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The effect of DLC-coating deposition method on the reliability and mechanical properties of abutment's screws

Dimorvan Bordin[a], *Paulo G. Coelho*[b,c], *Edmara T.P. Bergamo*[d], *Estevam A. Bonfante*[e], *Lukasz Witek*[b], *Altair A. Del Bel Cury*[d,*]

[a] *Department of Restorative Dentistry, University of Guarulhos, Guarulhos, SP, Brazil*
[b] *Department of Biomaterials and Biomimetics, New York University, College of Dentistry, New York, NY, USA*
[c] *Hansjörg Wyss Department of Plastic Surgery, NYU Langone Medical Center, 550 First Avenue, New York, NY 10016, NY, USA*
[d] *Department of Prosthodontics and Periodontology, Piracicaba Dental School, University of Campinas, Piracicaba, SP, Brazil*
[e] *Department of Prosthodontics and Periodontology, University of São Paulo - Bauru School of Dentistry, Bauru, SP, Brazil*

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ABSTRACT

Objective. To characterize the mechanical properties of different coating methods of DLC (diamond-like carbon) onto dental implant abutment screws, and their effect on the probability of survival (reliability).

Methods. Seventy-five abutment screws were allocated into three groups according to the coating method: control (no coating); UMS – DLC applied through unbalanced magnetron sputtering; RFPA-DLC applied through radio frequency plasma-activated ($n = 25$/group). Twelve screws ($n = 4$) were used to determine the hardness and Young's modulus (YM). A 3D finite element model composed of titanium substrate, DLC-layer and a counterpart were constructed. The deformation (μm) and shear stress (MPa) were calculated. The remaining screws of each group were torqued into external hexagon abutments and subjected to step-stress accelerated life-testing (SSALT) ($n = 21$/group). The probability Weibull curves and reliability (probability survival) were calculated considering the mission of 100, 150 and 200 N at 50,000 and 100,000 cycles.

Results. DLC-coated experimental groups evidenced higher hardness than control ($p < 0.05$). *In silico* analysis depicted that the higher the surface Young's modulus, the higher the shear stress. Control and RFPA showed $\beta < 1$, indicating that failures were attributed to materials strength; UMS showed $\beta > 1$ indicating that fatigue contributed to failure. High reliability was depicted at a mission of 100 N. At 200 N a significant decrease in reliability was detected for all groups (ranging from 39% to 66%). No significant difference was observed among groups regardless of mission. Screw fracture was the chief failure mode.

* *Corresponding author at*: Department of Prosthodontic and Periodontology, Piracicaba Dental School, Avenida Limeira, 901, Areião, Piracicaba, SP 13414-018, Brazil. Tel.: +55 19 2106 5294.
E-mail address: altair@unicamp.br (A.A. Del Bel Cury).

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Significance. DLC-coating have been used to improve titanium's mechanical properties and increase the reliability of dental implant-supported restorations.

1. Introduction

When prosthesis service involves sliding between metallic surfaces, as occurs in implant-abutment-screw contact area, severe wear (known as galling) may occur. Under loading conditions, materials with lower Young's modulus adheres locally to the contacting opposite surface and forms hard "galls" where wear initiates [1,2]. Any force that causes slippage between threads might reduce preload, which is supposed to maintain the joint clamped, potentially leading to screw failure [3]. Clinical findings have reported screw failure incidences of 10.8% [4] for single restorations over the years. This complication is highly undesirable as it involves additional clinical appointments and, consequently, increases treatment costs.

Diamond-like carbon (DLC) coating has been extensively indicated to coat biomedical components since it improves a wide range of surface goods without compromising their bulk properties [5]. Concerning implant-supported prostheses, the rationale behind DLC-coating indication lies on its biocompatibility, high hardness, wear resistance, and low friction coefficient [6,7] which contributes to an improved preload [6,8,9] and helps to avoid the screw's thread wear due to micromotion [9]. Additionally, its smooth surface finishing acts as a protective barrier that prevents surface wear debris and toxic elements release [10–14].

Nonetheless, the literature seems to be contradictory regarding the performance of DLC-coating when applied to implant dentistry. Some studies have reported improved mechanical behavior such as higher loosening torque [6,8,9] and wear resistance [9] of DLC-coated screws, while others have reported no significant difference between coated *vs.* non-coated ones for instance in probability of survival under fatigue tests [7,15–17]. These inconsistent results rely on film properties, such as adhesion characteristics, hydrogen content and density, which seems to be directly dependent on carbon coating method [18,19]. Carbon form has a great variety of crystalline structures due to its three hybridization forms: sp^1, sp^2, and sp^3. As expected, an increased surface hardness and Young's modulus correlate with an increased sp^3 percentage and a decreased hydrogen content since its configuration is similar to diamond. In contrast, an increased sp^2 content provides a similar behavior to pure graphite which is less desired for high-load demanded components [20]. Additionally, mechanical properties rely on substrate temperature during DLC coating and can be commonly improved with increased ion energy per condensed carbon atom according to the coating method [20].

The unbalanced magnetron sputtering (UMS) is a typical and controllable method to produce hydrogen-free films sputtering pure graphite targets using an Argon plasma source through physical vapor deposition (PVD) [21]. Magnetos are used in order to increase the ionization degree of plasma and increase PVC deposition rate on the substrate [21]. On the other hand, radio-frequency plasma activated (RFPA) is a chemical vapor deposition (CVD) method which involves the decomposition of a selected precursor gas, such as methane, ethane, ethylene, acetylene, *etc.*, at high temperature and low pressure to improve the ion radical fraction. The energy for the chemical reaction is provided by plasma, usually created by radio frequency between two electrodes in a chamber filled with the reacting gases [21,22].

Considering the strength degradation of implant-abutment-screw-prosthesis system in function and the fact the DLC-coating enhances material's mechanical properties, the present study conducted a coating mechanical characterization by nanoindentation and also, a finite element analysis to study the stress distribution behavior under loading; additionally, a step-stress accelerated life-testing (SSALT) was performed to evaluate the probability of survival (reliability) and failure mode of DLC-coated screws through different coating methods (UMS and RFPA).

The postulated hypotheses were: (1) DLC-coating methods would equally improve the mechanical properties of coated screws, compared to uncoated controls in both *in silico* analysis and nanoindentation testing. (2) Crowns cemented to implant abutments torqued with DLC-coated screws would present higher probability of survival than uncoated controls when subjected to fatigue (SSALT).

2. Materials and methods

2.1. Experimental design

Seventy-five abutment's screws were allocated into three groups according to DLC-coating method (n = 21/group), Uncoated (Control); DLC applied by radio frequency plasma-activated method (RFPA) and DLC applied by unbalanced magnetron sputtering method (UMS).

2.2. Nanoindentation testing (in vitro)

Twelve abutment's screws (n = 4 per group) were embedded into acrylic resin and prepared for nanoindentation testing by grinding (400–1200 grit SiC abrasive papers) and polishing (diamond suspension solutions of 9–1 μm particle size) under water irrigation through a grinding machine (Metaserv 3000, Buehler, Lake Bluff, IL, USA).

Nanoindentation was performed using a triboindenter (TI 950, Hysitron, Minneapolis, MN, USA) mounted with a Berkovich diamond three-sided pyramid probe. A loading profile was designed after a pilot test. A peak load of 4000 μN was applied, followed by 10 s of holding and 3 s for unloading the probe. For each specimen, 10 nanoindentations were

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