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THE STRUCTURAL PROPERTIES OF Ta DOPED DLC COATINGS FOR THE DENTAL APPLICATIONS USING HiPIMS

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ABSTRACT

Diamond-like Carbon (DLC) coatings are considered to be one of the most valuable engineering materials for various industrial applications, such as biomaterials and tools. Metallic and non-metallic elements and/or compound are added to the DLC coatings to develop the structures and properties (mechanical, tribological, etc.). In this study, Ta doped DLC was grown on Ti6Al4V alloys using High Power Impulse Magnetron Sputtering (HİPIMS)-Closed Field Unbalanced Magnetron Sputtering (CFUBMS) system. In the process, only the Ta target current was changed as 3A, 4A and 5A. XPS was used to determine the bond structure and percentage of the elements of Ta-DLC coatings. The microstructure and thickness of Ta-DLC coatings were examined by SEM. The structure of a-C with amorphous structure in Ta-DLC coatings was characterized by Raman. The internal stress in Ta-DLC coatings was analyzed using FTIR. The results showed that the microstructure of Ta-DLC was very dense and low internal stress.

INTRODUCTION

Diamond-like Carbon (DLC) coatings are considered one of the most valuable engineering materials for industrial applications such as manufacturing, transportation, biomaterials and microelectronics. DLC coatings are very attractive due to their high surface hardness, good friction behavior and high protection against wear. Its unique mechanical properties result from the proper optimization of the sp3/sp2 binding ratio, which can be controlled by the energy of carbon and gas ions coming to the surface of the growing film [1].

Metal-doped DLC (Me-DLC) coatings exhibit many properties, including low internal stress, high thermal stability and oxidation resistance, better toughness and tribological properties compared to pure DLC coatings. In addition, Me-DLC coatings offer some functional properties such as optical properties, magnetic properties and biological properties through metal atoms insert to the nano-structure formed in the carbon matrix [2].

Tantalum (Ta) has interesting properties for corrosion, heat and abrasion resistant protective coatings due to very high melting point, toughness and immunity to chemical attacks. In addition, the inertness of Ta in contact with body fluids and body tissues and excellent tissue compatibility leads to use in biomedical implants and orthopedics [3].

DLC and Me-DLC coatings can be grown using Closed Field Unbalanced Magnetron Sputtering (CFUBMS) method. In conventional magnetron sputtering processes, the energy supplied to a growing DLC coating is generally insufficient to achieve high sp3 binding rate. The main reason for this is the low ionization rate of the sputtered carbon atoms due to the low ion bombardment on the growing film and the high ionization potential of carbon atoms compared to other metals [1]. Recently, the development of the High Power Impulse Magnetron Sputtering (HiPIMS) process has attracted for the growth of hard DLC coatings [4]. The HiPIMS technique is capable of providing very high target peak current, high peak power density and high ionization rate than conventional radio frequency (RF), direct current (dc) and medium frequency (MF) magnetron sputter technologies. The HiPIMS process has many advantages in film deposition such as low processing temperature, good adhesion, superior film quality (high density and low surface roughness) [5-6].

HiPIMS is a PVD type that operates in low duty cycles (0.5-10%) and short high peak power (max 3 kW / cm2) [7-8]. In this method, the amount of ions in plasma increases as a result of the application of short positive pulses adjacent to the characteristics of the negative sputter pulses. In this way, sp3 content in DLC coatings has higher rates [1].

In this study, for use in dental implants the structural properties of Ta-DLC coatings grown with HiPIMS and CFUBMS were investigated.

EXPERIMENTAL DETAILS

The dental implant material Ti6Al4V alloy discs (ø20X3mm) were used as substrates. The substrates were etched in H_2SO_4 and HCl acids respectively and the surface roughness values were reached as Ra≈1-3 μm. Ta-DLC was grown on Ti6Al4V alloys using HiPIMS-CFUBMS system (Figure. 1). In the process, only the Ta target current was changed as 3A, 4A and 5A. The constant parameters are given in Table 1.

Table 1: The deposition parameters

Variable Parameters	**R1**	**R2**	**R3**
Ta target current (A)	3	4	5
Constant Parameters			
Frequency(Hz)	500		
C target voltage (-V)	900		
Working Pressure (Pa)	0.33		
C_2H_2 *amount (sccm)*	3		
Ti interlayer	Ti: 5A (10min)		
Ta-DLC deposition time (min)	90		
Substrate bias voltage (-V)	90		
Duty cycle (%)/Duty time (µs)	6/120		

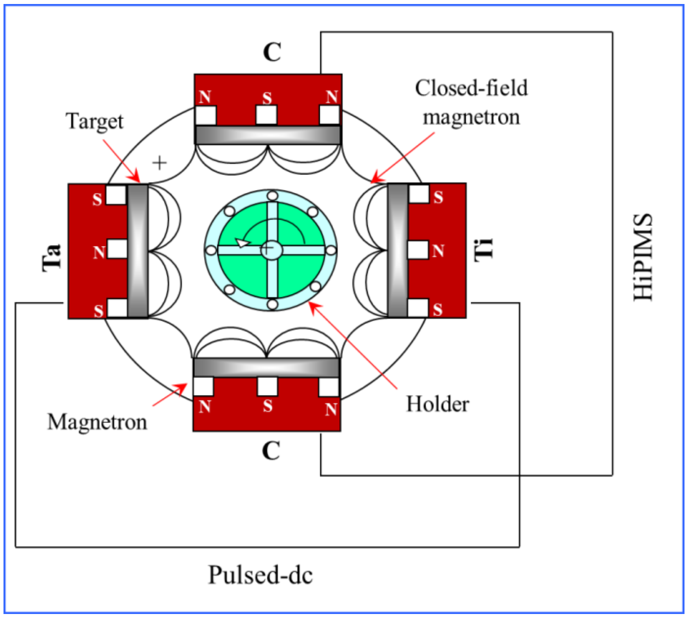

Figure 1: HiPIMS-CFUBMS system

The microstructure and thickness of Ta-DLC coatings were examined by SEM (ZEISS SİGMA 300). The Raman device (WITech alpha 300R) was used to determine the a-C structure having amorphous structure in Ta-DLC coatings. In addition, sp^2 / sp^3 ratios were calculated by Raman. FTIR (Bruker VERTEX 70v) and was used to detect C-C and C=C bonds and the shift peaks in Ta-DLC coatings.

RESULTS AND DISCUSSION

Cross-sectional SEM images of Ta-DLC coatings are given in Figure.2. The highest film thickness obtained from Ta-DLC coatings was 1.318 μm from the R3 film and the lowest film thickness was obtained from R1 film as 817 nm. The highest film thickness was obtained with the highest Ta target current (5A). Efeoglu et al. reported that Ta-DLC film thickness increases with increasing Ta target current [9]. Decho et al. stated that the film thickness increased with increasing duty cycle while keeping other parameters constant [10]. For this reason, Ta-DLC coatings are coated under high duty cycle.

FT-IR graphs of Ta-DLC coatings are given in Figure. 3. All coatings have a C-C and C = C bond. In the literature, it has been reported that as the shift in wave number increases, permanent internal tension increases for DLC coatings [11]. In this study, wave number increased with increasing Ta target current.

The results of RAMAN analyses show that all coatings have D and G bands (Figure. 4). The lower the D peak, the better the quality and performance of DLC coatings. The highest sp^3/sp^2 ratio was 0.527 in the R3 film and the lowest sp^3/sp^2 ratio was 0.471 in the R1 film [12-13].

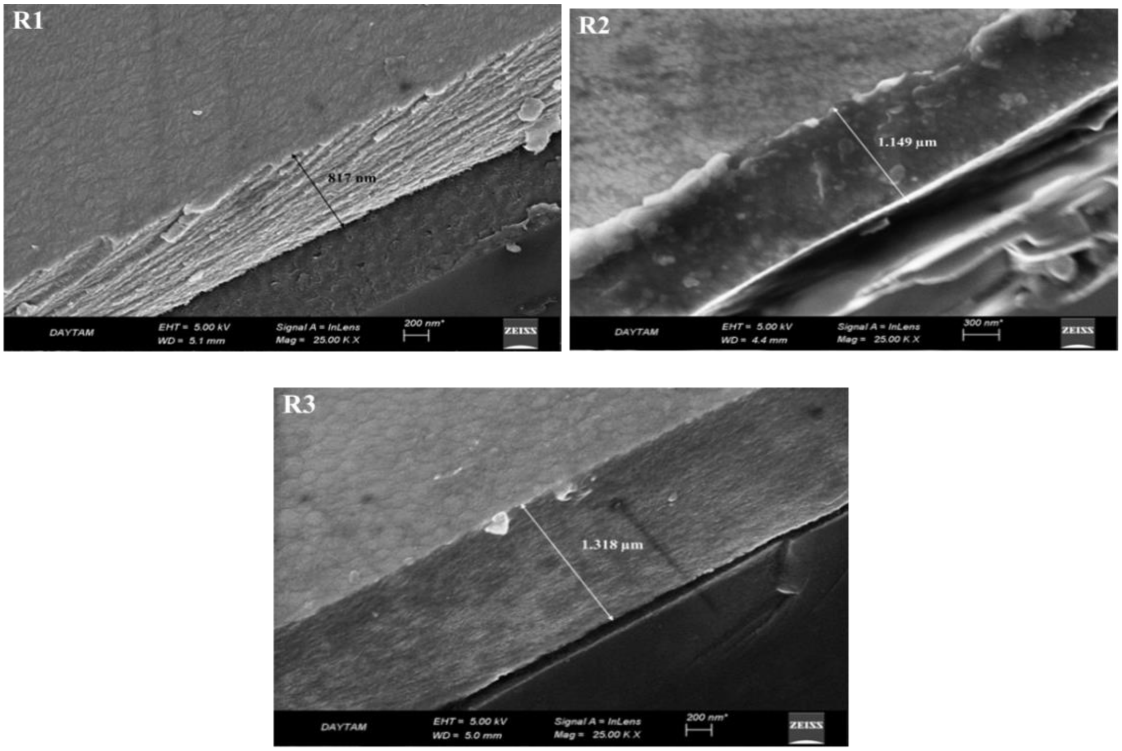

Figure 2: Cross-section SEM images of Ta-DLC deposited with R1, R2 and R3 parameters

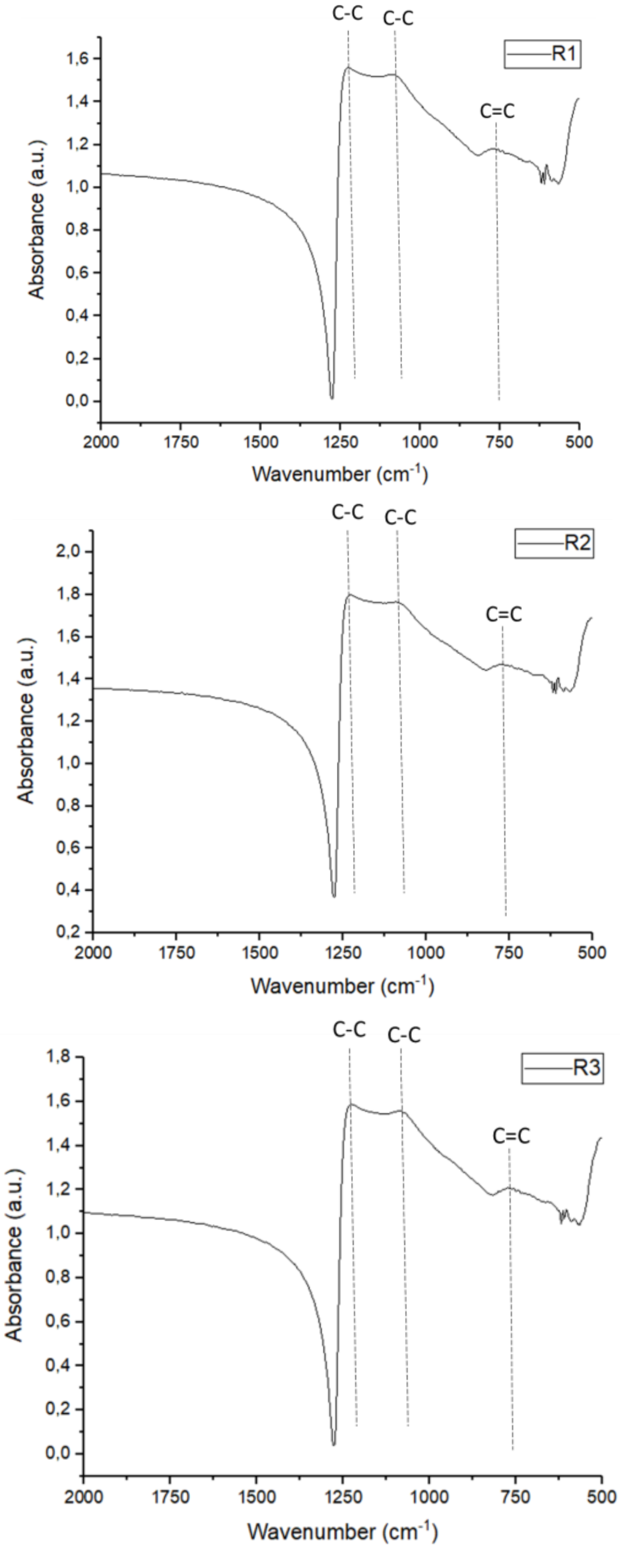

Figure 3: FT-IR graphs of Ta-DLC deposited with R1, R2 and R3 coating parameters

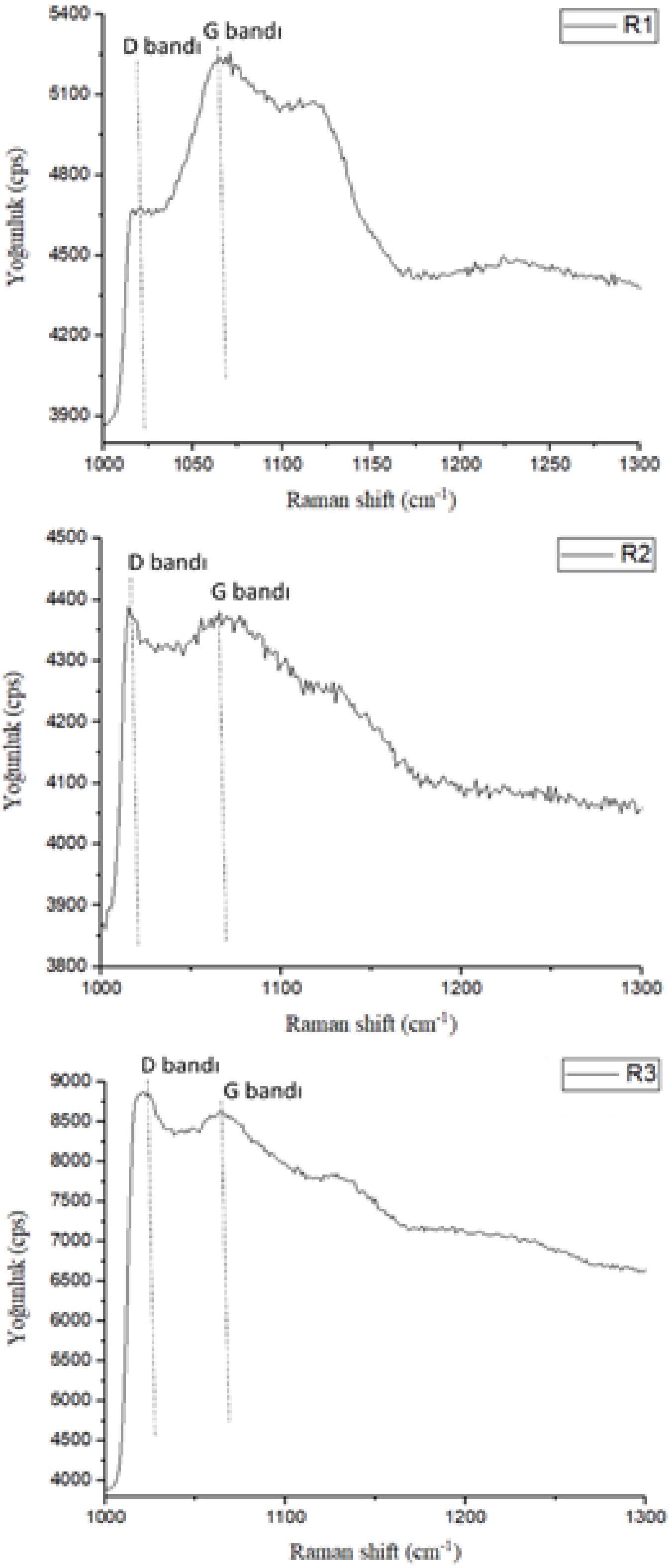

Figure 4: RAMAN graphs of Ta-DLC deposited with R3 coating parameters

CONCLUSION

The results of this article are given below:

- The highest film thickness obtained from Ta-DLC coatings was 1.318 μm from R3 parameter and the lowest film thickness was 817 nm from R1 parameter.
- The film thickness was affected directly proportional to the Ta target current.
- In the Raman graphics, it was observed that all coatings had D and G bands. These bands have an effect on the properties of coatings. The lower the D peak is the better performance. The highest sp^3 / sp^2 ratio was 0.527 in the R3 film and the lowest sp^3 / sp^2 ratio was 0.471 in the R1 film.
- C-C and C = C bonds in all coatings were synthesized according to FT-IR graphics
- It is known that C-C represents sp^3 and C = C represents sp^2 and is associated with mechanical properties and hardness.

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