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Pulsed DC- Plasma Assisted Chemical Vapor Deposition of α-rich Nanostructured Tantalum Film: Synthesis and Characterization

H. Ghorbani[a], A. Abdollah-zadeh*[a], A. Poladi[b], M. Hajian[c]

[a] *Department of Materials Eng., Tarbiat Modares University, Tehran, Iran*
[b] *Faculty of Materials Science and Eng., Semnan University, Semnan, Iran*
[c] *Department of Materials Science and Eng., Shahrood University of Technology, Shahrood, Iran*

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ABSTRACT

This paper is an attempt to synthesize nanostructured tantalum films on medical grade AISI 316L stainless steel (SS) using pulsed DC plasma assisted chemical vapor deposition (PACVD). The impact of duty cycle (17-33%) and total pressure (3-10 torr) were studied using field emission scanning electron microscopy (FESEM), grazing incidence x-ray diffraction (GIXRD), nuclear reaction analysis (NRA), proton induced x-ray emission (PIXE) and Rockwell indentation methods. The optimized deposition conditions for making the best film characteristics in terms of deposition rate, purity and maximum α-phase was recognized. Also, the results showed that using a near stoichiometric TaN interlayer in this technique improves the film adhesion strength and considerably increases Ta film purity. The NRA analysis results indicated that the pulsed DC-PACVD is capable of producing Ta films with negligible amount of residual hydrogen which makes films needless to post bake treatment.

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1. INTRODUCTION

In recent studies, much attention has been given to surface modification of AISI 316L stainless steel for biomedical applications [1,2]. AISI 316L SS implants are susceptible to pitting, especially when used in-vitro. This is particularly important due to the release of toxic ions such as nickel and chromium ions in biologic environments which may lead to allergic or inflammatory reactions [3–5]. Tantalum (Ta) based coatings are one of the most appealing candidates to eliminate the aforementioned problems, owing to their good mechanical properties, corrosion resistance and biocompatibility [6,7]. Tantalum coatings usually consist of two crystalline phases, namely α-Ta and β-Ta. The stable α-Ta has body-centered cubic (BCC) lattice structure known as ductile Ta phase. The metastable β-Ta (with tetragonal lattice structure) is hard, brittle and susceptible to cracking [8]. Thus, it is necessary for mechanical applications to have an α-rich Ta coating.

Deposition of Ta coating with prevailing α-phase depends on substrate conditions as well as deposition parameters. As for the substrate condition, some sputtering experiments have indicated that both heating the substrate and using a tantalum nitride (TaN_x) interlayer can effectively enhance the growth of α-Ta [9,10]. It is likely that the TaN_x interlayer reduces the lattice mismatch and tensile residual stress at Ta/steel interface [10]. Deposition of Ta thin films can be performed by various techniques such as physical vapor deposition (PVD) [11,12], chemical vapor deposition (CVD) [13–15] and molten salt [16,17]. However, there are some drawbacks using these methods, such as non-uniform deposition (for PVD) and high temperature operation condition (for CVD and molten salt). Nevertheless, CVD is a well-established technique from industrial viewpoint because it can coat complicated components with good adhesion [18]. In recent years, plasma assisted chemical vapor deposition (PACVD) has been introduced as a new alternative technique to

*Corresponding Author's Email: zadeh@modares.ac.ir (A. Abdollah-zadeh)

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alleviate the problems of high temperature CVD process. This high quality CVD method utilizes plasma to produce nanostructured coatings at temperatures below 450˚C [19–22]. Plasma can be generated through various power supplies including DC, AC, AC radio frequency (RF), pulsed DC, etc. [23].

Most studies on deposition of Ta based films by PACVD have been performed via RF power supplies [24–26]. However, despite the benefits of RF plasma sources in producing high quality films, these power supplies provide low deposition rate. Furthermore, the RF systems are complex and it is difficult to scale them up for commercial applications [27]. Comparative studies on sputtering power supplies have shown that using the pulsed DC power provides the best combination of deposition rate, film quality, setup cost and complexity, amongst other conventional power supplies (i.e. RF, DC, AC, etc.). In other words, utilizing the pulsed DC power enhances deposition throughput and facilitates large scale and cost effective plasma deposition. These have resulted in the growing consideration to industrial application of pulsed DC [28–31]. In this regard, given the lack of true understanding about the impact of pulsed DC parameters especially duty cycle on characteristics of Ta films deposited by PACVD, the more investigation is required. In this paper, the effects of the important parameters of pulsed DC-PACVD including total pressure and duty cycle on film purity, deposition rate and phase structure were studied. In addition, the optimal duty cycle and total pressure for deposition of high quality Ta film with maximum α phase were determined. Furthermore, the TaN interlayer was utilized in order to systematically investigate the variation of film properties.

2. MATERIALS AND METHODS

The schematic illustration of the pulsed DC-PACVD system is shown in Figure 1. The base pressure reached 10^{-3} torr and the work pressure was in the range of 3-10 torr.

$TaCl_5$ powder with purity of 99.99% was used as the tantalum precursor. $TaCl_5$ was heated up to 140˚C in a stainless steel sublimator and a 10 sccm H_2 carrier gas transferred its vapor to the chamber. The gases of H_2 (99.999%), Ar (99.999%) and N_2 (99.999%) were entered into the chamber through mass flow controllers. In order to remove residual contaminants and to enhance film adhesion, sputter etching was performed prior to deposition being started. The overall chemical reaction taking place at 350˚C in the chamber is given in Equation (1):

$$2TaCl_5 + 5H_2 \quad 2Ta(s) + 10HCl(g) \qquad (1)$$

Finally, a TaN film was deposited to examine the influence of interlayer on Ta film characteristics. Deposition parameters of pulsed DC-PACVD which were applied in this study, are listed in Table 1.

AISI 316L SS substrates dimensions were 20×20×2 mm. Specimens were ground, polished and ultrasonically cleaned in acetone and ethanol before loading into the chamber. The crystallographic structure of the films was studied by grazing incident x-ray diffraction (GIXRD) using CuK_α radiation (λ=0.1542 nm). A field emission scanning electron microscopy (FESEM) equipped with an energy dispersive x-ray spectroscopy (EDS) was applied to characterize film morphology and composition.

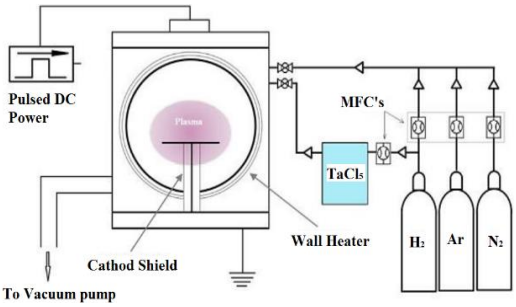

Figure 1. Schematic of Pulse DC-PACVD process

TABLE 1. The process conditions for pulse DC-PACVD of Ta coatings on 316L stainless steel at 350˚C

	Sample No	P (torr)	D.C %	Gas flow ratio (%)			Deposition time (min)	F (kHz)	I (A)
				H_2	Ar	N_2			
Sputter etch	10	11	45	20	75	15	20	8	4
Tantalum monolayers	1	3	17	60	40	0	60	11	2
	2	3	25	60	40	0	60	11	2.5
	3	3	33	60	40	0	60	11	2.5
	4	5	17	60	40	0	60	11	2.5
	5	5	25	60	40	0	60	11	2.5
	6	5	33	60	40	0	60	11	2.5
	7	10	17	60	40	0	60	11	2.5
	8	10	25	60	40	0	60	11	2.5
	9	10	33	60	40	0	60	11	2.5
TaN interlayer	11	5	25	50	25	25	30	11	2.5

The compositional analysis, nitrogen depth profile and stoichiometry of TaN_x interlayer were measured by nuclear reaction analysis (NRA) and proton induced x-ray emission (PIXE). Rockwell adhesion test was also carried out on Ta films, according to DIN-VDI3198 standard.

3. RESULTS AND DISCUSSION

Table 2 represents a qualitative assessment of Ta films phase structure which has estimated from the intensities of the α and β tantalum peaks in GIXRD patterns. The crystallite sizes which were calculated using the Scherrer equation are also given in Table 2. According to the results, the films which produced at the pressure of 3 torr did not show any crystallinity under the x-ray characterization. These samples probably contain an amorphous structure. By increasing the pressure up to 5 torr in all duty cycles, the crystalline structure appears. So, it can be concluded that the total pressure is more effective on formation of crystal structure in films than the duty cycle. It is also shown in Table 2 that increasing the duty cycle at a constant pressure leads to grain growth.

It is known that the grain growth is a thermally activated process. It is accepted that by increasing the total pressure and duty cycle, the surface temperature rises owing to the intensified ion bombardment and the higher pulse on time, respectively [32,33]. However, according to Table 2, it can be deduced that the thermal effect of duty cycle on grain growth is dominated. In other words, the surface temperature rises considerably at higher duty cycle and promotes the diffusion of Ta atoms. Accordingly, this thermal effect affects the film phase structure. It is evident from Table 2 that at the duty cycle of 25% and the total pressure of 5 torr the nucleation of α-Ta is prevailing.

Figure 2 shows that the deposition rate improves as the pressure increases and declines as the duty cycle rises. In fact, the greater duty cycles corresponds to more exposure of surface under the discharge bombardment. Under this condition, it is expected that more number of atoms might be sputtered from the surface and thereby, deposition rate reduces [18]. The unusual reductions in deposition rate at duty cycle of 17% and pressure of 3 torr may be due to the unsaturated plasma atmosphere in terms of gaseous species concentration and ratio of ions to neutral particles.

In order to recognize the relationship between deposition rate and film quality, the purity of Ta in deposited films was determined. As shown in Figure 3, the highest percentage of Ta is observed at duty cycle of 25% and pressure of 5 torr. The film obtained at duty cycle of 33% and pressure of 10 torr contains the largest amount of impurities.

TABLE 2. Summary of the xrd results of ta films on aisi316l stainless steel

Sample No.	P (torr)	Duty Cycle %	Crystallite size (nm)	Film phase structure
1	3	17	-	x-ray amorphous
2	3	25	-	x-ray amorphous
3	3	33	-	x-ray amorphous
4	5	17	60.3	Strong β(002)
5	5	25	87.4	Strong α(110), weak β(002)
6	5	33	106.5	Mixed α(110) and β(002)
7	10	17	66	Strong β(002)
8	10	25	96.1	Strong β(002), weak α(110)
9	10	33	138	Strong β(002)

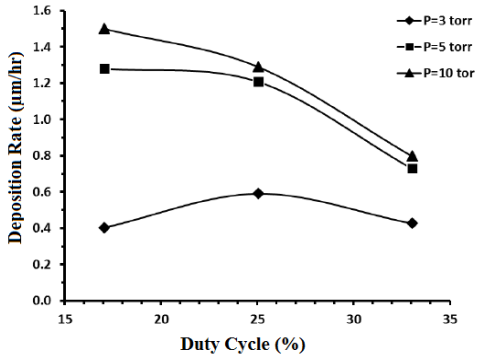

Figure 2. Effect of duty cycle on tantalum deposition rate under different total pressures

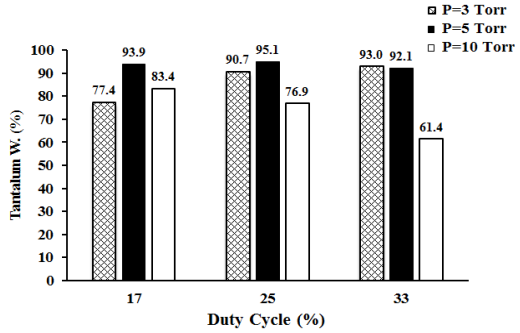

Figure 3. Purity of PACVD tantalum coatings deposited at various duty cycles and total pressures obtained from EDS elemental analysis

Therefore, it can be concluded that at the highest deposition rate, the purity of tantalum is not necessarily maximum. The optimal condition for deposition of Ta films in terms of maximum α-Ta nucleation, the least contamination and the desired deposition rate was acquired at 5 torr pressure and 25% duty cycle. It seems that in this duty cycle and pressure ranges the plasma

conditions is in such that the nucleation and growth are simultaneously at the optimum state. In other words, from the viewpoint of process, at pressure of 5 torr, the reactants get enough residence time in chamber until the deposition reaction efficiently takes place and the duty cycle of 25% offers the most appropriate pulse off time, required to remove by products from the surface. Nevertheless, even a small amount of brittle β-Ta in this case can potentially act as a source of crack leading to premature destruction of films [8]. Knowing this, it is ideal to achieve a completely α-phased structure with attempts to minimize the amount of β phase. Some previous studies on sputtering and RF PECVD of Ta films have proposed that utilizing a TaN_x interlayer can be helpful to improve the nucleation of α-Ta [10,24]. So, for further investigation about the effects of TaN_x interlayer on Ta film properties in this technique, a stoichiometric TaN film was deposited according to the conditions given in Table 1 before starting the deposition of Ta film in optimal condition (mentioned above). Figure 4 depicts the GIXRD patterns of Ta single layer which deposited at optimal condition (pressure of 5 torr and duty cycle of 25%) compared with the Ta/TaN double layer coating. As can be seen, by using TaN interlayer, a predominant α-Ta structure is acquired. Gladczuk et al. [10,34] exhibited that the mismatch between α-Ta (110) plane and TaN (111) planes is less than 2%. This conformity leads to reduce in film interfacial energy and results in preferential nucleation of α-Ta on TaN interlayer. The through thickness depth profile of Nitrogen in Ta/TaN film also shows that the concentration of N in interlayer is almost 50%, confirming the formation of stoichiometric TaN (Figure 5a). The width of high nitrogen area is approximately 0.38 µm.

Another issue concerning the deposition on steel by PACVD is the hydrogen atoms which remain in film structure. It is known that diffusion of residual hydrogen into the steel substrate may probably cause the hydrogen embrittlement. The through thickness concentration profile of hydrogen in Figure 5b verified the negligible hydrogen content (<1.1 at. %) in Ta film deposited through pulsed DC-PACVD. The concentration of residual hydrogen in this condition is low enough to cause hydrogen embrittlement in steel substrate. So, it can be concluded that the films produced in this process are needless of post bake treatment. In another experiment, Suh et al. [24] observed that the amount of residual hydrogen in Ta films deposited by RF PACVD exceeds 1.5% which is higher than what is observed in this study (Figure 5b).

In order to compare the film purity of Ta and Ta/TaN films, the PIXE quantitative elemental analysis was applied (Table 3). It is revealed that the presence of stoichiometric TaN interlayer promotes the Ta film purity. This is likely that TaN layer acts as a barrier and prevents sputtering of substrate elements and their entrance into the deposition atmosphere. The top surface microstructure and the cross sectional overview of Ta/TaN film is illustrated in Figure 6. The film surface is consist of dense grain facets (Figure 6b) which provide a crackless and uniform coverage for steel substrate. However, in the absence of sufficient film adhesion, the surface protection will not occur. Hence, to study adhesion, the Rockwell indentation test was carried out on Ta films according to DIN-VDI-3198 standard (Figure 7). Normally, delamination is associated with the amount of shear stress in the interface [35]. Furthermore, brittleness, lattice mismatch and residual stress cause film decohesion and delamination.

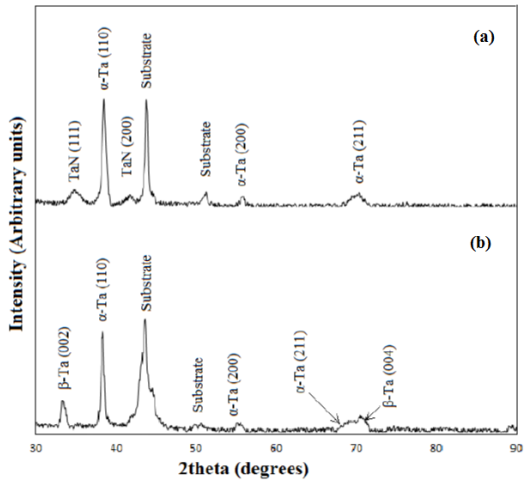

Figure 4. XRD patterns of Ta coating on TaN seed layer and (b) Ta single layer

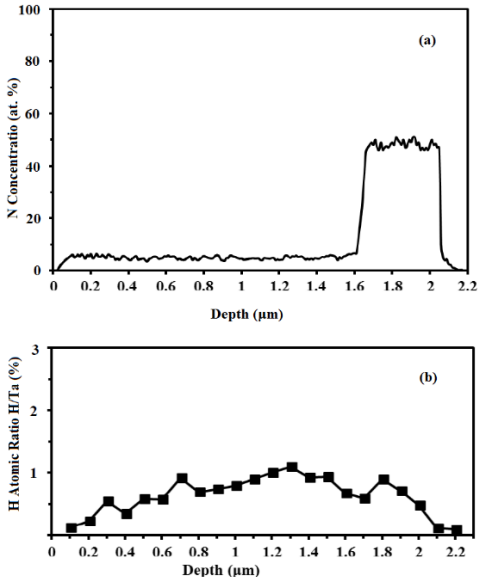

Figure 5. NRA results for PACVD Ta/TaN films as a function of (a) nitrogen and (b) hydrogen depth profile

TABLE 3. Quantified elemental analysis of single and double layer tantalum coatings measured by PIXE (at. %)

	Ta	Fe	Cr	Ni	Mn
Ta single layer	93.42	6.45	0.09	0.11	0.04
Ta/TaN double layer	98.38	1.31	0.03	0.05	0.02

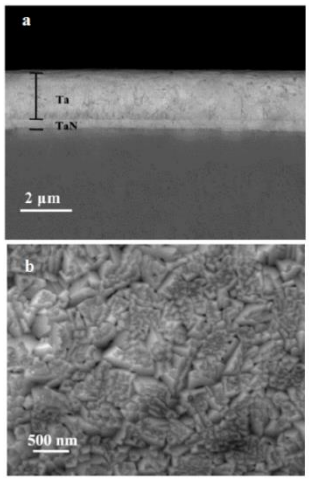

Figure 6. FESEM of (a) cross section and (b) top surface of tantalum coating deposited on TaN seed layer

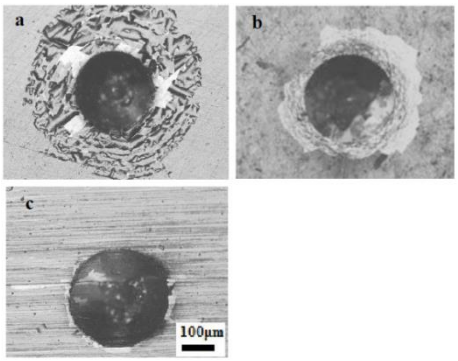

Figure 7. Qualitative adhesion test for film grown at (a) 5 torr pressure and 17% duty cycle with a β- rich monolayer Ta film, (b) the film that grown at 5 torr pressure and 25% duty cycle with a α- rich monolayer tantalum film and (c) TaN/Ta bilayer indicating the maximum alpha content

According to DIN-VDI-3198 criteria, the film grown at 5 torr pressure and 17% duty cycle (Figure 7a) and the film that grown at 5 torr pressure and 25% duty cycle (Figure 7b) have not acceptable adhesion. This can be attributed to the presence of residual stress as well as considerable amount of brittle β-Ta. On the other hand, the negligible delamination in Ta/TaN film (Figure 7c) implies its desirable adhesion strength. It can be concluded that, in addition to modification of the film phase structure, introducing the TaN interlayer eliminates the lattice mismatch strain between Ta and steel substrate and improves the film adhesion strength.

4. CONCLUSIONS

In summary, Ta films were successfully synthesized on biomedical grade AISI 316L SS through pulsed DC-PACVD. The impacts of duty cycle and total pressure on the film characteristics were investigated. It was found that the Pressure is mainly responsible for the formation of crystal structure while the duty cycle acts as an effective factor in the grain growth. Furthermore, the optimum condition in terms of film purity, deposition rate and α-phase nucleation was achieved at 5 torr pressure 25% duty cycle. Also, it is revealed that utilizing stoichiometric TaN as an interlayer, apart from the significant enhancement in α-Ta nucleation, improves the film adhesion strength. Likewise, TaN interlayer acts as a barrier layer and prevents the substrate elements to be sputtered and take part into the deposition process. Negligible concentration of residual hydrogen, which was detected by NRA, implies that the pulse DC-PACVD does not require post bake treatment.

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H. Ghorbani[a], A. Abdollah-zadeh[a], A. Poladi[b], M. Hajian[c]

[a] *Department of Materials Eng., Tarbiat Modares University, Tehran, Iran*
[b] *Faculty of Materials Science and Eng., Semnan University, Semnan, Iran*
[c] *Department of Materials Science and Eng., Shahrood University of Technology, Shahrood, Iran*

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Characterization

چکیده

در این مقاله تلاش شده است تا فیلم نانوساختار تانتالوم با استفاده از روش رسوب شیمیایی از بخار به کمک پلاسمای پالسی (pulsed DC-PACVD) بر روی فولاد زنگ نزن ۳۱۶L با کاربری پزشکی ایجاد گردد. تاثیر چرخه‌ی کاری (از ۱۷ تا ۳۳٪) و فشار کل فرایند (از ۳ تا ۱۰torr) به‌وسیله‌ی میکروسکوپ الکترونی روبشی نشر میدانی(FESEM)، پراش اشعه ایکس(GIXRD)، آنالیز بازخورد هسته‌ای(NRA)، آنالیز (PIXE) و دستگاه سختی‌سنج راکول بررسی شد. شرایط بهینه رسوب‌دهی با هدف حصول پوششی با بهترین مشخصات، از حیث نرخ رسوب، خلوص و حداکثر رسوب تانتالوم فاز آلفا تعیین شد. همچنین، نتایج نشان داد که در این روش، استفاده از میان‌لایه‌ی نیترید تانتالوم با نسبت نزدیک به استوکیومتری ۱:۱ علاوه بر افزایش استحکام چسبندگی، خلوص پوشش تانتالوم را نیز به طور قابل توجهی بهبود می‌دهد. نتایج آنالیزNRA نشان داد که روش pulsed DC-PACVD قادر به تولید پوشش تانتالوم با میزان هیدروژن باقی‌مانده-ی ناچیز است به طوری که پوشش حاصله را از انجام عملیات حرارتی تکمیلی بی نیاز می‌سازد.

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