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Correlation of the Debye sheath thickness and (Cr,Al)N coating properties for HPPMS, dcMS, CAE and PCAE processes

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ABSTRACT

Physical vapor deposited (PVD) coatings are widely-used in tool and component applications, where the predominant aims are the enhancement of the lifetime and an improvement of the economic efficiency. The achievement of these aims amongst others strongly depends on a homogeneous distribution of the coating properties with respect to the entire functional surface, in particular on complex-shaped tools. In order to obtain a sufficient coating homogeneity, pulsed high power plasma processes such as the high power pulsed magnetron sputtering (HPPMS) or the pulsed cathodic arc evaporation (PCAE) has to be chosen. In order to characterize the influence of both technologies on the coating homogeneity, the Debye sheath thickness s_D at the substrate side was determined, which is expected to strongly correlate with the coating homogeneity on complex surfaces, since it is a measure for the shielding of charged coating atoms. Measurements with varying pulse parameters were performed. In addition, as reference a direct current magnetron sputtering (dcMS) and cathodic arc evaporation (CAE) process were conducted, respectively. As it is widely used as protective coating for tools in many applications, the coating system (Cr,Al)N was chosen. In the first step, the Debye sheath thickness was determined by means of Langmuir probe. A Debye sheath thickness from s_D = 430 µm to s_D = 48 µm was found for the different processes and parameters. In the second step, the (Cr,Al)N coatings were deposited with selected process parameters on structured tungsten carbide substrates. The coating morphology, coating thickness, surface roughness and chemical composition were observed to be more homogeneous for processes with a lower Debye sheath thickness. The best results were obtained for the HPPMS and PCAE technology. In summary, processes and parameters were identified which can be used for the coating of complex structured tools.

1. Introduction

Physical vapor deposited (PVD) coatings are widely used in tool and component applications [1,2]. Predominant aims of the PVD coating application are the enhancement of the lifetime and an improvement of the economic efficiency [3]. The achievement of these aims amongst others strongly depends on a homogeneous distribution of the coating properties with respect to the entire functional surface, in particular on complex shaped tools [4]. In many cases, in PVD processes this homogeneity is hard to achieve due to the line-of-sight characteristics [5]. Besides the commonly used rotation of the substrates or extensive mounting concepts, another possibility to overcome this issue is the choice of pulsed high power plasma processes such as the high power pulsed magnetron sputtering (HPPMS) or the pulsed cathodic arc evaporation (PCAE) [6]. The HPPMS is known as an advancement of the direct current (dc) and middle frequency (mf) magnetron sputtering (MS) [7–9]. The PCAE technology represents an improvement of the cathodic arc evaporation (CAE) technology, where especially the amount of the so-called droplets is significantly decreased [10–12]. The advantages as well as an extensive comparison of both technologies are described in a review by Anders [13].

According to this, magnetron sputtering processes, especially the HPPMS, exhibit in the absence of arcs homogeneous coating properties on plane surfaces. For CAE and PCAE processes without filtering concepts, the homogeneity of the coating properties on plane surfaces, for example the roughness values, is reduced due to the contamination with droplets [13]. Regarding complex structured surfaces, further investigations are necessary to determine the influence of both technologies on the homogeneity of the coating properties, since these are less reported compared to the influence of both technologies on the coating properties regarding plane surfaces. In order to characterize the influence of the different technologies on the coating homogeneity, the related plasma properties, especially the Debye sheath thickness s_D, can be used [14,15]. Due to the positive charge in this area, the transport of positive charged ions from the target to the substrate surface is strongly influenced. If the Debye sheath thickness is too large, compared to the

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width and depth of complex structures, the plasma cannot penetrate the structure and the substrate surface is shielded from arriving film-forming ions [14,15]. Therefore, it can be expected that a decreased Debye sheath thickness leads to an increased homogeneity of the coating, especially on complex surfaces with sharp edges, since the shielding of the structures is reduced, when the Debye sheath is thin and can adjust itself homogeneously to the surface [14,15].

Hence, in the present work measurements on the Debye sheath thickness s_D as well as on the coating properties with respect to pulsed high power plasma processes HPPMS and PCEA with varying process parameters pulse-on-time t_{on} and frequency f were carried out. Additionally, the investigations were carried out as reference with a dcMS and a CAE process, respectively. In order to determine the Debye sheath thickness and the correlating homogeneity of coatings, the ternary coating system Cr-Al-N was chosen. Due to its flexibility regarding the adjustment of the coating properties, it is widely used as protective coating for tools [16,17]. (Cr,Al)N has been subject of several investigations regarding the plasma and coating properties [18–23]. In a first step, the plasma was analyzed using a Langmuir probe system. From the results of the measured U-I curves the Debye sheath thickness s_D was determined. An overview regarding the capability of Langmuir probe measurements can be found in the review of Britun et al. [24]. In the second step, (Cr,Al)N coatings were deposited on structured substrates with selected process parameters to determine the coating homogeneity. The coatings were analyzed with respect to the morphology and coating thickness s by means of scanning electron microscopy (SEM), the arithmetic mean roughness Ra using confocal laser scanning microscopy (CLSM) and the chemical composition by means of energy dispersive X-ray spectroscopy (EDS).

2. Materials and methods

2.1. Coating unit configuration and process parameters

The investigations on the dcMS and HPPMS processes were carried out using an industrial scale PVD coating unit, CC800/9, CemeCon AG, Würselen, Germany. The vacuum chamber has a dimension of 1000 mm × 1000 mm × 1000 mm (Fig. 1a–b). Two cathodes, one equipped with a dcMS power supply and one with a HPPMS power supply are available. Both cathodes were equipped with a rectangular chromium target with 20 aluminum plugs with a purity of 99.9% for chromium and 99.5% for aluminum. The target size was 500 mm × 88 mm. For the substrate-oriented plasma diagnostics by means of Langmuir probe, the cathodes were mounted parallel to the chamber wall. The Langmuir probe was attached to a flange on the opposite door as shown in Fig. 1a. For the coating deposition, the cathodes were mounted parallel to the movement axis of the substrate table, on which the substrates were attached as shown in Fig. 1b. The distance between the cathodes and the Langmuir probe as well as the substrates was x = 60 mm. To obtain comparable results from the plasma diagnostics and the coating deposition, the probe and the substrates were both positioned in front of the target racetrack.

The investigations on the CAE and PCAE processes were conducted using a coating unit Metaplas Ionon PVD 20″, Oerlikon Balzers Coating Germany GmbH, Bingen, Germany. The cylindrical vacuum chamber has a cross-section dimension d = 460 mm and a height h = 530 mm (Fig. 1c–d). The coating unit was equipped with a circular cathode, which was either connected to a dc or a pulsed power supply, aixcon PowerSystems, Stolberg, Germany. A powder-metallurgically manufactured target with a diameter d = 63 mm and a chemical composition x(Cr) = 50 at.% and x(Al) = 50 at.% was mounted on the cathode. The differing chemical composition, compared to the magnetron sputtering processes, was necessary, since a comparable target could not be manufactured for the CAE/PCAE cathode. For the substrate-oriented plasma diagnostics, the Langmuir probe was attached to a flange on the opposite side of the coating unit as shown in Fig. 1c. For the coating deposition, the substrates were attached to the substrate table and positioned in front of the cathode as shown in Fig. 1d. The distance between the cathodes and the Langmuir probe as well as the substrates was x = 230 mm. To obtain comparable results from the plasma diagnostics and the coating deposition, the probe and the substrates were both aligned to the center of the target.

To avoid damaging the Langmuir probe, for all processes the substrate heating was switched off. Furthermore, the substrate table rotation in both coating units was switched off to obtain comparable results from the plasma diagnostics and the deposited coatings.

For the dcMS and HPPMS (Cr,Al)N processes Ar was used as process gas and N_2 was used as reactive gas, respectively. The Ar flow was j_{Ar} = 200 sccm. The N_2 flow was regulated using a pressure control mode to achieve p = 430 mPa for the dcMS and p = 450 mPa for the HPPMS processes. Both pressure values were chosen with respect to the operating point of each process. Hence, the nitrogen flow was approximately j_{N2} = (40 ± 10) sccm. For the dcMS and HPPMS processes the cathode was powered with an average power P = 1 kW, respectively. This value was chosen, because for higher average powers a measurement by means of Langmuir probe was not possible. For the plasma diagnostics of the HPPMS processes the pulse-on-time t_{on} was varied between t_{on} = 40 µs and t_{on} = 200 µs at a constant frequency f = 500 Hz. Hence, the correlating peak power was changing from P_p = 89 kW to P_p = 23 kW. The frequency was varied from f = 500 Hz and f = 2000 Hz at a constant pulse-on-time t_{on} = 40 µs. Thus, the peak power was changing from P_p = 89 kW to P_p = 32 kW. With respect to the determined Debye sheath thicknesses which are presented in Section 3.1, (Cr,Al)N coatings were deposited using the dcMS process and HPPMS processes with t_{on} = 40 µs and f = 2000 Hz as well as t_{on} = 200 µs and f = 500 Hz. A bias voltage U_B = − 50 V was applied. The coating time was adapted to a coating thickness of s ≈ 3 µm. Furthermore, to increase the adhesion between the substrates and the coatings, a (Cr,Al) interlayer with s ≈ 100 nm was deposited using Ar as process gas. The dcMS and HPPMS process parameters are presented in Table 1.

Since for reactive CAE and PCAE (Cr,Al)N processes no process gas like Ar is required, only pure N_2 was used as reactive gas. Hence, there was an unavoidable difference to the magnetron sputtering processes, since for these processes pure N_2 as process gas would result in a significant target poisoning and is therefore not reasonable. However, the different process gas should be concerned during the evaluation of the results. The N_2 flow was adapted to achieve a process gas pressure p = 2000 mPa for both process types. For the CAE process, the cathode was powered with a cathode current I = 60 A and a cathode voltage U = (13.0 ± 0.5) V. For the PCAE processes, the cathode current was I = 60 A and the cathode pause current was I_{off} = 40 A. The cathode voltage was U = (13.0 ± 0.5) V. For the plasma diagnostics, the frequency was varied from f = 91 Hz to f = 143 Hz at a constant pulse-on-time t_{on} = 5 ms. Furthermore, the pulse-on-time t_{on} was varied between t_{on} = 5 ms and t_{on} = 9 ms at a constant frequency f = 91 Hz. The investigated process parameters were limited by the pulse unit and the used $Cr_{50}Al_{50}$ target. With respect to the determined Debye sheath thicknesses, which are presented in Section 3.1, (Cr,Al)N coatings were deposited using the CAE process and a PCAE process with t_{on} = 7 ms and t_{off} = 4 ms. The coating time was adapted to a coating thickness of s ≈ 3 µm. A bias voltage U_B = − 50 V was applied, which was comparable to the dcMS and HPPMS coating processes. The CAE and PCAE process parameters are presented in Table 2.

Due to the requirements on the geometry and the simplified pretreatment, the coatings were deposited on tungsten carbide substrates THM12, Ceratizit GmbH, Empfingen, Germany. To reflect complex structures, a cut with a depth of h_c = 1.50 mm and a width of d_c = 0.75 mm was machined, as shown in Fig. 2. Hence, the aspect ratio was AR = 2. Furthermore, the top and the flanks of the substrate were polished to an arithmetic mean roughness Ra ≈ 10 nm. The substrates were positioned in the coating units so that the top of the