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Graphene Oxide Modified Anodic Ternary Nanobioceramics on Ti6Al7Nb Alloy for Orthopedic and Dental Applications

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Abstract

With increased demands of artificial bone-like tissue specified orthopedic and dental replacement, medical grade of titanium alloys such as Ti6Al7Nb implants (Ti67IMP) have been considered due to their specific density and corrosion resistance with relatively lower moduli than other metals which leads to a better compliance with the modulus of bone. However, without any external force and overloading, the failure might be happen after implantation which need to repeat the surgery step. Thus, in order to minimize the risk of implant loosing and improve biocompatibility, surface modification is required to facilitate the stability of implant trough healing process. The development of nanotubular bioceramics can improve the implants' surface properties and provide rapid osseointegration. It is notified that the considered nanobiomaterials with controlled morphology can effect on antibacterial and drug delivery activities of implants. Herein, mixed oxide nanotubes (titania-niobia-alumina) was fabricated on Ti67IMP using PVD magnetron sputtering and subsequent electrochemical anodization. Initially, a well-adherent niobium (Nb) film was PVD sputtered under optimized coating conditions, then nanotubular arrays were grown on Nb/Ti67IMP surface after subsequent anodization and thermal treatment. In the final stage, the as-prepared graphene oxide (GO) nanosheet was transparently loaded on anodic nanotubes to reinforce ternary ceramic film. The microstructural features, wettability behavior and *in-vitro* bioactivity of the nanostructured coating were examined. Based on *in-vitro* bioassay analysis, a thick apatite layer was formed on the implant surface after primary days of immersion in simulated body fluid (SBF).

Keywords: Nanoceramics; Graphene oxide; Ti6Al7Nb alloy; Wettability; Orthopedic and dental implnats.

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1. Introduction

Generally, among metallic implant materials, clinical titanium (Ti) alloys are widely considered due to their excellent mechanical properties, biocompatibility and corrosion resistance. However, this class of biomaterials are still not able to meet all required characteristics for long term replacement. Thus, surface modification through various coating treatments can be applied to enhance properties of implantable devises. The fabrication of bioceramic films leads to the provision of the reasonable stability of artificial tissues and promote rapid osseointegration after surgery [1–5].

To date, several researches have been conducted on the surface treatments of Ti alloys via various physical and chemical coating methods such as plasma electrolytic oxidation (PEO) [6], chemical vapor deposition (CVD) [7], thermal spraying technique [8], physical vapor deposition (PVD) [9,10], and electrochemical anodization [11–13]. Among the common techniques, PVD magnetron sputtering is matched to deposit both metallic and ceramic films on different substrates without care of conductivity aspects and number of superlattice interfaces. This method was investigated through parametric conditions and optimized well-adherent outputs. Moreover, artificial intelligent models were proposed to formulate and predict PVD variables to obtain the preferable coatings properties in minimum experiments [14]. On the other hand, the preparation of anodic nanotubular configurations has been considered as the most influential process to improve the biofunctionality of the metallic implants [15–17]. For instance, it has been reported that TiO_2 nanotubes (TiO_2 NTs) provide higher *in-vitro* corrosion resistance as compared to the smooth Ti [18,19]. Duo to the influence of phase compositions on the performance of implant materials, many studies have been conducted to examine the effects of thermal treatment on the crystallization and *in-vitro* bioactivity of TiO_2 NTs in SBF [20]. In addition to Ti and its alloys, the self-assembly electrochemical anodization could be caused for a wide range of metals like vanadium, zirconium, hafnium, niobium, tantalum, tungsten or alloys. They are also capable of forming well-arranged nanostructured coatings for orthopedic and dental implant devices [21–23]. Considering the high capability of carbon-based materials in medical implant applications, many studies have been directed to investigate the effects of the use of carbon nanostructures (CNSs) especially graphene in orthopedic applications [24–26]. Accordingly, the fabrication of hybrid composites of nanotubular arrays and functionalized graphene such as GO could be a new phase of future developments in the field of nanomedicine industry.

To the best of our knowledge, there is no any obvious report regarding to the fabrication of multilayered GO/ternary ceramics nanocomposite. Thus, in the present study, a hybrid approach was adopted to produce GO-modified mixed oxide nanotubes on Ti67IMP using PVD magnetron sputtering and subsequent electrochemical anodization. The phase composition, morphological properties, surface wettability, and *in-vitro* bioactivity of the nanostructured coating were investigated.

2. Experimental procedure

2.1. Sample Preparation

SPD-processed Ti67IMP plates (Baoji Liu Wei special Material and Equipment produce Co. Ltd China) were wire-cutted to dimension of $20 \times 10 \times 2$ mm^3 and were utilized as substrate during all coating experiments. Prior to the sputtering process and growing ternary oxide nanostructures, all specimens were ground using SiC emery papers (1000 to 2400 grit) and polished in diamond slurry for mirror-like shiny view. After that, the polished surfaces were sonicated at 50 °C in acetone for 10 min, wash with deionized water and dried at 100 °C for 90 min.

2.2. Deposition of Nb Layer

The clean polished Ti67 was put to PVD sample holder to sputter well-adhered Nb thin film on implant surface under optimized parametric condition (DC power: 350 W, bias: 90 V, argon flow rate: 20 sccm) [27]. The PVD coating machine model SG control engineering Pte Ltd series equipped with pure Nb target (Lesker Company) was utilized in this work. During the sputtering process, the fixed distance of 150 mm was applied between the substrate and target. Ti67 specimens preheated with digital heater to 350 °C for 20 min before deposition. The argon atmosphere was utilized in this approach to eliminate the passive oxide layer. The PVD chamber was highly vacuumed to reach the decrease pressure below 2.0×10^{-5} Torr. The coating process was done at temperature of 220 °C for 120 min.