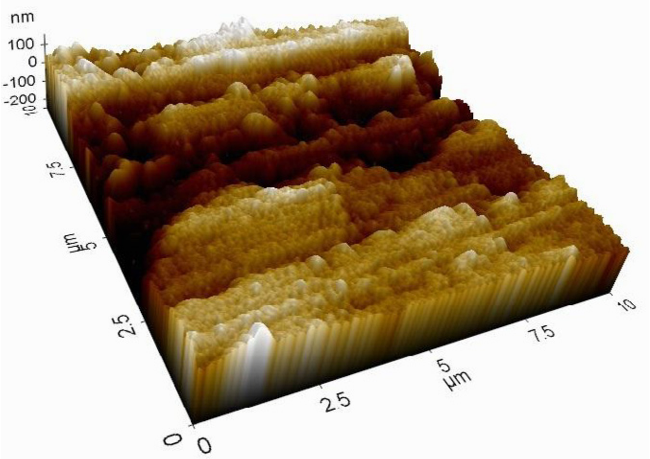

Fig. 3. 3-dimensional surface roughness images taken by AFM at DC -500V

3.3. Interaction between substrate bias and surface roughness

According to Tanoue et al. 2009 [12], pulse substrate bias voltage potential leads to more bombardments of ions without excessive modification if compared to DC. However, based on one-way anova boxplot shown in Figure 4, there is huge improvement obtained from PDC -200V to PDC -500V compared to PDC -500V to PDC -800V. Moreover, variance of surface roughness slightly increased from PDC -500V to PDC -800V. These phenomena reflected optimum limits in application of PDC substrate bias. Whereby, beyond this threshold it might lead to excessive bombardments of ions. In contrast, application of DC beyond this threshold may result in worst variance conditions.

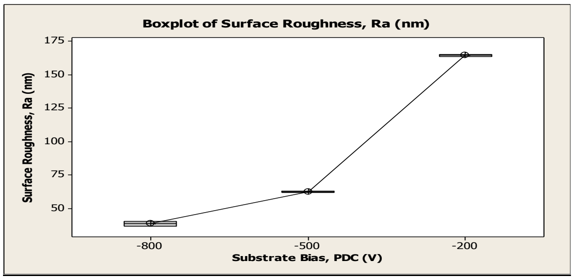

Fig. 4. One-way ANOVA: Surface Roughness, Ra (nm) versus Substrate Bias, PDC (V).

Response function between PDC bias and surface roughness tabulated in Table 3 indicates a high confident level with the value of R-sq more than 99% and zero percentage of error, P.

Table 3. Anova Table for Response Function Of Surface Roughness

Source	Degree of freedom	Sum of Squares	Mean square	F-value	P
Substrate bias PDC	2	17913.91	8956.95	2639.35	0.000
Error	3	10.18	3.39		
Total	5	17924.09			

Figure 5 depicts comparison of one-way ANOVA analysis between PDC and DC at -500V which shows large differences in surface roughness. The high variance of DC is might be due to arcing and uncontrollable sputtering which tend to produce more irregular surface structures. Furthermore, it shows similar trend with results obtained by Tanoue et al. 2009 [12].

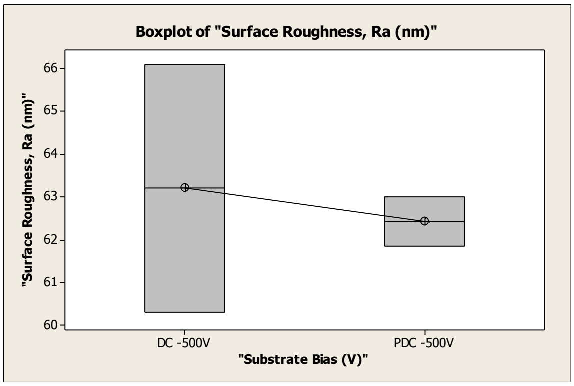

Fig. 5. One-way ANOVA: Surface Roughness, Ra (nm) versus Substrate Bias (V).

Substrate biasing during in situ cleaning of PVD process has significant effect on substrate's surface morphology. It was clearly observed from differences between untreated samples and those with bias substrate. Its surface was gradually changed from spikes to more globular structures. In addition, further increase in bias voltage for PDC resulted in finer and homogenous surfaces which brought to enhancement in substrate's surface energy. It is an important criterion to ensure high adhesion between coating and substrate materials.

Analysis done on surface roughness showed huge improvement trend between low and high bias voltages, for instances, PDC -200V and -500V. But, it is less effective at higher voltage PDC -800V due to observed reduction homogeneity. However, PDC showed more advantages if compared to conventional DC biasing, it is not only capable in generating finer surface structure but able to reduce the surface roughness variances. This finding is important as an alternative way in achieving higher adhesion strength besides other well known methods.

Further fine tuning of the pulse direct current application at substrate bias during in situ cleaning should be conducted in order to find the optimum voltage potentials, pulse frequency, and temperature for other substrate materials selection.

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