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Improvement of Austenitic Stainless Steel Implant via Dual Layer Coating of TaN-DLC Using Sputtering and PACVD Methods

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ABSTRACT

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In the current study, in order to improve the properties and performance of SS316L implants, the surface of these implants was coated using two methods of sputtering and plasma-assisted chemical vapor deposition (PACVD). TaN and diamond-like carbon (DLC) layers were applied using sputtering and PACVD methods, respectively. Structural examinations by field-emission scanning electron microscopy (FESEM) showed that the TaN layer was formed in a compact and quasi-spherical morphology. The final DLC layer was also formed in a compact and spherical morphology. Raman spectroscopic results showed that the D and G peaks with suitable heights were at 1356 cm⁻¹ and 1588 cm⁻¹, respectively, indicating the successful formation of DLC. Atomic Force Microscopy (AFM) images indicated that the grain size was in the range of 20 to 35 nanometers, and the presence of very fine DLC grains contributed to reducing the surface roughness to Ra = 1.02 μm, indicating a 67.5% reduction. Cell adhesion test results up to 48 hours indicated better performance of DLC compared to TaN. Thus, the TaN-DLC two-layer coating is introduced as a new coating to improve the performance of implants.

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1. INTRODUCTION

Medical implants are recognized as one of the important innovations in the medical industry. These implants are used as therapeutic artificial substitutes for patients who require replacement of their body parts due to serious injuries or chronic diseases. To create high-quality and durable implants, there is a need to use

materials that are compatible with the human body and have suitable mechanical properties such as strength, elasticity, and hardness, as well as a designed structure that follows a method of production that incurs less cost and time. For example, implants are made from polymer, metal, ceramic, or a combination of these materials [1-3].

Implants made of biocompatible materials are one of the suitable solutions for temporary or permanent

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replacement of defective or damaged bones in the human body [4]. While medical-grade stainless steels are known for their corrosion resistance and high mechanical strength, their potential long-term lifespan and potential side effects such as the release of metal ions in the human body are a concern. SS316 has become one of the most widely used biomaterials for implants due to its easy design and manufacturing, good mechanical properties and resistance to corrosion, as well as its lower cost compared to titanium alloys. However, its insufficient wear performance limits its direct use as a prosthesis [5,6]. One possible solution to prevent the destruction of implants is the use of alternative materials such as polymeric and ceramic composites. These materials are highly resistant to wear and also have high biocompatibility properties [7-8].

Research has shown that these materials can reduce the level of wear and, consequently, reduce the production of metal ions in the human body. Surface modification techniques can also lead to resistance to the destruction of metal implant surfaces and improve biological materials [9-11]. Due to their unique physical and chemical properties, diamond-like carbon (DLC) coatings are attractive alternative biocompatible coatings to traditional coatings. Since DLC is typically identified as an amorphous hydrogenated carbon (a-C:H) with hybridization of sp^2 and sp^3 bonds, they possess properties such as high hardness, low friction coefficient, good wear resistance, chemical inertness, and high electrical resistance [12-13]. The use of DLC coatings in biological implants is very useful due to advantages such as wear resistance, high load-bearing capacity, antibacterial properties, and facilitating bone absorption. Additionally, DLC coatings can also act as a barrier to other biological materials and reduce their wear [12]. Therefore, the use of DLC coatings as biological coatings in medical implants is promising and can lead to improved performance and longevity of implants. One of the problems with DLC coatings is their lack of adhesion. The formation of an interlayer of metal carbide or nitride (such as Ti, Ta, Si, etc.) can increase the adhesion of DLC coatings to the metal surface. This interlayer is usually created using physical vapor deposition (PVD) or chemical vapor deposition (CVD) [14-15]. This layer acts as a bridge between the DLC coating and the metal surface and can prevent the coating from separating from the metal surface and improve adhesion [16-17].

Wang et al. [18] demonstrated that adding a titanium carbide or nitride interlayer between steel and DLC film can improve the bonding strength by increasing the Ti-C bonding strength due to the formation of strong covalent bonds between Ti and C. Lee [19] showed that the use of duplex coatings can significantly improve tribological performance. In fact, the anti-wear performance of duplex coatings is almost ten times higher than that of single-layer DLC films. In addition, duplex coatings have

better resistance to damage. These improvements are due to the combination of mechanical and tribological properties of the interlayer. Wu's results [20] also showed that the use of a Ti-C interlayer in DLC film has a significant improvement in wear resistance and friction coefficient. Moreover, this interlayer can reduce scratch cracks and create greater adhesion, and overall, the use of Ti-C interlayer can help improve the performance of DLC film. The TaN coating, as an interlayer, can indeed modify the surface roughness and provide a smoother surface. This, in turn, allows the DLC coating to be applied on a more even and uniform surface. As a result, the DLC coating exhibits improved mechanical properties and better performance.

The aim of this study is to investigate the formation of DLC coating without the presence of TaN interlayer, in the presence of TaN interlayer, and to examine its biocompatibility.

2. MATERIALS AND METHODS

This research used SS316 stainless steel samples with dimensions of 2 mm x 20 mm x 20 mm. Initially, the samples were grinded with sandpaper up to number 5000 and then polished with 0.01-micrometer diamond paste for 30 minutes. Using the MSS160 RF magnetron sputtering device and a 99.999% pure Ta target, the samples were coated at a base pressure of 2.8×10^{-3} mbar and a power of 185 W for 60 minutes at a temperature of 350 degrees Celsius with a 30Ar/10N₂ gas mixture. Then, the samples were coated using the Hindivac MSPT12 PACVD system, under a working pressure of 45 mTorr, power of 150 W, and a deposition time of 60 minutes, using a gas mixture of 3 Ar/1 C₂H₂.

To examine the structure of the samples, a TESCAN MIRA3 field emission scanning electron microscope (FESEM) was used, and the surface roughness and topography of the samples were examined using an atomic force microscope (AFM). To determine the type and amount of bonding, an Upright microscope (CW YAG: DPSS Nd laser model) with a power range of 10-90 mW, a resolution of 6 μm^{-1} , and a wavelength of 512 nm was used to investigate the bonding types and amounts.

In this study, MG63 cells from the Pasteur Institute of Iran were prepared. RPMI culture medium with 10% FBS serum was used for cell growth. The cells were then maintained in an incubator at 37 degrees Celsius, 95% humidity, and a 5% injected CO₂ level. The biocompatibility of each coating, the cell survival rate, and growth were qualitatively tested using the MTT assay. In this assay, the activity of mitochondrial dehydrogenase enzymes in live cells is examined. In this test, the (MTT) solution is affected by the dehydrogenase enzymes present in the cell's mitochondria, and is

converted into insoluble purple formazan. The amount of formazan produced is naturally proportional to the number of live cells. The intensity of coloration is examined by a simple spectrophotometric test. DAPI

staining was used to examine the number of live cells on the coatings.

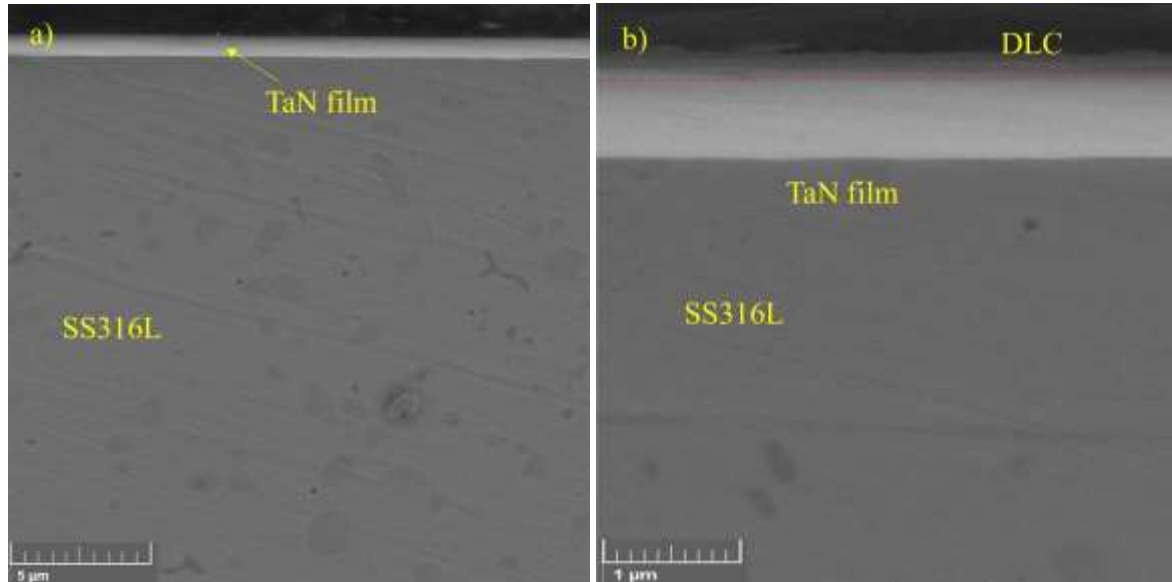


Figure 1. FESEM images of the cross-section surface of coatings: (a) TaN, and (b) TaN-DLC

3. RESULTS AND DISCUSSION

In Figure 1, FESEM images of the cross-section surface of TaN coating formed by magnetron sputtering and a TaN-DLC bilayer coating created by the combination of two methods, magnetron sputtering and PACVD, are shown. In part (a) of Fig. 1, it is evident that a uniform TaN coating with a thickness of about 700 nm has been formed on the substrate. Considering the nature of the magnetron sputtering process, in which nucleation occurs due to the ballistic atom bombardment towards the substrate, growth is also universal, and there is no sign of preferred growth [21]. In other words, the TaN coating grows uniformly on the underlying substrate. This results in the homogeneity of the TaN coating, meaning that all points of the coating grow in the same manner and at the same rate. In part (b) of Fig. 1, the formation of a thin DLC layer with an approximate thickness of 150 nm on the interlayer of TaN is shown. Based on the high magnification of Fig. 1b, the upper layer of the coating has also been formed completely uniformly. Accordingly, the TaN-DLC bilayer coating has been formed on the SS316L substrate.

Figure 2 shows FESEM surface morphology of coatings. Since the magnetron sputtering process is a relatively fast process, the structure of the TaN coating can be amorphous, semi-crystalline and crystalline with stable and semi-stable phases [22]. Due to the high speed of the process, nucleation dominates over growth, and the coating grains, which are very uniform and small, form the coating morphology, as can be seen in Fig. 2a. There

is no trace of grain or inconsistency in the magnified image. This indicates the formation of a healthy coating without any secondary phases or impurities. In Figure 2b, the morphology of the TaN-DLC coating is also shown. In this sample, the coating is completely uniform and free of any defects. Generally, in thin film coatings, since the thickness is very small, the morphology of the coating is affected by the topography of the substrate. In fact, the grooves resulting from grinding and polishing cause the coating to grow in parallel bands. Since grinding and polishing effects were less observed in the preparation of the substrate in this study, TaN and consequently DLC coatings were formed completely smooth.

The TaN coating is formed as a result of bombardment by the ballistic launch of atoms on the surface, which appears as some natural pits or craters in the process. However, DLC coating is formed on the substrate surface based on vapor phase movement, so all the pores and grooves are filled. As a result, the number of grooves or craters in samples with TaN-DLC bilayer coating is less than TaN coating, which can be clearly observed in the comparison of both samples in Figure 2. Consequently, this issue will also have an impact on the surface roughness, which will be discussed further below.

Typically, thin film coatings of Ta are formed in a columnar structure on the substrate, which is why their morphology appears spherical or semi-spherical. However, with the addition of C and N and the formation of TaC or TaN, due to the high density of the resulting coating, there is no sign of a columnar structure in the

microscopical images of the cross-sectional surface. To prove this point, one can refer to the FESEM images of the cross-sectional surface shown in Fig. 1.

In the present study, interstitial N atom in Ta atoms of dense coating leads to an increase in the mechanical properties of the coating. However, on the other hand, the incorporation of N in the system leads to a distortion in the lattice, which on one hand prevents the growth of coating grains and on the other hand increases the mechanical properties of the system due to its fine structure. The distortion or strain created in the lattice results in a severe plastic deformation within the lattice. This process, along with the high speed of the coating process, i.e., the rapid ballistic bombardment of Ta atoms, creates a localized amorphous field within the structure. In fact, the created coating is a combination of crystalline and amorphous structures, which has been proven in previous studies [23,24]. The conditions for the formation of the TaN phase with long-range order are not provided by the combination of amorphous and crystalline phases. In other words, the non-observance of the morphology of the coating has various reasons such as the high volume of the amorphous phase, severe fineness, and the removal of columnar structure, all of which indicate the formation of a dense coating.

The presence of both crystalline and amorphous phases in the coating creates a balance between softness and brittleness, leading to an increase in mechanical properties. Amorphous phases improve corrosion resistance and toughness, while crystalline phases are responsible for increasing hardness and strength. As the grain size and crystallite size decrease, it leads to an increase in grain boundary suppression and mismatch, ultimately resulting in an increase in hardness [24]. The lack of coherence between coating phases through the creation of anti-phase boundaries also leads to an increase in hardness.

DLC coating morphology includes carbon nanoclusters. These clusters are formed by the extensive aggregation of carbon atoms on the interlayer of TaN. The slow deposition rate in PACVD leads to the formation of larger clusters, which are visible in Fig. 2b. This phenomenon is consistent with previous research [25]. The FESEM image in Fig. 2b shows the formation of very small grains in the nanometer order, which undoubtedly affect the roughness and mechanical properties of the coating.

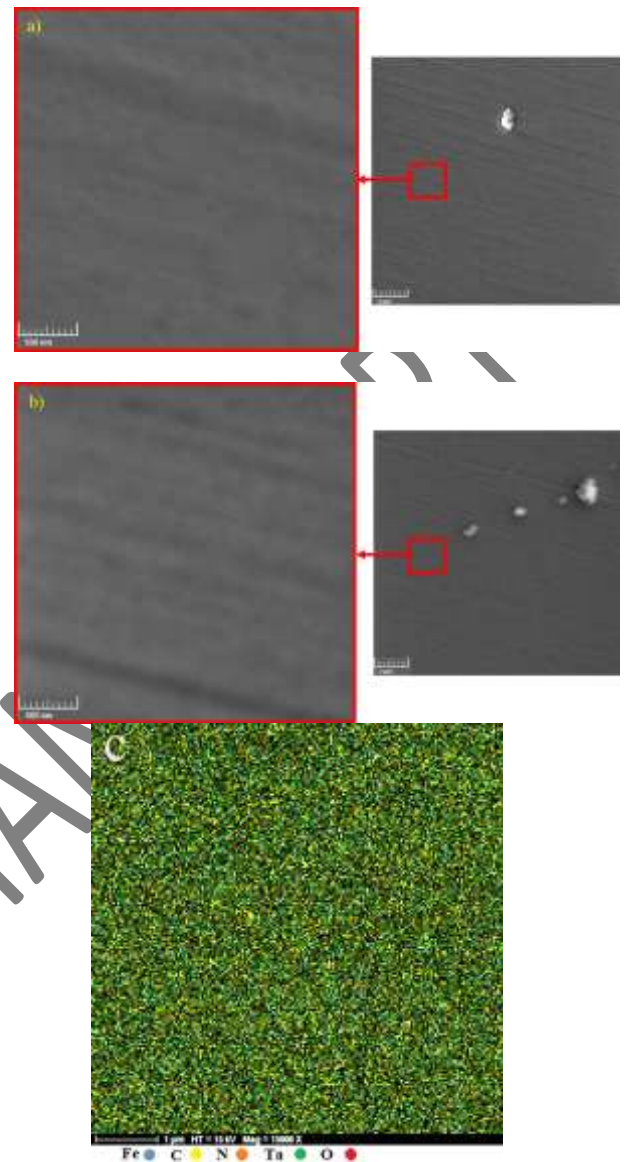


Figure 2. FESEM images of coating morphology: a) TaN, and b) TaN-DLC c) MAP of TaN-DLC.

Figure 3 shows the Raman pattern of the thin DLC layer formed on the interlayer of TaN. Based on this image, the D peak at 1356 cm^{-1} is formed, indicating simple bonds between carbon-carbon atoms, and the G band peak is formed in the range of 1588 cm^{-1} , indicating vibrations of the six-membered carbon rings. These two peaks, along with these two characteristics, indicate the successful formation of the thin DLC layer [26,28].

In the graph shown in Fig. 3, there is a prominent G peak due to the tension of the bond bands of all sp^2 paired atoms in the rings and chains of the structure. The low-height D peak also indicates the vibrational modes of sp^2 atoms in the ring structure. The position of the G peak is

of great importance as it affects the I_D/I_G ratio and the sp^2/sp^3 ratio [28]. As the sp^2/sp^3 ratio increases, the position of the G peak increases compared to the I_D/I_G ratio in an amorphous structure. In general, carbon obtained from ethylene prevents the growth of poly crystalline film and leads to the formation of amorphous DLC coating [29].

By reducing the peak G value, the proportion of sp^3 linkages changes and the breakage of graphite in the low coverage system decreases. The sp^3 degree is highly important because it indicates the presence of residual stress in the system and prevents the formation of large clusters in the DLC film, as shown in Fig. 2b. Proper clustering increases the peak G value and, as a result, increases the disorder in the system by increasing the sp^3 content and leads to the formation of an amorphous structure. This is while, with an increase in the I_D/I_G ratio, less sp^3 content remains in the structure [30].

The presence of H_2 in the system increases the peak D value, and the loss of H_2 also causes the DLC network to graphitize. During the coating process, H_2 molecules penetrate the coating in a reverse manner and leave the coating. At this moment, the non-bonding sp^2 atoms are connected to each other and form six-membered rings to minimize the energy within the system. The sp^2 atoms are rearranged again and lead to a short-range order and stability of the amorphous structure [31].

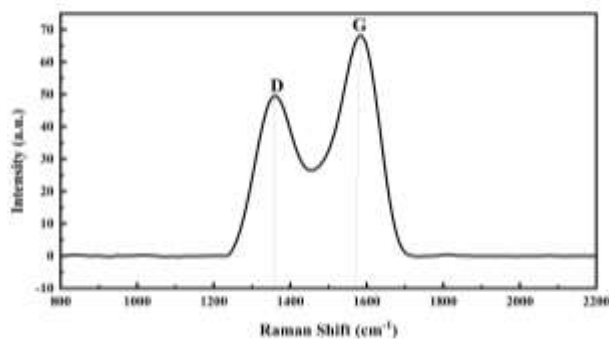


Figure 3. Raman spectroscopy pattern of the thin DLC film on the TaN interlayer.

In Fig. 4, AFM images of two samples, TaN and TaN/DLC, are shown. At first glance, it is clear that the surface roughness has been reduced by applying the thin DLC layer. The grain size of TaN is about 30 nm with a surface roughness of $R_a = 3.14 \mu\text{m}$, which has been reduced to 21 nm and $R_a = 1.02 \mu\text{m}$ after applying the DLC layer. The presence of fine grains also leads to an increase in the strength and a decrease in the surface roughness. This is because the probability of surface roughness occurrence is significantly reduced with the presence of uniform fine grains.

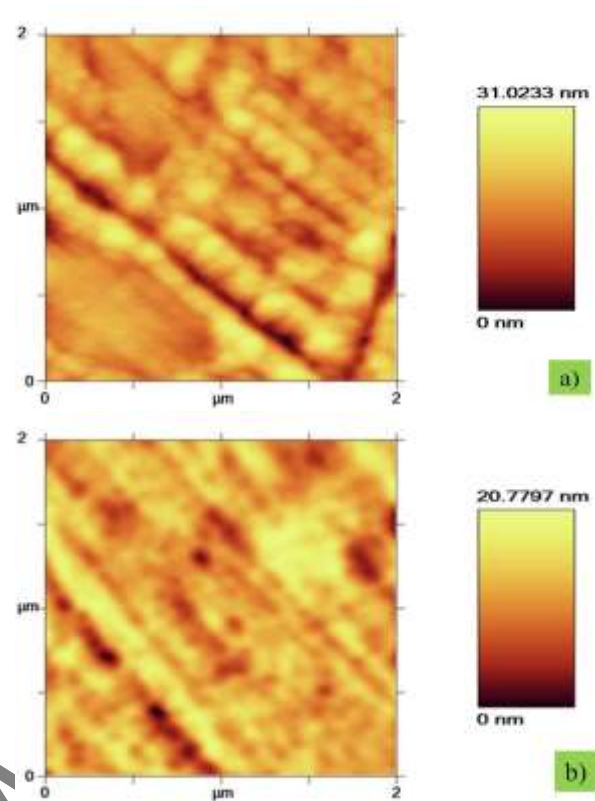
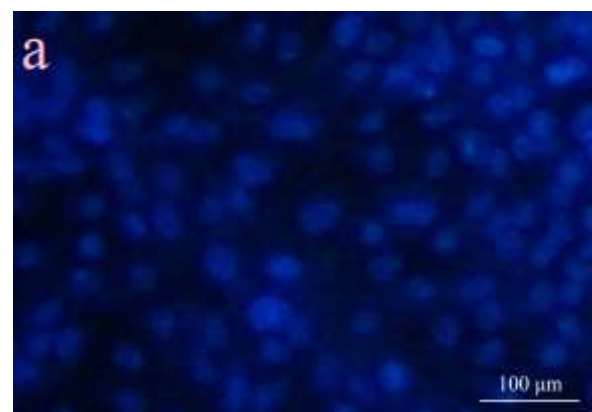


Figure 4. AFM images of coated samples: a) TaN, b) TaN-DLC.

In Fig. 5, the adhesion, spreading, and morphology of MG63 cells on two TaN and TaN-DLC samples after 48 hours of culture are shown. At first glance, it is clear that the number of remaining cells on the TaN-DLC sample is higher than the TaN sample. However, upon closer examination, it can be seen that the cells are growing and expanding very well and uniformly in all directions. On the surface of the TaN-DLC sample, mesenchymal cells are spreading and adhering to the substrate in all directions. This event marks the beginning of cell spreading to the surface by filopodia. This indicates a stronger cellular adhesion and better cell growth on the DLC layer compared to TaN.



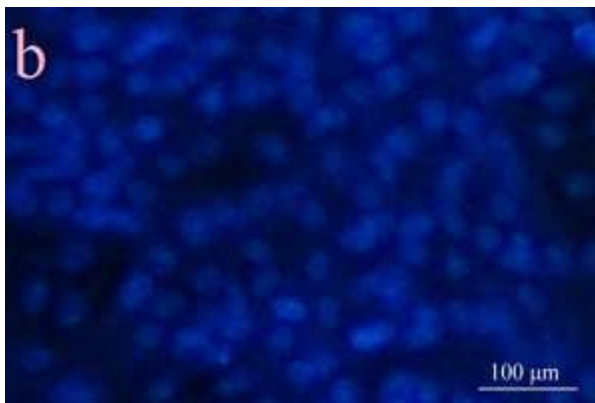


Figure 5. Fluorescent microscope images with DAPI staining of live cells a) TaN coating b) TaN-DLC coating.

4. CONCLUSION(S)

Based on the conducted research, the following can be concluded as the main findings:

1. By combining the two methods of sputtering and PACVD, it is possible to successfully produce a TaN-DLC bilayer coating on the SS316L substrate as a coating for an implant.
2. Both coating layers were formed uniformly with a surface roughness of $R_a = 3.14 \mu\text{m}$ for the interlayer TaN layer and $R_a = 1.02 \mu\text{m}$ for the top DLC layer, indicating a 67.5% reduction in surface roughness.
3. Both coating layers were formed with nanoscale quasi-spherical nodules.
4. The higher surface smoothness of the DLC coating was due to its much finer grains compared to TaN, as well as its wider amorphous range.
5. The DLC surface showed significantly better adhesion and cell growth for MG63 cells.

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