



PHYSICAL VAPOUR DEPOSITION ON CORROSION RESISTANCE: A REVIEW

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ABSTRACT

Physical Vapour Deposition (PVD) is one of the versatile surface modification method commonly used in the manufacturing of coatings, powders, fibres, and monolithic components. The applications of PVD are cutting tools, decorative, aerospace industries and biomedical application. The main advantages of PVD coating as compared with other surface modification methods is low processing temperature thus can be used on variety of materials. However PVD coating has limitation due to low corrosion resistance especially when it contact with Cl ion in sea water and body fluids. Hence, this review explains the overview of PVD coating on corrosion resistance in body fluids and sea water. The types of PVD coating and parameter commonly used as well as issue related to PVD coating is discussed in this paper.

Keywords: physical vapor deposition, corrosion resistance, thin solid film, CAPVD, multilayer coating.

INTRODUCTION

In recent years, physical vapour deposition (PVD) is an atomistic deposition process where target material is changed from a solid or liquid source (in the form of atoms or molecules) to vapour in a vacuum or low pressure gaseous (or plasma) environment before it is changed back into a solid form and transferred to the substrate [1]. PVD can produce a hard coating such as TiN, TiCN, TiAlN, and CrN [2]. The advantages of this technique is that it can be used with low processing temperatures (<500 °C) and provide a wide range of coating thickness. The problems with high processing temperatures it that they have a detrimental effect on the physical and mechanical properties of the base material, which restricts the types of substrates that can be used, promote unexpected phase transitions, and create excessive residual stresses due to the difference in thermal expansion between the deposited material and substrate [3].

The processing parameters used to create a PVD coating are important factors for determining the quality of the coating. The common processes and parameters used to create a PVD coating are:

- a) Generation of particles from the target materials
- b) Transport and film growth
- c) Particle energy, density, substrate temperature, and reactive gas properties.

Even PVD has very versatile and many advantages as compared with others surface modification method, it has limitation when it contact to Cl⁻ ion which is appears in sea water and body fluids. Several studies have been conducted to solve this issue. Past reports highlighted that coated PVD layer consists of pores, pin holes and columnar growth which act as channels for the

aggressive medium to attack the substrate. Duplex and multilayer coatings seem able to address this issue at certain extent but at the expense of manufacturing time and cost. This review focuses on the application of physical vapor deposition to improve corrosion resistance in aqueous solution and body fluids. It discussed about the major problem in PVD coating and review the solution used by previous studies. The limitation and the future direction on PVD coating also discussed. This review also provides overview about the types of PVD coating and parameter used in previous studies.

TYPES OF PVD COATING

PVD processes are characterized by high coating density, strong adhesion; multi-component layers, low substrate temperatures, and can use a myriad of coating and substrate materials. The main categories of PVD processing are vacuum deposition (evaporation), sputter deposition, and arc vapour deposition [1].

Vacuum deposition

Vacuum deposition, also called vacuum evaporation, is a PVD process that changes the target material from a solid to a vapour before it reaches the substrate where it is condensed back to a solid state with little or no collisions with gas molecules in the space between the target and substrate material [1]. This process is similar to the process of boiling. The different between of them is in the boiling process, the boiling point is defined by the temperature where the material changes from a liquid to a gas at one atmosphere of pressure. During evaporation, vapour pressure is approximately 0.1-1 Torr depending upon the evaporation rate. In other words, the evaporation temperature is much lower than the boiling point. [1]

Evaporation can occur in vacuum and vapours other than the target material are almost entirely removed before the process begins. The reason this process is



performed in vacuum is because the vacuum reduces gaseous contamination in the deposition system and it prevents foreign particles from reacting with atoms to produce unwanted compounds. When evaporation is conducted at ambient temperatures with reactive gases, compound films can be deposited at higher rates and lower temperatures. For example, TiC and TiN coatings can be deposited by evaporating Ti in the presence of C₂H₂ and N₂ plasma [5].

Sputter deposition

A sputtering process is a non-thermal vaporization process that uses atoms from a target material are ejected from a solid surface by momentum transfer using an atomic-sized energetic bombarding particle, which is usually a gaseous ion, accelerated from plasma. Currently, the sputtering technique is used in the fabrication of commercial coatings and to develop new coatings in the laboratory [2]. The sputtering process can be done in a vacuum or in plasma. In a vacuum, it will be performed by using an energetic ion bombardment of target material and an ion gun or low-pressure plasma. In this case, the sputtering particles have only a few or no gas phase collisions in the space between the target and the substrate. In plasma, the sputtering process can be completed using a higher plasma pressure (5–30 mTorr) where energetic particles are sputtered or reflected from the sputtering target and “thermalized” by gas phase collisions before they reach the substrate surface [3]. Ceramics and refractory metals that are difficult to deposit by evaporation and are more easily deposited using sputtering.

For efficient momentum transfer, the atomic weight of the sputtering gas should be close to the atomic weight of the target, so for sputtering light elements, neon is preferable. For sputtering heavy elements, krypton or xenon is used. Reactive gases can also be used to sputter compounds. A simple DC glow discharge can be used to sputter conductive targets but radio frequency sputtering is preferred for insulating targets [1].

The most influential parameters for magnetron sputter deposition were magnetron power, ion current density, substrate bias, gas pressure, target composition, and substrate temperature (Malvern Ltd). The morphology and elemental analysis were observed using a JEOL SEM/EDX (JSM 6400). These parameters directly affected the mechanical, tribological, structural, and thermo-oxidation properties of coatings [7]. Generally, sputtering PVD coatings can be used for single and multilayer metal conductor films used for microelectronics and semiconductor devices, compound conductor films for semiconductor electrodes, optical coatings, and decorative applications. One advantage of sputtering coatings is that any material can be sputtered and deposited, including elements, alloys, or compounds. Other advantages are that the sputtering target can provide a large-area vaporization source and sputtering conditions can easily be reproduced, from run to run. One of the limitations of sputtering is that it produces non-uniform coatings. Special fixturing, tooling, or source designs must be used to deposit films

with uniform properties and most of the sputtering energy heats the target, which must be cooled. Additionally, sputter vaporization rates are low compared to those achieved by thermal vaporization. Very low ionization rates are major disadvantage of conventional sputtering process as the plasma is tightly confined to the face of the target and low ion current densities are collected on the substrate [8].

Arc vapor deposition

Arc vapour deposition is a PVD technique that uses arcing to strike material from the target that creates a small (usually a few microns wide) highly energetic emitting area known as a cathode spot. Material will vaporize after the arc strikes the target material after which it will condense on a substrate to form a thin film. This technique can be used to deposit metals, ceramics, and composite films. The arc ion plating process has a higher degree of ionization compared to other ion plating methods. As a result, a dense coating can be deposited at lower temperatures using this technique [4]. Typically, CA-PVD has high deposition rate. The good adhesion of the films is due to high metal ion energy. This method is environmentally clean, and flexible [5]. However, this method has limitations such as poor surface morphology due to the formation of macro particles. Micro particles (or micro droplets) are droplets of target materials with low melting points (e.g. aluminium in the case of (Ti,Al) N coating). It is also believed that very fast evaporation during the cathodic arc process produces excess atoms that are not completely ionized before they arrive at the substrate surface [6].

Many researchers have developed ways to reduce the micro droplets using a filtered arc [11-14]. Their results show that the filter successfully reduced the number of micro particles in a coating and they reduced the deposition rate [8]. Instead of using a filtered arc system to reduce micro droplets, other researchers have studied the effect of PVD parameters on micro droplets formation. For instance, a few studies have examined the relationship between the number of micro particles and nitrogen pressure. They reported that due to the formation of a compound on the target surface, the number of micro particles decreased when nitrogen pressure increased [7, 8]. Similar studies were conducted by Akari who looked at the effect of N₂ flow rates on micro droplets formation. He reported that the number of micro droplets decreased when N₂ flow rates increased from 0.2 to 1.2 Pa. By contrast, the number of micro droplet increased with increasing N₂ flow rates of 1.2 to 3.5 Pa [17]. A group of researchers studied the effect of substrate temperature on the number of micro droplets. They found that the number of micro droplets decreased when substrate temperature increased [9]. Shah *et al.* [10] tried to evaluate the effect of substrate temperature on microdroplet on Ti-13Zr-13Nb alloy, they reported that increased substrate temperatures increased the coating thickness of the TiN coatings as well as decreased the number of micro droplets.

In another study, Shah *et al.* [20] investigated the effect of nitrogen gas flow rate on Ti-13Zr-13Nb alloy.



They found that the microdroplet decreased as the nitrogen gas flow rate increased. In general, nitrogen gas flow rates and substrate temperatures had a significant effect on micro droplet formation. Although many of researchers have studied the effect of processing parameters on micro droplet formation, further comprehensive research is needed to produce superior coatings.

Principle arc vapor deposition arc

The arc evaporation process starts by striking a high current, low voltage arc on the target material. The localized temperature at the cathode spot is extremely high (around 15 000 °C), which results in a high velocity (10 km/s) jet of vaporized cathode material, leaving a crater behind on the cathode surface. The cathode spot is only active for a short time before it self-extinguishes and reignites in a new area close to the previous crater. This behaviour causes the arc to appear as if it is moving. Ion bombardment at the cathode and electron bombardment at the anode heat the electrodes. Most of the ejected material is thermally evaporated atoms but some is ejected as molten droplets or solid particles from the cathode. A high percentage of the vaporized atoms are ionized in the arc vaporization process because of the high electron density [1]. The schematic diagram of CAPVD process is provided in Figure-1.

The high power density arc causes a high level of ionization (30-100 %), multiple charged ions, neutral particles, clusters, and macro-particles (droplets). When a reactive gas enters the chamber during the evaporation process, dissociation, ionization, and excitation can occur during interaction with the ion flux, which produces a compound film. The formation of micro particles from the cathode target is a serious problem since the quality of coating is degraded. This problem has been investigated by a number of research groups and quite a few innovative solutions have been put forward as solutions problems such as cooling the cathode, decreasing the arc current, and accelerating the movement of arc spots [16]. Other processing parameters include target composition, substrate temperature, bias voltage, gas flow and an ion bombardment rate is believed influence the formation of microdroplets.

ISSUE IN PVD FOR CORROSION RESISTANCE

Although the PVD coating has many advantages, a hard PVD layer is often not completely solid due to defects in the coatings (such as pores, pinholes, and columnar growth) that may have formed during deposition. Examples of crater and pinhole defects created during Ti₂N coating on NdFeB substrate are shown in Figure-2 these defects may create channels for an aggressive medium to attack the substrate, thus affecting the electrochemical behaviour of the coatings. In order to improve the corrosion properties of implant materials, researchers varied PVD parameters and applied multilayer coating to coated samples.

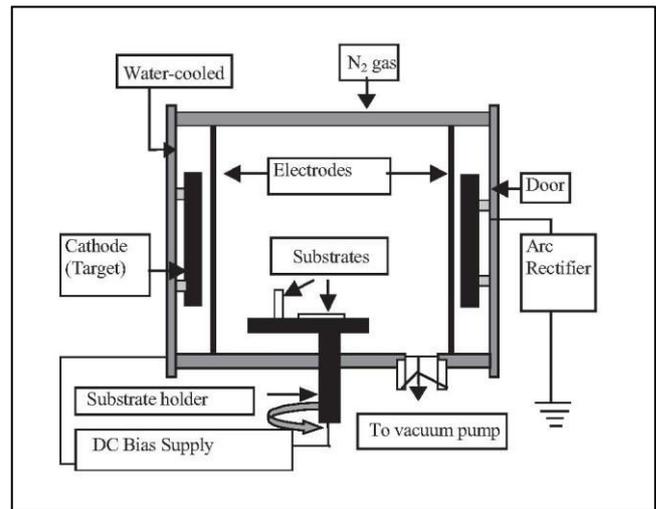


Figure-1. Schematic diagram of CAPVD process [11].

Three common PVD parameters include bias voltage, substrate temperature, and N₂ flow rate. Lin *et al.* [23] studied the effect of substrate bias voltage on corrosion properties of chromium carbide thin film on AISI D2 Steel. They reported the Chromium Carbide coated on steel tended to be nobler as bias voltage become more negative. Flores *et al.* [24] evaluated the effect of substrate bias voltage on the corrosion of TiN/Ti multilayer on steel.

They noticed that the corrosion potential was nobler for the samples deposited with the substrate grounded than with bias. Sui and Cai [25] studied the effect of bias voltage on the electrochemical corrosion behaviour of hydrogenated amorphous carbon (a-C:H) film on NiTi. They claimed the a-C:H (10kv) and a-C:H (20 kv) films could block Ni ions out-diffusion from NiTi alloys. Caicedo *et al.* [26] compared the effect bias voltage on corrosion resistance for TiCN and TiNbCN on AISI 4140. They found that as the negative bias substrate increased, the corrosion resistance of the coated samples decreased because of the porosity of the coating produced by ion bombardment at higher bias voltages. Hsu *et al.* [27] studied the effect bias voltage on mechanical properties and corrosion resistance of CrTiAlN on stainless steel. They reported that depositing CrN on a substrate other than TiAlN was more efficient when the bias increased. Shah *et al.* investigated the effect of bias voltage on Ti-13Zr-13Nb alloy. They reported that samples coated with -125V bias voltage provides the least number of microdroplets and results in highest corrosion resistance [28].

In summary is bias voltage is a significant parameter that has a direct influence on corrosion resistance regardless of coating material and substrates combination. However, the literature shows that there are contradictions in the reports as to which polarity provided better corrosion resistance. Negative bias has been said able to increase corrosion rates owing to decreased porosity in the coated samples as a result of higher ion bombardment [12]. Most of these claims were reported

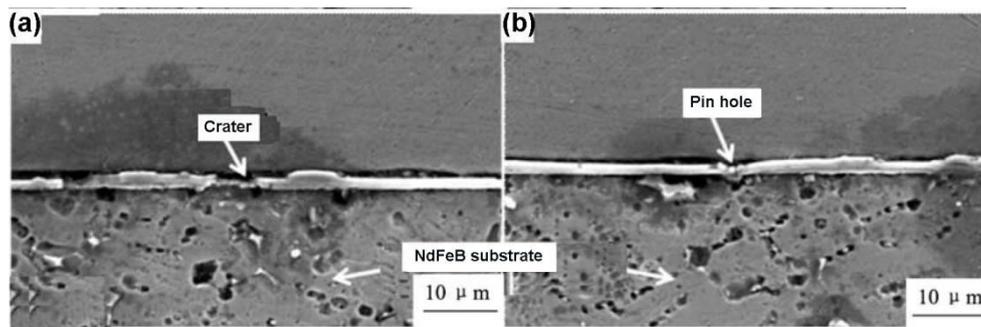


Figure-2. Cross sections of multilayer Ti_2N ceramic coatings on NdFeB substrate (a) crater with thin layer of ceramic coating (b) and pin hole in the ceramic coating [13].

by researchers who worked with various PVD coatings on steel-based substrates. It was not known if the same effects would be observed on TiN coated biomedical implant materials including titanium-based alloys.

Substrate temperature is one parameter that influences corrosion resistance. A team of researchers studied the effect of substrate temperatures for CrN deposited on tool steel in terms of improved corrosion resistance. They reported that increased substrate temperatures led to less corroded area fraction [18]. Other researchers studied the effect of nitrogen doped amorphous carbon (a-C:N) coatings on silicon at different substrate temperatures (100, 200, 300 °C). They observed that corrosion resistance decreased as substrate temperatures increased [29]. Several researchers investigated the effect of substrate temperatures (100 to 500 °C) on carbon coatings applied to 316L austenitic stainless steel. They found that corrosion rates were relatively low and nearly constant up to 300 °C after which they increased when the deposition temperature was increased [30]. In another study, researchers improved corrosion resistance by varying substrate temperatures. They observed that a coating of ZrN on titanium applied at high deposition temperatures causes the exposure of the thin ZrN films to the atmosphere lead to the formation of the oxides and oxynitrides on the surface and an increased resistance to pitting corrosion resistance [31].

As mentioned earlier, N_2 flow rates improve corrosion behaviour. Some investigators studied the effect of (Ti,Cr)N coatings on 316L stainless steel at different nitrogen gas flow rates (0, 1.2, 2.0, 2.4, 3 sccm) in cathodic and anodic environments. They concluded that in cathodic environments, the corrosion resistance of TiCrN increased as the nitrogen gas flow rates increased from 0 to 1.2 sccm but decreased when the flow rate increased to 3 sccm. By contrast, as nitrogen gas flow rates increased to 3 sccm in anodic environments, the corrosion resistance of the coated samples increased significantly [32]. In another study, researchers coated glass with CrN at different nitrogen flow rates. They found that films with lower nitrogen contents exhibited better corrosion behaviour in relation to their microstructure [33]. Another group of researchers studied methods for optimizing the N_2 flow rate of CrN coatings applied to 316L stainless steel. They

reported that the CrN coated SS316L at 20 sccm nitrogen performed the best.

The composition and phase structure of the films change as a function of the nitrogen content, which directly influenced the corrosion resistance of the coated plates [34]. It can be concluded that nitrogen gas flow rates and substrate temperatures are significant factors that improve corrosion resistance. Even though the literature contains contradictory reports on the range needed to produce the best corrosion resistance, the consensus is that increasing the substrate temperature and nitrogen gas flow rate improves corrosion resistance because it removes defects.

MULTILAYER COATING

Permeable defects in PVD coating can be reduced or eliminated by interrupting columnar growth with multilayered coatings. For instance, Park *et al.* [35] prepared interlayers by nitriding, sputtering, and oxidizing TiN coatings on AISI 304. They concluded that the interlayer created using a sputtering process improved corrosion resistance. In another report, Massiani *et al.* [36] studied the effect of PVD and ion implantation of Ti-6Al-4V under an application of TiN. They reported that the ion implantation coating used as interlayer improved corrosion resistance. In another study, Massiani *et al.* [37] investigated the effect of multilayer coatings (TiN/Ti/substrate). They noted that multilayer coatings created by sputtering were better than single layer coatings. In another report, Ries *et al.* [38] investigated the effect of Ti/TiN multilayers. They found that steel coated with Ti/TiN multilayers demonstrated better corrosion resistance than uncoated steel. Creus *et al.* [39] compared the effect of Al/Ti/steel, $AlTiO_2$ /Ti/steel and Al_2O_3 /Al/Ti/steel on corrosion resistance in a NaCl solution. They observed that a double-layer coating of Al/Ti/steel provided better corrosion resistance than a monolayer Al coating in the saline solution. In another study, Lakatos and Hanzel [40] evaluated the effect of multilayer coatings (TiN/Fe/steel, TiN/Ti/Fe/steel, TiN/Ti/TiN/Ti/Fe steel, CrN/steel) on corrosion resistance. They showed that a multilayer coating provided higher corrosion resistance than bilayers and single layers due to a small amount of porosity.



Chen *et al.* [14] carried out a study that compared duplex (TiN/Ti) and single (TiN) layer coatings on AISI 304. They reported that the Ti interlayers not only provided better protection to corrosion but it also enhanced the bond of the TiN/substrate interface.

Vacandio *et al.* [44] applied AlN/AlN_x to mild steel and stainless steel. They noted that the presence of an AlN_x under layer decreased the corrosion resistance of the films because of galvanic coupling. In a separate study, they investigated multilayer coatings (AlN/Ni and AlN/Ti) using different interlayer process such electrochemical (Ni), electroless, and sputtering (Ti) [45]. They concluded that interlayers created using electroless nickel provided the best corrosion resistance. The AlN/electrochemical nickel and AlN/titanium samples were in the same range.

Liu *et al.* [15] studied single (TiN and CrN) and multilayer (TiN/Ti CrN/Cr) coatings on mild steel created using plasma-assisted physical vapour deposition. They reported that multiple layers improved corrosion performance of PVD TiN- and CrN-coated mild steel due to the limited number of coating defects. The dense structure and fine equiaxed crystallites made these coatings less permeable to the corrosive medium.

Hovsepian *et al.* [16] applied Cr using an electroless process and CrN/NbN using unbalanced magnetron sputtering (UBM), steered cathodic arc evaporation, combined steered cathodic arc and UBM sputtering on 316 stainless steel. They found that ABS techniques using multilayer coatings improved corrosion resistance owing to the creation of smoother surfaces and less growth defects. Flores *et al.* [17] deposited multilayer coatings (TiN/Ti, TiN/Ti/TiN/Ti, TiN/Ti/TiN/Ti/Ti) on 304 stainless using magnetron sputtering. They showed that the pitting potential of the three period multilayers deposited with the substrate grounded was almost three times nobler than the substrate

Subramanian and Jayachandran [49] used nickel via brush plating under TiN coating on AISI 316L. They noted that the nickel interlayer improved corrosion resistance and had less passivation current density compared to single TiN layers and uncoated samples. Ali *et al.* [22] investigated the effectiveness of Nickel/copper via electroplating and TiN via CAPVD on NdFeB. They observed that the corrosion resistance of TiN was better compared to the corrosion resistance of the electroplated interlayers (Ni/Cu). Tian *et al.* [50] applied chromium nitride/Cr to 316L stainless steel using a PVD method. They found that the chromium nitride/Cr coating exhibited improved corrosion resistance and better stability to passive film. Bobzin *et al.* [51] prepared amorphous carbon (a-C) and a chromium base as interlayers under a hydrogenated amorphous carbon coating (a-C:H). They reported that multilayer coatings offered good corrosion protection for mild steels in aggressive solutions.

The studies discussed above show, it is summarized that an interlayer coating generally improves the corrosion resistance of coated samples. Duplex coating alone were not able to protect the substrate from corrosion due to permeable defects. Multilayer coatings are needed

to improve corrosion resistance. The success of multilayer coatings is governed by its structure and properties, which depends on the type of process used to produce the coating. These processes can be categorized as surface modifications for interlayer such as PVD, ion implantation, electroless, electrochemical, and brush plating. These processes successfully protect the substrate from corrosion while plasma nitriding and oxidation were not suitable as they created pores that the electrolytes could easily penetrate. Although multilayer coatings provide a potential method to improve corrosion resistance, the cost to produce them is very high. Other alternatives, such as surface modification using mechanical methods may be more suitable but they are rarely discussed in the literature.

CONCLUSION AND FUTURE RESEARCH RECOMMENDATIONS

Coating is widely used in application such as cutting tools, decorative, aerospace industries and biomedical application. The advantage of this method is deposited at low processing temperature. However, PVD coating has limitation Past reports indicated that coated PVD layers have pores, pin holes, and columnar growths that act as channels for aggressive mediums to attack the substrate [22,50-52]. Duplex and multilayer coatings address this issue but at the expense of manufacturing time and cost. Therefore, an alternative method is needed to reduce the penetration of body fluids and react with bare substrate. One of possible surface modifications to PVD coatings uses a mechanical treatment. Several studies have demonstrated that sand blasting PVD coatings increases the compactness and hardness of the coating, which leads to lower wear rates [53-60]. However, very limited literature exists on surface mechanical treatment especially on the application of ultrasonic vibration to reduce corrosion attack of TiN coated Ti based implants. Most researchers have reported the behaviour of mechanical treatment on wear rate mechanism only. Therefore, a detailed study is needed to evaluate the effect of ultrasonic treatments on PVD-TiN coated Ti-13Zr-13Nb alloys in terms of corrosion resistance. It is also suggested that the more deep research can be performed on application of biomedical especially when this coating contact with tissue and body fluids. This application can be used in replacement of The application of biomaterial in musculoskeletal implants include dental implants, artificial hips, and knees prostheses and incorporate the screws, plates, and nails in these devices.

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