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# Influence of duty cycle on biocompatibility of nanostructured tantalum film deposited by pulsed DC PACVD

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## Abstract

Nanostructured Tantalum (Ta) thin films were deposited by plasma assisted chemical vapor deposition (PACVD) at duty cycles of 17%, 25%, and 33%. FESEM and MTT assay indicated that the Ta film deposited at duty cycle of 25% provides the best surface biocompatibility because of its flawless and uniform structure. Furthermore, Rockwell adhesion test indicated that the best film adhesion observed in Ta film that deposited at duty cycle of 25% and this attributed to its dominant α-phase structure.

**Keywords:** Nanostructure; Tantalum; PACVD; Biocompatibility

## Introduction

AISI316L stainless steel (SS) is widely used in medical industry. It is mainly applied in biomedical implants, stents and medical devices [1,2]. However, long term implantation of them may result in pitting and releasing of toxic metallic ions in body environment [3,4]. Therefore, a surface modification process is necessary [5]. It is well accepted that tantalum thin film improves its corrosion resistance and biocompatibility [6].

Usually tantalum includes two phase structure: α-phase which is ductile with bcc lattice structure and β-phase which is brittle and susceptible to crack with tetragonal lattice structure [7]. So, for mechanical application it is preferred to get a α-phased microstructure with maximum uniformity and density. Among all deposition techniques, the sputtering has been the most conventional method for deposition of tantalum in low temperature [8,9]. However, sputtering is a line-off-sight method which distance from surface to target influences the film quality and thickness specially for coating the parts with complex geometry [10]. In recent years, plasma assisted chemical vapor deposition (PACVD) has introduced as a low temperature, non-line-off-sight method which provides the better film adhesion and surface covering [11].

Among all parameters affecting the film properties in pulsed DC PACVD, the duty cycle is one of the important parameters which affects film growth and morphology. Given that, the true understanding about the relationship between duty cycle and Ta film characteristic in pulsed DC PACVD is lacking.

So, the aim of this paper is to study the effects of duty cycle on biocompatibility of the Ta films deposited by pulsed DC PACVD on 316L SS.

## Materials and methods

Tantalum was deposited on 316L SS coupons using a pulsed DC PACVD system at 300 ℃, pressures of 5 torr and duty cycles of 17%, 25%, and 33% for 90 min. $TaCl_5$ was used as a tantalum precursor in deposition reaction. Prepared cells were cultured on tantalum coated and uncoated surfaces for 1, 3 and 5 days. Then the MTT assay was used to determine relative cell viability. The fresh medium containing MTT solution (5 mg/ml in PBS) was added in a 5:1 ratio. After 2 h incubation at 37 ℃, the medium was removed and the precipitate was dissolved in dimethyl sulfoxide (DMSO). The absorbance was analyzed using a microplate

reader (BioTek ELX×800) at 540 nm. In order to characterize the films morphology a field emission scanning electron microscopy (FESEM) was applied. The grazing incident x-ray diffraction (GIXRD) using $CuK_{\alpha}$ radiation was applied to study the crystallographic structure of films. Rockwell adhesion test was also conducted on Ta films, according to DIN-VDI3198 standard.

## Results and discussion

According to Fig. 1, Ta films that deposited at duty cycles of 17% and 33%, cell proliferation after the first day, is negligible while for films that obtained at duty of 25% it proceeds. As shown in Fig. 2, at duty cycle of 33% the deposited films are more porous compared to that obtained at 25%. In addition, cracks and discontinuities which are evident in film which produced at duty cycle of 17% limit surface protection against corrosion. In higher duty cycle, 33%, because of intensified atomic collisions, tantalum atoms lose their kinetic energy and have not enough energy to diffuse along surface and to find suitable position in lattice. So, the surface porosities and voids are expectably raised in this condition (Fig. 2c). However at lower duty cycle, 17%, deposition rate is reduced and nucleation is restricted. Consequently, non-uniform growth occurs which leading to creation of cracks and porosities at duty cycle of 17% (Fig. 2a). Discontinuities in films can provide less cell viability through substrate corrosion and release of toxic alloy elements in culture environment. But at duty cycle of 25%, it seems that enough pulse off time ($T_{off}$) in this duty cycle provides favorable condition in order to exit gas removals and a balanced nucleation and growth of Ta lead to a nanostructured and non-porous film without any cracks.

However, in order to achieve a modified AISI 316L surface structure via Ta film deposition, apart from a proper surface protection, the films should have appropriate adhesion strength. The adhesion strength of deposited film is evaluated according to DIN-VDI-3198 criteria and is shown in Fig. 3. As can be seen, the trivial delamination and radial cracks in vicinity of the indentation area in the film that deposited at duty cycle of 25% (Fig. 3b) imply an acceptable and strongly adhered coating. On the other hand, films which produced at duty cycles of 17% (Fig. 3a) and 33% (Fig. 3c) have not acceptable adhesion strength. This can be due to the considerable amount of brittle β-Ta phase which can produce residual stress in films. The GIXRD patterns of deposited films have been depicted in Fig. 4. The non-crystalline structure in duty cycle of 17% and dominant β-phase is evident in Fig. 4a and Fig.4c respectively. It is

revealed that nano-structured Ta film with prevailing α-phase is achieved at duty cycle of 25% (Fig. 4b). The presence of small amount of brittle β-Ta may act as a source of crack and affect surface protection.

## Conclusions

Deposition of nanostructured Ta on 316L SS at duty cycles of 25% using pulsed DC PACVD provided the best cell viability and cell proliferation compared to those for 17% and 33%. This attributed to the dense and uniform nanostructured tantalum. On the other hand the study of films adhesion demonstrated that the best film adhesion achieves at duty cycle of 25% which caused by its predominant ductile α-phase microstructure.

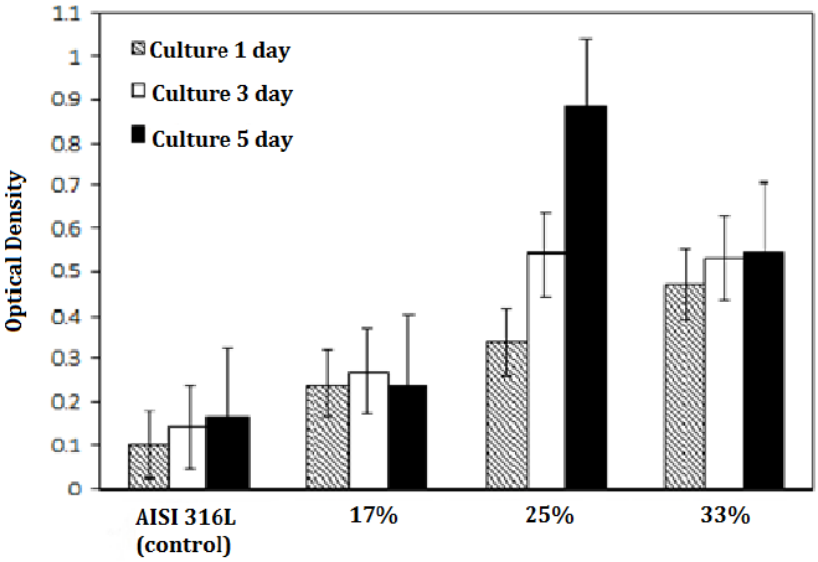


**Fig. 1. MTT assay of cells on Ta films deposited at duty cycles of 17%, 25%, and 33% and on uncoated 316L SS as a control sample.**

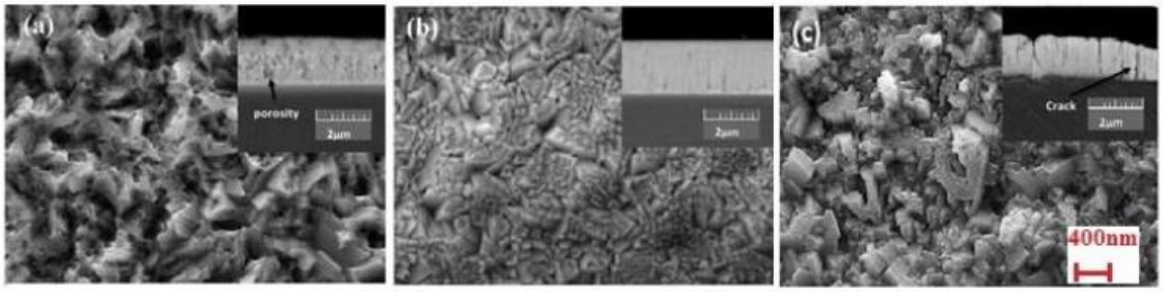


**Fig. 2. FESEM images of top surface and cross section of Ta films deposited at duty cycles of (a) 17%, (b) 25%, and (c) 33%.**

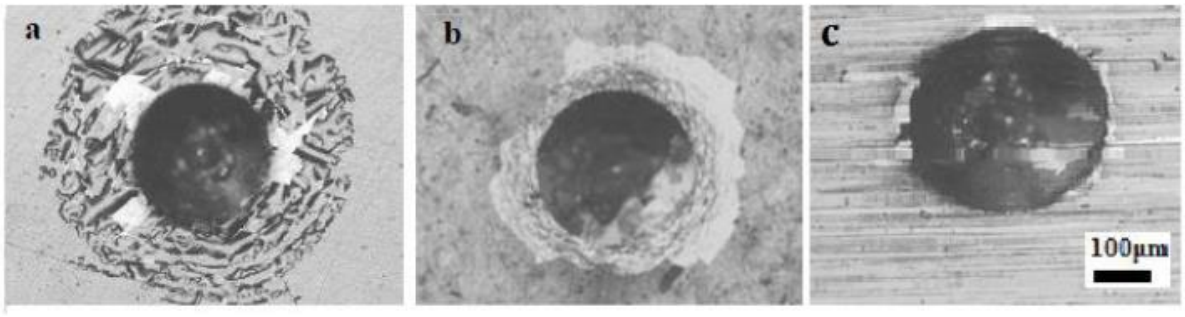


**Fig. 3. Qualitative adhesion test for Ta films deposited at duty cycle of (a) 17% (b) 25%, and (c) 33%.**

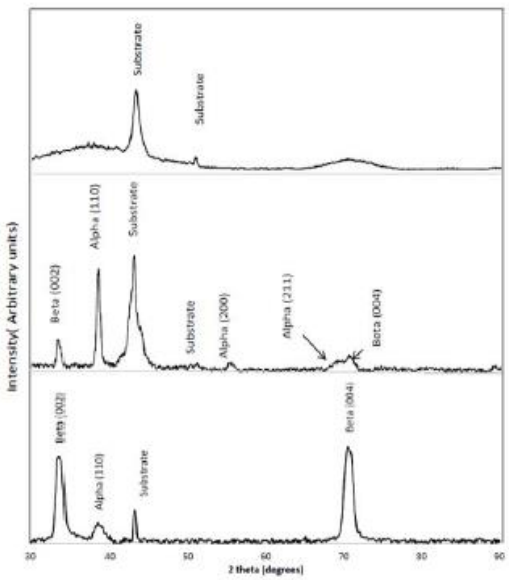


**Fig. 4. GIXRD patterns of Tantalum films deposited at duty cycle of (a) 17% (b) 25%, and (C) 33%.**

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