

Laser-assisted Zr/ZrO₂ coating on Ti for load-bearing implants

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Abstract

Oxidized Zr alloys have been shown to exhibit lower friction and superior wear properties, suggesting that they could be used in hip and knee implants. However, conventional oxidation of Zr alloys above 500 °C, in dry air, for several hours has been shown to have detrimental effects on the substrate's properties. In this work, we deposited pure Zr on Ti, then oxidized the coating using a continuous-wave Nd:YAG laser, which facilitated localized heating to elevated temperatures without affecting the substrate. Laser-assisted oxidation resulted in a 7 μm thick fully dense ZrO₂ layer on Zr in which an increase in oxidation kinetics was evident due to an increase in the laser power and/or the oxygen partial pressure. Due to its high surface energy and wettability, the wear rate of laser-oxidized Zr was two orders of magnitude less compared to that of as-deposited Zr. The oxidized coatings showed comparable in vitro biocompatibility to that of pure Ti and excellent in vitro cell–material interactions. This article reports the processing of Zr/ZrO₂ coatings on Ti using lasers, and the influence of laser parameters and oxygen partial pressure on the coating's mechanical, microstructural, wear and in vitro biological properties using human osteoblast cells.

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1. Introduction

The long-term failure mode of current load-bearing metal implants, such as total hip arthroplasty (THA) and total knee arthroplasty (TKA), is primarily the wear of ultrahigh molecular weight polyethylene (UHMWPE), which leads to osteolysis, implant loosening and instability [1–4]. The wear of UHMWPE primarily arises from hard particulate debris, such as bone cement, bone chips and spalled oxide films, embedded in the UHMWPE liner and scratching the harder metal/ceramic counterface, making it rough and then abrading the polyethylene. Several attempts have been made to minimize the wear-induced osteolysis, including the use of design modifications [5], UHMWPE property modification [6] and alternate bearing couples, such as metal-on-metal and ceramic-on-ceramic

implants, thereby eliminating the use of UHMWPE [7–9]. Design modifications can only change the contact stresses and related fatigue wear of the UHMWPE [5], while the modification of UHMWPE properties has not yielded any significant improvement in wear performance [10]. In addition, roughening and scratches on the hard counterface of femoral components can increase UHMWPE wear via adhesive and abrasive wear mechanisms [11,12]. An alternative and more effective solution is to modify the hard counterface articulating with UHMWPE to reduce its wear and debris generation. Therefore, a hard counterface providing low friction with UHMWPE and resisting roughening can reduce abrasive and adhesive wear, and thereby enhance the long-term stability and survival of load-bearing metal implants. Hard, inert ceramic surfaces appear to accomplish this long-term performance goal most effectively [13].

Ceramic coatings have been proposed for THA in the past as being wear-resistant and a barrier to metal ion release. These coatings were obtained using different vapor

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deposition techniques, such as physical vapor deposition, ion implantation and sputtering, and the coatings consisted of diamond-like carbon [14,15] or nitrides [16,17]. In spite of their quite low wear coefficients, these coatings have found little application in the field of THA/TKA due to their inherent brittleness and catastrophic fracture possibilities. Hard alloy or ceramic coatings can increase the hardness but frequently delaminate, and externally applied ceramic coatings are susceptible to brittle fracture, thus contributing to additional wear debris. Also, alloy coatings may not always decrease the wear of UHMWPE. Therefore, thick ZrO₂ scale grown in situ by self-oxidation of Zr provides an alternative to alloy/ceramic coatings and monolithic ceramic prostheses [18,19]. Laboratory tests under simulated wear conditions show that, compared to CoCrMo alloy, an oxidized Zr articulation surface exhibits a much lower friction coefficient [20] and UHMWPE wear rates that are lower by more than a factor of five [21,22] when articulating against UHMWPE. Moreover, in vivo experiments and clinical trials demonstrate that Zr components are hypoallergenic, biocompatible and osteoconductive [23–25]. Finally, Zr and its alloys have no Ni impurities and can be applicable to patients with Ni or Co allergies. OXINIUM™ (oxidized zirconium) is one such commercially available material for joint replacements manufactured by Smith & Nephew Orthopaedics, Memphis, TN.

Performance of oxidized Zr in prosthetic service depends on the oxide layer's adherence to the substrate, resistance to spalling and maintenance of a smooth surface during articulation. It is also important for the substrate to have sufficient strength to provide support for the more brittle overlying scale and to support a thick oxide layer to resist articulation forces. For orthopedic application, oxide scale thicknesses of ~5 μm are generally achieved on Zr and Zr–Nb alloys after several hours of oxidation in dry air between 600 and 700 °C [26]. For thicker coatings one needs to oxidize the metal/alloy at significantly higher temperatures or for longer times. Such an exposure might have a detrimental effect on mechanical properties of the substrate due to excessive oxygen diffusion into the base metal, which could change its microstructural features [27,28]. In addition, high-temperature oxidation has been found to develop undesirable voids in the oxide scale [29], lowering its stability during service. In this work, we propose instantaneous oxidation of Zr using high-energy lasers. We first deposited pure Zr on Ti, then oxidized the Zr coating using Laser Engineered Net Shaping (LENS™), which facilitates localized heating of Zr to elevated temperatures without significantly affecting the substrate properties. In addition, being a computer-aided design- and layer-based manufacturing process, LENS™ enables us to fabricate innovative, unitized implants [30,31], such as functionally graded Ti acetabular shells, with open porosity on one side (in contact with the bone) to improve cell–material interactions and a hard ZrO₂ film on the other side (in contact with UHMWPE liner) to decrease the liner wear rate, which can significantly

improve the implant's life in vivo. A schematic of such a gradient structure is shown in Fig. 1. However, the present article focuses on the fabrication, coating characterization and in vitro biocompatibility of laser-assisted Zr/ZrO₂ coating on commercially pure (cp) Ti for potential applications in load-bearing metal implants.

2. Materials and methods

Zr metal powder (CERAC Inc., Milwaukee, WI) with 99.8% purity and particles size between 45 and 106 μm was used. Zirconium coatings of ~1.5 mm thickness, having diameters of 10 and 20 mm, were deposited on a substrate of 3 mm thick rolled cp Ti plates using LENS™-750 (Optomec Inc. Albuquerque, NM) equipped with a 500 W continuous-wave Nd:YAG laser. A detailed description of the operation and capabilities of LENS™ can be found elsewhere [32]. Initial Zr coatings were fabricated in a glove box containing an argon atmosphere with O₂ content less than 10 ppm to limit oxidation of Zr during processing. After process optimization, a laser power of 400 W, a scan speed of 10 mm s⁻¹ and a powder feed rate of 19 g min⁻¹ were used to deposit Zr metal on the Ti substrate. These coatings were subsequently oxidized in the same glove box, using LENS™, with oxygen partial pressures of 7.37 × 10⁻⁵, 1.47 × 10⁻⁴ and 2.09 × 10⁻¹ atm (hereafter referred as O₂ contents of 2%, 4% and 21%, respectively). While the O₂ partial pressure was varied, the laser power was also varied from 100 to 200 and 300 W at a scan speed of 5 mm s⁻¹ to grow ZrO₂ layers of different thickness.

Cross-sectional microstructures of the samples were examined using scanning electron microscopy (SEM) and field emission SEM (FEI – Quanta 200F). Constituent phases in the top layer of as-deposited and oxidized Zr coatings were identified using a Siemens D 500 Kristalloflex diffractometer with Cu K_α radiation (1.54056 Å) at 20 kV within the 2θ range of 20–80°, and compared with those of as-received powder. Vickers microhardness measurements (Leco, M-400G3) were also made on the as-fabricated and oxidized Zr coatings using a 300 g load and on as-received powder using a 50 g load for 20 s, and the average value of 10 measurements was reported. In order to track the oxygen diffusion across the deposit, a series of microhardness indentations were placed from one end of the deposit to the other, with neighboring indents being separated by 0.1 mm using a Vickers microhardness tester at 300 g load applied for 15 s.

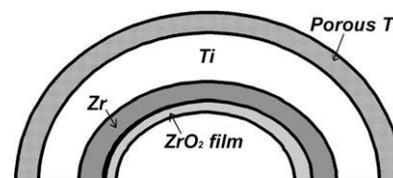


Fig. 1. Schematic diagram showing the cross section of a novel acetabular shell design.

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