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Effects of working gas pressure on zirconium dioxide thin film prepared by pulsed plasma deposition: roughness, wettability, friction and wear characteristics

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ABSTRACT

In joint arthroplasty one of the main issues related to the failure of prosthetic implants is due to the wear of the ultra-high molecular weight polyethylene (UHMWPE) component. Surface treatments and coatings have been recognized as enhancing methods, able to improve the tribological properties of the implants. Therefore, the main objective of this work was to investigate the possibility to fabricate yttria-stabilized zirconia (YSZ) coatings on a metal (AISI 316-L) substrate by means of Pulsed Electron Deposition, in order to improve the tribological behavior of the polymer-metal coupling, by reducing the initial wear of the UHMWPE component. In order to optimize the coating characteristics, the effects of working gas pressure on both its morphological and tribological properties were analyzed. Morphological characterization of the films was evaluated by Atomic Force Microscopy (AFM). Coating wettability was also estimated by contact angle (CA) measurement. Tribological performance (coupling friction and wear of UHMWPE) was evaluated by using a ball-on-disc tribometer during highly-stressing tests in dry and lubricated (i.e. NaCl and serum) conditions; friction and wear were specifically evaluated at the initial sliding distances - to highlight the main effect of coating morphology - and after 100 m - where the influence of the intrinsic materials properties prevails. AFM analysis highlighted that the working pressure heavily affected the morphological characteristics of the realized films. The wettability of the coating at the highest and lowest deposition pressures (CA ~ 60°, closed to substrate value) decreased for intermediate pressures, reaching a maximum CA of ~ 90°. Regarding tribological tests, a strong correlation was found in the initial steps between friction coefficient and wettability, which decreased as the distance increased. Concerning UHMWPE wear associated to coated counterpart, at 100 m a reduction rate of about 7% in dry, 12% in NaCl and 5% in presence of serum was obtained compared to the uncoated counterpart. Differently from what highlighted for friction, no correlation was found between wear rate and morphological parameters. These findings, in agreement with literature, underlined the effect of the deposition pressure on the morphological properties, but suggested that physical characteristics are influenced too. Further research on the deposition process will be required in order to improve the tribological performance of the coating at long distances, addressing – above all - orthopedic applications.

1. Introduction

Surgical replacement currently represents a very common approach for osteoarthritic joints with a really good rate of success (Di Puccio and Mattei, 2015). However, concerning this procedure, the wear phenomenon between the mating surfaces still represent a main issue. This critical occurrence implies debris development, mass loss, geometrical changes of the components and, definitely, leads to the failure of the implant itself (Di Puccio and Mattei, 2015; Ingham and Fisher, 2005). The aseptic loosening could happen 10-20 years after the surgery, depending on patient's level of activity, body weight, quantity and quality of synovial fluid, loads history, anatomical location, surgery, design and materials used to realize the prosthesis itself (Ching et al., 2014).

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So far, several approaches have been proposed to reduce wear rate, improve the durability of the implant and delay the need of surgical revision. The most widespread solutions exploit the use of metal- or ceramic-on-plastic coupling (Proffen et al., 2013), with the latter representing the best compromise in terms of wear rate and friction coefficient, despite the high brittleness (Rahaman et al., 2007). Recently, surface engineering was approached with the development of highly adherent and wear resistant coatings, considering - above all - ceramics and diamond-like carbon solutions (Lappalainen and Santavirta, 2005). Coatings could indeed enhance the surface characteristics without changing the inherent properties of the underlying bulk materials (Ching et al., 2014).

In this perspective, several coating methodologies have been proposed, including physical (PVD) (Hendry, 2001) and chemical vapor deposition (CVD) techniques (Ching et al., 2014). In order to obtain optimal performances in terms of overall (i.e. abrasive, fatigue and corrosive) wear, it is mandatory to well define the coating properties throughout the deposition process, considering both morphological and structural characteristics (Ching et al., 2014) and without neglecting biocompatibility (Goodman et al., 2013).

However, correctly planning coating characteristics is a tough multi-parametric problem, even just considering the wear issue. Friction and wear rates depend in fact by both mechanical and chemical interactions that occur within the coupling (Huang et al., 2015). Several tribological analyses addressing biological application demonstrated that the degree of wetting of a lubricant - mimicking in vivo conditions - can influence the overall friction and wear performance (Kwok et al., 2005; Gispert et al., 2006). Therefore, to achieve optimal tribological efficiency, it is necessary to finely control the lubrication effect. More in details, the wettability of engineered surfaces in static and dynamic conditions depends on the lubricant chemical properties, but it can be strongly influenced by surface roughness itself (Kubiak and Mathi, 2009; Kubiak et al., 2011). One aspect of dynamic lubrication theory is in fact the assumption that the lubricant - moving at the same velocity of the surfaces, which it is in contact with (Reynolds, 1886) – could carry much of the load, through hydrodynamic pressure. However, if the surfaces are hydrophobic, water-based lubricants have difficulty in attaching to them and so hydrodynamic action is less likely to occur (Curran et al., 2013). Wettability of the components towards a specific lubricant can be quantified in terms of the apparent contact angle (Kubiak et al., 2011) and can be correlated to the surface roughness through multi-scale models (Belaud et al., 2015). Regarding surface roughness, recent works underlined the possibility to change the “ultra-smooth” paradigm in approaching wear issue with the analysis of surface patterns and micro texturing, which work on increasing the contact load capacity and the thickness of lubricant film, eventually reducing the friction in the coupling itself (Menezes and Kailas, 2016).

In this perspective, the possibility to coat bulk materials theoretically allows to work on all these factors without influencing the basic characteristics of the underlying materials and thence enhancing the overall wear behavior of the prosthesis coupling. Recently, among different solutions, Pulsed Electron Deposition (PED) technique has demonstrated the possibility to realize ceramic thin films on metal substrates (Boi et al. 2015). Additionally, adhesion, proliferation and viability of mesenchymal stem and osteoblast cells were observed, suggesting good biocompatibility of the nanostructured zirconia films (Bianchi et al., 2016). Structural and compositional characterization of the coatings have highlighted a crystalline structure with cubic phase and impurities lower than 1% (Bianchi et al. 2013).

However, an extended study of the influence of deposition parameters on morphology and wear behavior is still lacking. Addressing this issue, the present work evaluates the effects of working gas pressure on the behavior of yttria-stabilized zirconia (YSZ) films, particularly at morphological level. Afterwards, the potential benefits of realizing a coating onto AISI 316-L components were analyzed, in terms of tribological performance in coupling with ultra-high molecular weight polyethylene (UHMWPE), with the aim to reduce the wear rate of the polymeric component in the very first stages of sliding contact. More in details, thin film morphology was characterized by thickness, roughness (RMS) and contact angle (CA) analyses;.

Furthermore, tribological performance of coated and uncoated components was evaluated against UHMWPE at short distances by using a ball-on-disc tribometer and considering both dry and wet conditions (i.e saline solution and bovine serum).

2. Experimental

2.1. Materials

Yttria-stabilized zirconia (YSZ) coatings were deposited by ablating a tetragonal zirconia (zirconium dioxide) target, stabilized with 3 mol% Y_2O_3. Cylindrical targets (Ø ~ 3 cm, thickness ~ 0.7 cm) were produced starting from pure 3Y-TZP powder (Tosoh, Tokyo, Japan), by cold isostatic pressing (30 MPa, 60 s) and sintering at 1500 °C for 1 h (process performed by ISTEC-CNR, Faenza, Italy).

YSZ coatings were deposited on AISI 316-L balls (Ø = 6.00 ± 0.00 mm, H_i = 0.85 ÷ 1.22 GPa, E_i = 200 GPa, Ra = 0.1 µm, Grade 100, R.G.P. International S.r.l, Milan, Italy) for tribological tests. AISI 316-L flat samples (29 × 29 mm, thickness ~ 6 mm, Ra = 1.92 ± 0.06 µm, CITIEFFE Srl, Bologna, Italy) were used as a substrate for CA measurements. Silicon wafers (p-type doped mono-crystalline (100) native silicon, size 20 × 20 mm, thickness 3 mm, Fondazione Bruno Kessler, Trento, Italy) were used as substrates for morphological characterizations. UHMWPE disc samples (radius ~ 28.5 mm, thickness ~ 11.3 mm, Ra = 3.575 ± 0.279 µm, International Standard ASTM F-648, ISO-5834-1/2, CITIEFFE srl, Bologna, Italy) were used for the tribological analysis.

Prior to deposition and tests, all samples were ultrasonically cleaned in serial baths of water, isopropanol and acetone (10 min each) and dried under N_2 flux.

2.2. Coating deposition

The technique used to realize YSZ coatings was a Pulsed Electron Deposition (PED) process based on a commercial Pulsed Plasma Deposition gun (Gen III Advance Electron Gun, Organic Spintronics srl, Bologna, Italy). The deposition was achieved through the ablation of the target material by a fast pulse (100 ns) of high energy (10 J/cm^2) and high density (108 W/cm^2) electrons. The ablated material was then directed toward the substrate located at a suitable distance from the target (Bianchi et al., 2015). The substrate was mounted on a rotating sample holder specifically positioned at a distance of 65 mm from the rotating target surface. The distance between the surface of the target and the tip of the quartz tube (length ~ 10.5 cm, inner diameter ~ 3 mm, external diameter ~ 6 mm) was ~ 1 mm. The function of the quartz tube is mainly related to its role in the ablation of the target: it is specifically necessary for the channeling and the addressing of the high density electron beam. The vacuum chamber was initially evacuated down to a base pressure of 1.0×10^{-7} mbar by a turbomolecular pump (EXT255H, Edwards, Crawley, England) and then raised by controlled flow of oxygen (purity level = 99.999%) to the following working pressures: 1.5, 4, 6, 8 and 10 × 10^{-3} mbar. The working voltage, current and frequency were set at 17 kV, 3 mA and 5 Hz respectively, for a total deposition time of about 8 h. The deposition was performed without heating the substrate. The deposition parameters were adjusted in order to obtain an uninterrupted plasma plume and a suitable deposition rate (250 nm/h).

The purpose is to investigate the effect of deposition pressure on the morphology and tribological behavior of the coatings, maintaining constant the other deposition parameters (i.e voltage, frequency, distance and deposition time).