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Porous niobium coatings fabricated with selective laser melting on titanium substrates: Preparation, characterization, and cell behavior

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ABSTRACT

Nb, an expensive and refractory element with good wear resistance and biocompatibility, is gaining more attention as a new metallic biomaterial. However, the high price of the raw material, as well as the high manufacturing costs because of Nb's strong oxygen affinity and high melting point have limited the widespread use of Nb and its compounds. To overcome these disadvantages, porous Nb coatings of various thicknesses were fabricated on Ti substrate via selective laser melting (SLM), which is a 3D printing technique that uses computer-controlled high-power laser to melt the metal. The morphology and microstructure of the porous Nb coatings, which had pores ranging from 15 to 50 μm in size, were characterized with scanning electron microscopy (SEM). The average hardness of the coating, which was measured with the linear intercept method, was 392 ± 37 HV. In vitro tests of the porous Nb coating which was monitored with SEM, immunofluorescence, and CCK-8 counts of cells, exhibited excellent cell morphology, attachment, and growth. The simulated body fluid test also proved the bioactivity of the Nb coating. Therefore, these new porous Nb coatings could potentially be used for enhanced early biological fixation to bone tissue. In addition, this study has shown that SLM technique could be used to fabricate coatings with individually tailored shapes and/or porosities from group IVB and VB biomedical metals and their alloys on stainless steel, Co–Cr, and other traditional biomedical materials without wasting raw materials.

1. Introduction

Recently, new metallic biomaterials based on the expensive and refractory elements from IVB and VB groups such as Ta, Zr, and Nb, which have good wear resistance, are gaining more attention [1]. In vitro apatite formation test and in vivo histomorphometric study have shown that these materials are bioactive and can biologically bond to bone [2,3]. However, the high price of the raw materials and relatively high manufacturing costs to produce pure Ta or Nb implants have limited their widespread use [4]. The extremely high melting point of Nb (2468 °C), as well as its high affinity for oxygen, makes it difficult to fabricate Nb implants via conventional processing routes. Lately, the metal additive manufacturing (MAM) process, a 3D printing technique, offers a nearly unlimited flexibility by using high-power laser beam, and provides special opportunities for orthopedic and bone implants [5]. Compared with other MAM techniques, selective laser melting (SLM) allows layers and coatings of other refractory metal materials to be fabricated on the surface of metal implants [6]. A schematic of a SLM set-up is shown in Fig. 1. It is more accurate than other MAM techniques because it uses fiber laser, which possesses smaller spot sizes (<100 μm) and highly concentrated output energies compared with other lasers [7]. By combining the high-energy fiber laser with a computer-controlled laser beam system [8,9], this gives the ability to control the melting of Nb, and thus, the ability to create implants with tailored shapes and/or porosities. Further details about the SLM procedure and a description of the parameters have been reported in a previous study [10]. Recently, SLM has been used to fabricate common, metal-based medical materials, which contain Ti and its alloys, Co–Cr alloys, stainless steel, and others [11], that have been widely used for orthopedic and dental implants [12,13].

In this paper, we report the first example of an irregular, porous, Nb coating fabricated directly with SLM on pure Ti. We investigated the fabrication of porous Nb coatings on pure Ti, and performed in vitro studies to assess the cell attachment, morphology, and proliferation of the samples. These new porous Nb coatings could potentially be used for enhanced early biological fixation to bone tissue. In addition, this study has shown that SLM can be used to fabricate coatings with

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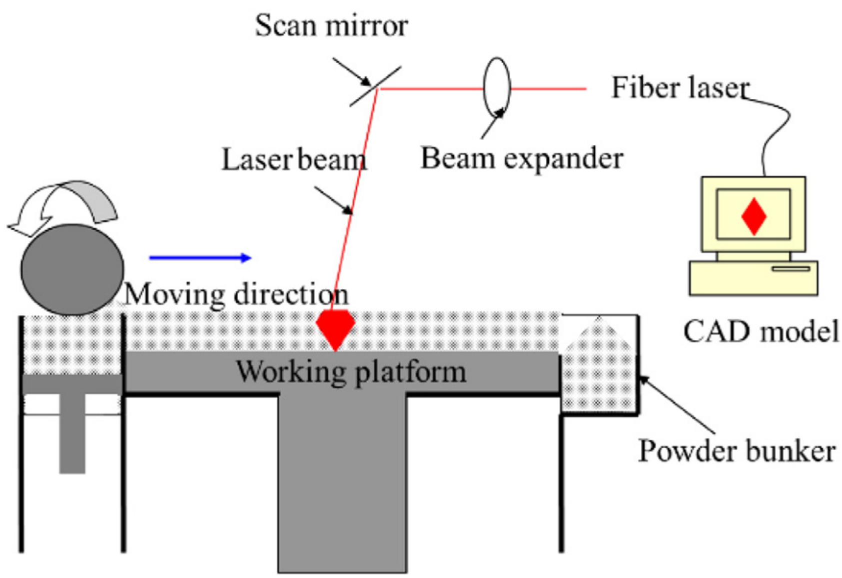

Fig. 1. Schematic of the selective laser melting (SLM) set-up.

individually tailored shapes and/or porosities from group IVB and VB biomedical metals and their alloys on stainless steel, Co–Cr, and other traditional biomedical materials, without wasting raw material.

2. Materials and methods

2.1. Materials

The materials used were Nb powder (99.5% purity, particle size = 15–125 μm, Xing Rongyuan Technology Co. Ltd., Beijing, China), and commercially available pure Ti (grade 2, Baoji Ti Industry Co., Ltd., Baoji, China). MC3T3-E1 osteoblast-like cells (ATCC catalog CRL-2593) were obtained from the American Type Culture Collection. All the other chemical reagents were local products of analytical grade.

2.2. Fabrication of the porous Nb coating

A porous Nb coating (thickness = 2.0 mm, diameter = 8 mm) was deposited on a Ti substrate with a SLM machine (HRPM-II, Rapid Manufacturing Center, Huazhong University of Science and Technology, China). The SLM processing was performed with a 200 W fiber laser (YLP-HP, IPG Photonics Corporation, Germany) in a high-purity argon atmosphere to limit the oxidation of Nb and Ti in the fabrication chamber. After process optimization, a laser power of 160 W, scanning speed of 200 mm/s, hatching spacing of 0.07 mm, and layer thickness of 0.04 mm were applied to melt Nb onto the Ti substrate. The optimization of the processing parameters ensured that the residual stress was not large enough to cause warping of the thin Nb coating. In addition, special scanning strategies were adopted to fabricate the porous Nb coatings. Before the fabricating process, 3D CAD data was prepared and then the powder was sliced into layers with a defined thickness in the vertical direction. As shown in Fig. 2, the scan lines of each subsequent layer are perpendicular to the previous layer. Therefore, the SLM process involves a series of repetitive procedures that form a layer of powder and then transfers specific geometric information onto the material by melting the powder with a laser beam. Under these processing conditions, the porous Nb coating was found to be visually sound, with a strong adherence to the Ti substrate.

2.3. Characterization of the porous Nb coating

The cross-sectional microstructure of the samples was observed with optical microscopy and field emission scanning electron microscopy (FE-SEM, JEOL-JSM7600F, Japan). The chemical composition of the Nb powder was measured with dispersive X-ray spectroscopy (EDS, X-Max 50, INCA, England). To reveal microstructural features, the chemically polished samples were then etched for 60 s with a solution containing 20 ml of HNO_3, 20 ml of HF, and 60 ml of H_2SO_4. To track any diffusion that may occur at the interface between the porous Nb coating and Ti substrate, a series of micro-hardness indentations were applied to the deposited Nb from one end to the other, with adjacent indents of 0.05 mm spacing, using a Vickers micro-hardness tester (HXS-1000AK, Shanghai, China) with a load of 100 g for 15 s.

2.4. In vitro tests

MC3T3-E1 osteoblast-like cells were cultured in an alpha minimum essential medium (α-MEM, Gibco, Grand Island, NY, USA) supplemented with 10% fetal bovine serum (FBS, Gibco, Grand Island, NY, USA) at 37 °C in a humidified, 5% CO_2 atmosphere. When the cells reached 80–90% confluence, they were trypsinized and suspended in the culture medium. Having been steam sterilized and placed in 24-well tissue culture plates

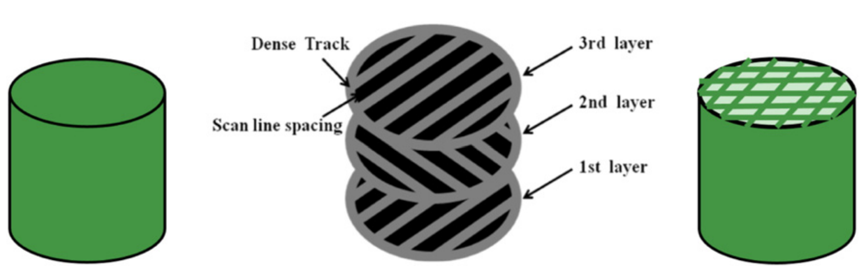

Fig. 2. A schematic of special scanning strategies used to fabricate the porous Nb coating on pure Ti.