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

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ABSTRACT

In order to improve the properties and performance of SS316L implants in the current study, their surface was coated using two methods of sputtering and Plasma-Assisted Chemical Vapor Deposition (PACVD). To this end, 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 DLC formation. Atomic Force Microscopy (AFM) images indicated that the grain size was in the range of 20-35 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 confirmed the better performance of DLC than that of TaN. Thus, the TaN-DLC two-layer coating is introduced as a new coating that can be used to improve the performance of implants.

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1. INTRODUCTION

Medical implants are recognized as one of the important innovations in medical industry. These implants are used as the therapeutic artificial substitutes for patients who are in need for replacement of their body parts caused by 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 characterized by 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 replacement of defective or damaged bones in the human body [4]. While medical-grade stainless steels are generally 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 an important concern. In recent years, 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 than

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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 to use alternative materials such as polymeric and ceramic composites. These materials are highly resistant to wear that 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 enhance the 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 their traditional counterparts. 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]. Use of DLC coatings in biological implants proved to be quite useful due to several advantages such as their wear resistance, high load-bearing capacity, antibacterial properties, and ability to facilitate bone absorption. Additionally, these coatings can also act as a barrier to other biological materials and reduce their wear [12]. Therefore, their application as the biological coatings in medical implants is promising that can also improve the performance and longevity of implants. However, one of the drawbacks of the 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 metal surface and prevents the coating from separating from the metal surface, hence improved adhesion [16-17].

Wang et al. [18] demonstrated that adding a titanium carbide or nitride interlayer between steel and DLC film improved 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 use of duplex coatings significantly improved the tribological performance. In fact, the anti-wear performance of the duplex coatings is almost ten times higher than that of single-layer DLC films. In addition, duplex coatings have better resistance to damage than others. These improvements result from the combination of both mechanical and tribological properties of the interlayer. Wu's study [20] also showed that use of a Ti-C interlayer in a DLC film had a significant improvement in both wear resistance and friction coefficient. Moreover, this interlayer could reduce the scratch cracks and create greater adhesion. Overall, use of Ti-C interlayer helped improve the performance of the 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 the DLC coating in the absence of TaN interlayer as well as in the presence of TaN interlayer and to examine its biocompatibility.

2. MATERIALS AND METHODS

This research used SS316 stainless steel samples with the dimensions of 2x20x20 mm. Initially, the samples were grinded with sandpaper up to the number 5000 and then polished with 0.01-micrometer diamond paste for 30 minutes. The MSS160 RF magnetron sputtering device and a 99.999% pure Ta target were employed to coat the samples at the base pressure of 2.8×10^{-3} mbar, power of 185 W for 60 minutes, and temperature of 350 degrees Celsius with a 30Ar/10N2 gas mixture. Then, the samples were coated using the Hindivac MSPT12 PACVD system under the working pressure of 45 mTorr, power of 150 W, and deposition time of 60 minutes using a gas mixture of 3 Ar/1 C_2H_2.

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) at the power ranging from 10-90 mW, resolution of 6 cm^{-1}, and wavelength of 512 nm was used to investigate the bonding types and amounts.

In this study, the MG63 cells from the Pasteur Institute of Iran were prepared. The RPMI culture medium with 10% FBS serum was used for cell growth. The cells were then maintained in an incubator at 37 degrees of Celsius with the humidity of 95% and injected CO_2 level of 5%. The biocompatibility of each coating, cell survival rate, and growth were qualitatively tested using the MTT assay through which, the activity of the mitochondrial dehydrogenase enzymes in live cells was examined. In this test, the (MTT) solution is affected by the dehydrogenase enzymes present in the mitochondria of the cell and is converted into insoluble purple formazan. The amount of the produced formazan 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 estimate the number of live cells on the coatings.

3. RESULTS AND DISCUSSION

Figure 1 illustrates the 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 magnetron sputtering and PACVD methods. As evident in Figure 1(a), a uniform TaN coating with the thickness of about 700 nm was formed on the substrate. Given the nature of the magnetron sputtering process where nucleation occurs as a result of the ballistic atom bombardment towards the substrate, growth becomes universal, and there is no sign of the preferred growth [21]. In other words, the TaN coating grows uniformly on the underlying substrate.

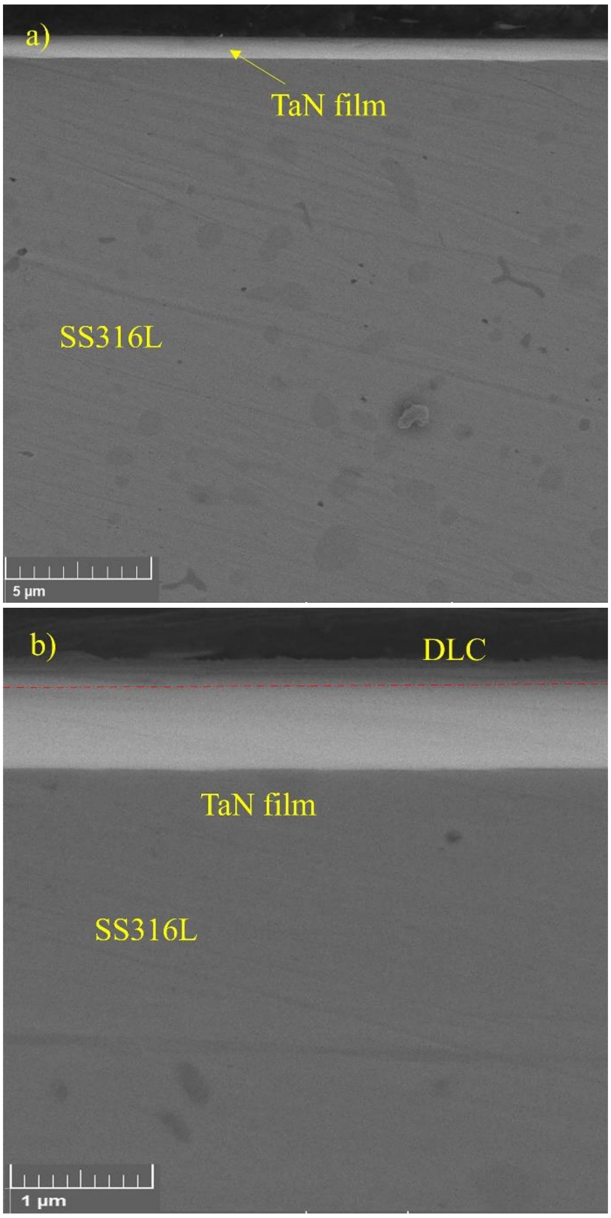

Figure 1. FESEM images of the cross-section surface of coatings: (a) TaN, and (b) TaN-DLC

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. Figure 1(b) shows the formation of a thin DLC layer with an approximate thickness of 150 nm on the interlayer of TaN. As observed in the high magnification of this figure, the upper layer of the coating was formed completely uniformly. Accordingly, the TaN-DLC bilayer coating was formed on the SS316L substrate.

Figure 2 shows the FESEM surface morphology of the 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].

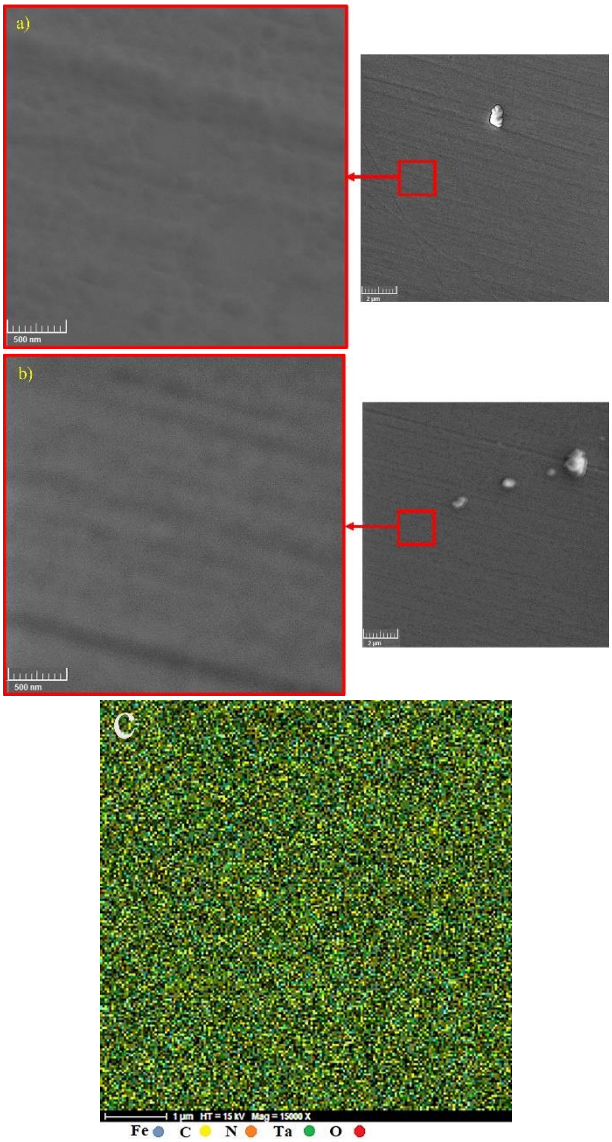

Figure 2. FESEM images of coating morphology: a) TaN, and b) TaN-DLC, and c) MAP of TaN-DLC.

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 demonstrated in Figure 2(a).

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. Figure 2(b) also shows the morphology of the TaN-DLC coating. In this sample, the coating is completely uniform and devoid of any defects. Generally, since the thickness value in thin film coatings is quite small, the morphology of the coating is affected by the topography of the substrate. In fact, the grooves resulting from grinding and polishing make the coating cones grow in parallel bands. Since the grinding and polishing effects were less observed in the preparation of the substrate in this study, TaN and consequently DLC coatings were formed completely smoothly.

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 as a result of which, all the pores and grooves are filled. A comparison between both samples in Figure 2 shows that the number of grooves or craters in samples with TaN-DLC bilayer coating is less than that with TaN coating. This issue has 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; this is the reason why their morphology appears in spherical or semi-spherical forms. However, followed by addition of C and N and formation of TaC or TaN, due to the high density of the resulting coating, there will be no sign of a columnar structure in the microscopical images of the cross-sectional surface. The FESEM images of the cross-sectional surface in Figure 1 can prove this statement.

In the present study, interstitial N atom in Ta atoms of dense coating causes an enhancement in the mechanical coating properties. On the contrary, incorporation of N into the system leads to a distortion in the lattice, which on the 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., 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, as already proven in previous studies [23,24]. The conditions needed for the formation of the TaN phase with long-range order cannot be provided by the combination of amorphous and crystalline phases. In other words, the non-observance state of the morphology of the coating results from several reasons such as the high volume of the amorphous phase, severe fineness, and removal of columnar structure, all indicating the formation of a dense coating.

The presence of both crystalline and amorphous phases in the coating creates a balance between softness and brittleness, hence an increase in the mechanical properties. Amorphous phases improve both corrosion resistance and toughness while crystalline phases enhance the hardness and strength. As the grain size and crystallite size decrease, the grain boundary suppression and mismatch increase, ultimately resulting in an increase in hardness [24]. Lack of coherence between the 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 facilitates the formation of larger clusters, as shown in Figure 2(b). This phenomenon is consistent with the results from the previous research [25]. The FESEM image in Figure b(2) shows the formation of very small grains in the nanometer order, which undoubtedly affects the roughness and mechanical properties of the coating.

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

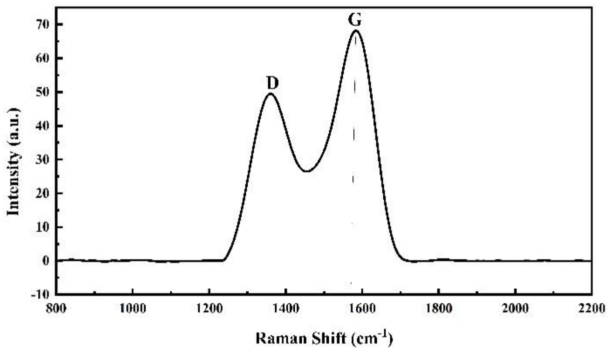

Figure 3. Raman spectroscopy pattern of the thin DLC film on the TaN interlayer

A prominent peak G can be detected in the graph in Figure 3 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 peak D also indicates the vibrational modes of sp^2 atoms in the ring structure. The position of the peak G is of great importance since it affects the I_D/I_G and sp^2/sp^3 ratios [28]. As the sp^2/sp^3 ratio increases, the position of the peak G also alters, compared to the ID/IG ratio in an amorphous structure. In general, the type of carbon obtained from ethylene prevents the growth of poly crystalline film and leads to the formation of amorphous DLC coating [29].

Upon reducing the value of peak G, the proportion of sp^3 linkages will change, and the breakage of graphite in the low coverage system will decrease. 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 Figure 2(b). Proper clustering increases the value of the peak G and consequently increases the disorder in the system by increasing the sp^3 content and causes the formation of an amorphous structure. This happens 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 value of peak D, 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. Once the sp^2 atoms are rearranged, the occurrence of a short-range order and stability of the amorphous structure can be ensured [31].

Figure 4 illustrates the AFM images of the two samples, i.e., TaN and TaN/DLC.

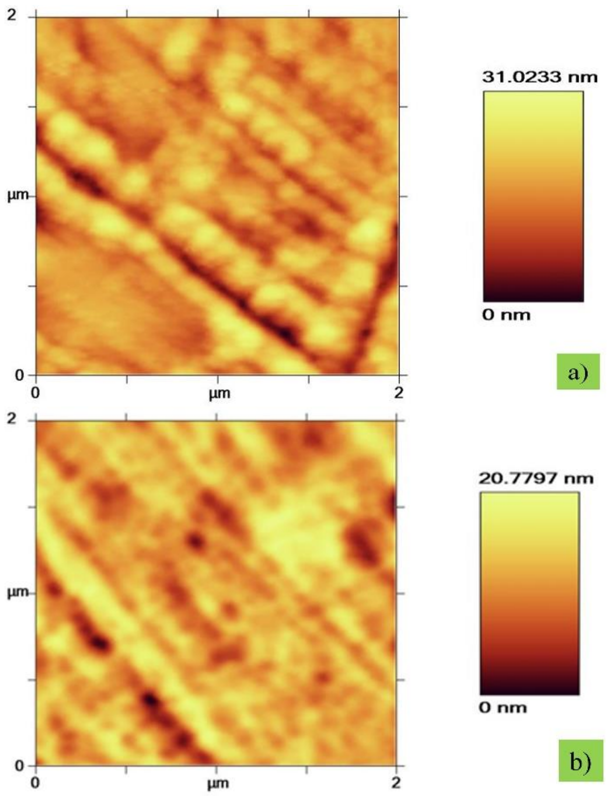

Figure 4. AFM images of coated samples: a) TaN, b) TaN-DLC

At the first glance, it is clear that the surface roughness can be reduced by applying the thin DLC layer. The grain size of TaN is about 30 nm with the surface roughness of Ra = 3.14 µm, which was later reduced by 21 nm and Ra = 1.02 µm after applying the DLC layer. The presence of the fine grains also leads to an increase in the strength and a decrease in the surface roughness mainly because the probability of obtaining the desired surface roughness is significantly reduced in the presence of uniform fine grains.

Figure 5 shows the adhesion, spreading, and morphology of the MG63 cells on two TaN and TaN-DLC samples after 48 hours of culture. As observed in this figure, the number of the remaining cells on the TaN-DLC sample is higher than that of 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 spread to the surface by filopodia which indicates a stronger cellular adhesion and better cell growth on the DLC layer than those of the TaN layer.

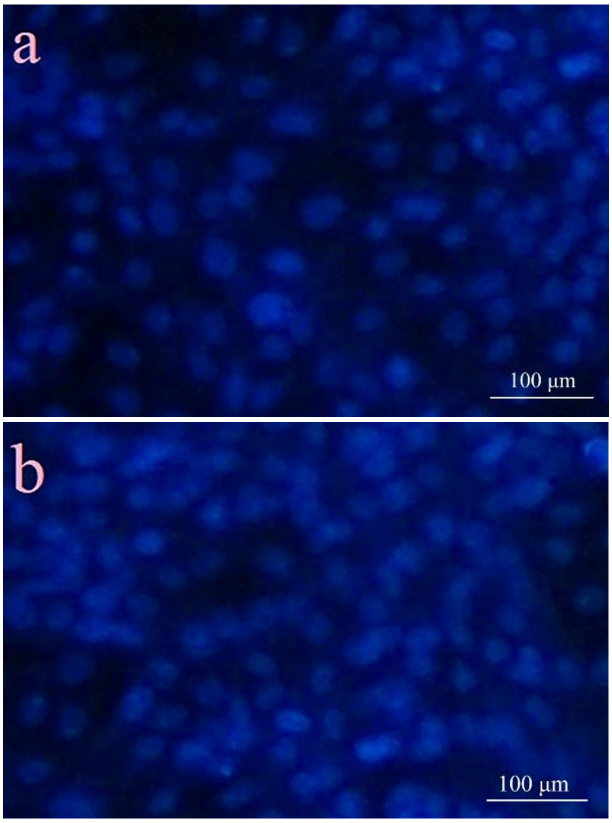

Figure 5. Fluorescent microscope images with DAPI staining of live cells a) TaN coating b) TaN-DLC coating

4. CONCLUSION(S)

The main findings of the current research are summarized in the following:

1. Once the two sputtering and PACVD methods are combined, it is possible to successfully produce a TaN-DLC bilayer coating on the SS316L substrate as a coating that can be used for an implant.
2. Both coating layers were formed uniformly with the surface roughness of Ra = 3.14 µm for the interlayer TaN layer and Ra = 1.02 µm for the top DLC layer, indicating a 67.5% reduction in the surface

roughness.
3. Both coating layers were formed with nanoscale quasi-spherical nodules.
4. The higher surface smoothness of the DLC coating resulted from its much finer grains as well as its wider amorphous range than those of TaN.
5. The DLC surface showed significantly better adhesion and cell growth for MG63 cells.

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