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Metal Release of Multilayer Coatings by Physical Vapour Deposition (PVD)

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Abstract

Orthopaedic implants are mostly fabricated by Stainless Steel, Titanium, and Ni-Cr Alloys that consist of Chromium (Cr), Cobalt (Co), Nickel (Ni) and Molybdenum (Mo). 10-15% of population are affected by metals (Ni, Co, Cr, Mo) hypersensitivity reaction. Multilayer coating by Physical Vapour Deposition (PVD) has been applied onto orthopedic implants to prevent metallic ions leaching into human body. This paper aims to study the metal ions leaching of Cr, Ni, Mo, and Co from multilayer coatings of Chromium (Cr), Chromium Nitride (CrN), Chromium Carbonitride (CrCN) and Zirconium Nitride (ZrN) by Physical Vapour Deposition (PVD). Cr, CrN, CrCN and ZrN have been successfully deposited onto stainless steel substrates by CAPVD. XRD analysis detected major peak in preferred orientation of (200) and other peaks with (111), (220), (311) for CrN cubic phase. For CrCN, XRD analysis detected only low intensity peaks of Cr₇C₃ and ZrN peaks with preferred orientation of (111), (200) with other peak (220), (311) and (222). A seven days metal released test by ICP-MS showed that generally all ions concentration for Ni, Co, Mo decreased from uncoated substrate to multilayer coatings except for Cr. Metal released showed higher concentration for coatings deposition with longer deposition time at 10 minutes.

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1. Introduction

Orthopaedic implants are mostly fabricated by Stainless Steel, Titanium, and Ni-Cr Alloys that consist of Chromium (Cr), Cobalt (Co), Nickel (Ni) and Molybdenum (Mo). Almost 10-15% of population are affected by metal hypersensitivity reaction, especially for Ni and followed by Co, Cr and Mo, with 22% from well-functioning implants and 60% from poorly-functioning implants. This resulting in many studies on metal ion release especially on metal-on-metal bearing hip joints made of Co–Cr–Mo alloys [1-5]. Metal ions leaching into human fluid are due to several mechanisms of corrosion such as fretting corrosion, stress corrosion and fatigue corrosion [1,2]. It has been reported concern for patients with orthopaedic implants of released metal ions level in their blood and urine [3,4].

A multilayer coating was deposited onto metal implants to prevent the leaching of metallic ions into human body. The multilayer coating will be developed to reduce the hardness from top to bottom in a gradient way to improve elastic modulus coating and extremely stable against mechanical stresses and strains and improved adhesion [6,7]. Zirconium (Zr) with excellent biocompatibility, chemical inertness and ceramic thin films of Zirconium Nitride (ZrN) with superior tribological properties with high wear resistance and low gliding friction had attracted researchers in biomedical application [8]. Chromium based coatings had been used extensively due to their excellence in corrosion resistance, hardness, as well as in bridging the differences in hardness and residual stress between softer base material and hard top coating of ZrN [9].

This paper aims to study the metal ions (Cr, Ni, Mo, Co) release of multilayer coatings of Chromium (Cr), Chromium Nitride (CrN), Chromium Carbonitride (CrCN) and Zirconium Nitride (ZrN) by Physical Vapour Deposition (PVD).

2. Experimental

2.1. Coating Deposition

The substrates were all ultrasonically cleaned in ethanol for 10 minutes to remove dirt, grease and residue from production process, followed by grounded and polished to mirror-finished. Substrates were then rinsed in deionised water and dried. Two sets of five minutes and ten minutes of deposition duration thin films were deposited by Physical Vapor Deposition (PVD) in the Argon (Ar), Nitrogen (N₂) and Methane (CH₄) gases on both sides of substrate. The geometry of multilayer coating are shown in Table 1.

Table 1. Coatings geometry.

Coating (Label)	Coating Arrangement
Chromium (Cr)	Substrate/Cr
Chromium Nitride (CrN)	Substrate/Cr/CrN
Chromium Carbonitride (CrCN)	Substrate/Cr/CrN/CrCN
Multilayer, 5 layers (ML5)	Substrate/Cr/CrN/CrCN/CrN/ZrN
Multilayer, 7 layers (ML7)	Subs./Cr/CrN/CrCN/CrN/CrCN/CrN/ZrN

2.2. Characterisation

The microstructure of the sample was analysed by Scanning Electron Microscopy (SEM). The compositions and phases present in the samples were analysed by using XRD in glancing incidence angle and Energy Dispersive X-ray (EDX). The metal release test was done by static immersion test according to JIS T 0304. Metal ions concentration was measured by Inductively Coupled Plasma Mass Spectrometry (ICP-MS). Three test coupons from each sample (Uncoated, Chromium coated, Chromium Nitride coated, Chromium Carbonitride coated, Multilayer) were immersed in 50 ml Simulated Body Fluid (SBF) in 37°C for seven days.

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