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The effects of parametric changes in electropolishing process on surface properties of 316L stainless steel

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ABSTRACT

Corrosion resistance and biocompatibility of 316L stainless steel implants depend on the surface features and the nature of the passive film. The influence of electropolishing on the surface topography, surface free energy and surface chemistry was determined by atomic force microscopy, contact angle meter and X-ray photoelectron spectroscopy, respectively. The electropolishing of 316L stainless steel was conducted at the oxygen evolution potential (EPO) and below the oxygen evolution potential (EPBO). Compared to mechanically polished (MP) and EPO, the EPBO sample depicted lower surface roughness (Ra = 6.07 nm) and smaller surface free energy (44.21 mJ/m^2). The relatively lower corrosion rate (0.484 mpy) and smaller passive current density (0.619 μA/cm^2) as determined from cyclic polarization scans was found to be related with the presence of OH, Cr(III), Fe(0), Fe(II) and Fe(III) species at the surface. These species assured the existence of relatively uniform passive oxide film over EPBO surface. Moreover, the relatively large charge transfer (R_{ct}) and passive film resistance (R_f) registered by EPBO sample from impedance spectroscopy analysis confirmed its better electrochemical performance. The in vitro response of these polished samples toward MC3T3 pre-osteoblast cell proliferation was determined to be directly related with their surface and electrochemical properties.

1. Introduction

Stainless steel is extensively used in various industrial applications such as automotive parts, in semiconductor industry and biomedical devices [1–3]. The low-carbon stainless steel is considered biocompatible material and is mainly used in many orthopedic devices and cardiovascular implants [4]. However, the mechanical stability and corrosion resistance of 316L are the paramount concerns for long-term applications. The in vivo utilization of this material therefore demands high quality surface, structural and biological properties [5].

In orthopedics, the bone/implant integration strongly depends on the surface properties of the prosthetic device. Also the integrity of implant/tissue interface and the biological response of surrounding tissues is considered extremely important for enhanced osseointegration at the early stage of implantation [6]. Dissolution of ions from the implant surface and its accumulation at the interface could be one of the main reasons causing inflammatory reactions in the body [7].

The presence of body fluids, minerals, chlorides, amino acids and proteins in the human physiological environment could promote corrosion of stainless steel implants. The ionic species such as iron (Fe), chromium (Cr), nickel (Ni) and molybdenum (Mo) could release and accumulate within the surrounding tissues or may be transported through the blood stream to the various parts of body. These ions may initiate inflammatory reactions within surrounding tissues [7–10]. The build-up of metal debris in the soft tissues of the body may lead to necrosis [11] and aseptic loosening of knee joint (common example of implant failure), which can be life threatening in older age [12]. Therefore, the formation of stable passive oxide film over the implant surface is necessary which could limit the release of metallic ions.

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For cardiovascular devices, the chemical composition, roughness and surface energy are the most important properties of stainless steel for its better thrombogenicity and electrochemical stability [13]. The non-homogenous surfaces could initiate adhesion and aggregation of platelets which may activate the plasmatic coagulation and immunological responses [14]. The intrinsic coagulation of proteins and platelets at the implant surface could induce clotting and thrombosis [15]. Furthermore, the surface characteristics could influence neointimal hyperplasia, which may result in the disruption of endothelial layer and triggers the cellular response at the wall of vascular muscle. Thus, rapid proliferation of the smooth muscle cells could promote restenosis [16]. Therefore, in order to reduce inflammation and immunological response at the implant/tissue interface, surface modification of implant materials (such as stainless steel) is considered extremely important [17].

The electropolishing is an electrochemical surface treatment process involving anodic dissolution of metal or alloy in an appropriate electrolyte to restore defect free and smooth surface [18]. During electropolishing, the simultaneous surface dissolution and brightening effect could generate smooth mirror-like surface. In this way the deleterious influence of surface defects, microstructural variations and preferred crystallographic orientations on the electrochemical properties and biological response could also be minimized. It is therefore considered that the electropolishing of 316L stainless steel implants could be an effective method to improve corrosion resistance, biocompatibility and service longevity [19].

In this research work, the electropolishing of 316L stainless steel was carried out under optimized conditions. The current–voltage (*V–I*) curves for electropolishing could be divided into two regions as '*below oxygen evolution*' and '*at oxygen evolution*' potential as shown in Fig. 1. Mechanistically, below the oxygen evolution potential, the electrochemical reactions during electropolishing are under pure kinetic controlled regime resulting in the synergistic dissolution and passivation of the surface whereas at the oxygen evolution potential the electropolishing is carried out during the dissociation of aqueous electrolyte [20]. The produced surfaces were characterized to examine their topographical, chemical, structural and electrochemical properties in the simulated body environment. Furthermore, their biological response was also investigated to evaluate the proliferation behavior of MC3T3 pre-osteoblast cells.

Table 1
Chemical composition of 316L stainless steel (wt.%).

C	Mn	Cr	Ni	Mo	N	P	S	Fe
0.03	2.00	17.0	11.5	2.00	0.02	0.045	0.030	Balance

2. Experimental

2.1. Sample preparation

The circular disks, 1.6 cm in diameter and 0.5 cm thickness were cut from a commercial grade 316L stainless steel rod. The stainless steel rod was purchased from onlinemetals.com and its chemical composition is given in Table 1 as provided by the supplier. The disks were ground sequentially on a Buehler® abrasive belt grinder by using silicon carbide papers of 240, 320, 400, 600, 800 and 1200 grit size. These disks were washed in deionized water followed by ultrasonic cleaning in ethanol for 15 min. The ground disks were electropolished both below and at oxygen evolution potential at Electrobright® (Macungie, PA, USA) facility. Briefly, 85% phosphoric acid (H_3PO_4) mixed with 93% sulfuric acid (H_2SO_4) in a volume ratio of 7:3 at 60 °C was used for electropolishing at the oxygen evolution potential (EPO). On the other hand, electropolishing below the oxygen evolution plateau (EPBO) was carried out in 100 ml methanol (CH_3OH) containing 300 ml of 93% sulfuric acid solution at 25 °C. In both cases of electropolishing, 10 V DC potential was applied for 5 min and copper (Cu) was used as cathode. In the following discussion, mechanically polished and electropolished disks at and below the oxygen evolution potentials are designated as MP, EPO and EPBO samples, respectively.

2.2. Surface characterization and X-ray photoelectron spectroscopy

The surfaces of MP, EPO and EPBO samples were examined by a scanning electron microscopy (SEM) (Sigma VP Carl Zeiss, Germany). The surface roughness was measured by atomic force microscopy (AFM) (Nanoscope IV MultiMode, Digital Instruments, USA) operated under tapping mode and scanning 10 μm × 10 μm

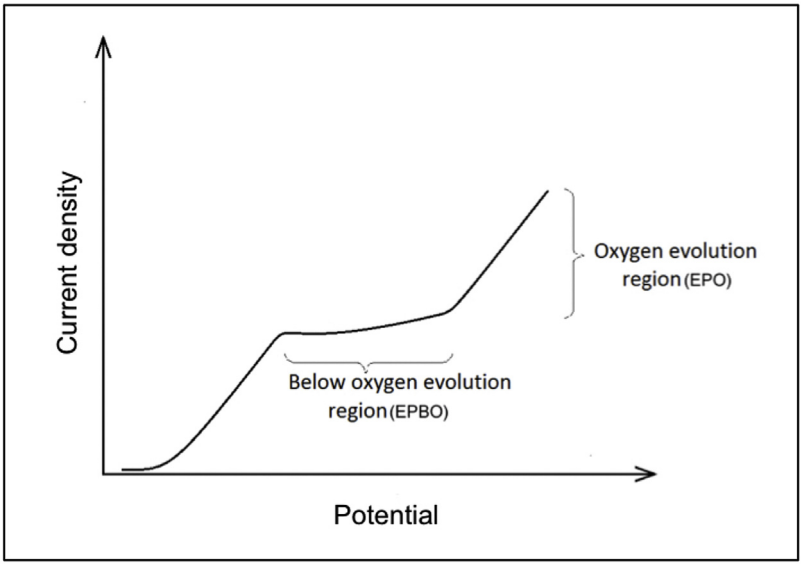

Fig. 1. Anodic polarization scan for 316L stainless steel showing two regions of electropolishing.